*Fibers and Polymers* 2011, Vol.12, No.7, 957-962 DOI 10.1007/s12221-011-0957-8

# Optimization of Dyeing Condition for Wool/Cotton Union Fabric with Direct Dye Using Box-Behnken Design

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(Received January 16, 2010; Revised October 19, 2010; Accepted May 25, 2011)

**Abstract:** Wool/cotton union fabric was dyed with a direct dye in union shade. The dyeing was performed in a single bath in relation to four factors: leveling agent (Lyogen SMKI, 0-1.5 % oww), electrolyte (sodium sulfate, 0-10 %), dyeing temperature (85-95 °C), and dyeing time (15-60 min). The dyeing was characterized by dye bath exhaustion (%), color strength (*K/S* value), washing fastness, and light fastness of dyed sample. Response surface analysis showed that the exhaustion of direct dye increased with electrolyte, dyeing temperature, and dyeing time while the *K/S* value followed a linear shape with leveling agent and dyeing temperature. An optimized recipe was formulated based on response surface strategy and numerical optimization solution.

**Keywords:** Cotton, Direct dye, Exhaustion, Union fabric, Wool

## Introduction

The rising demand for relaxed and resilient all-seasonal clothing, especially in the casual-wear market has awakened a renewed interest not only in all-cotton and all-wool but also in blends of cotton and wool. Since the wool/cotton blends have both the comfort of cotton and the resilience of wool, the clothes made of the blends are worn by many people year-round and these blends ranging from 80/20 to 20/80 of cotton/wool are preferentially found in sportswear, active wear, and home furnishings [1-3]. Apart from intimate blends, union fabrics with cotton warp and woolen weft are also used for blazer cloth, gabardine rainwear, shirt, and pajamas. Wool/cotton blended fabrics require unique wet processing as well as dry processing such as singeing to produce high quality goods [4]. There are several techniques are available for dyeing of wool/cotton blends to achieve union shade. The suitable dyeing technique depends on the fastness requirement, depth of shade, blend ratio, and cost [5].

The most common technique for achieving light and medium shade in union shade is using direct dyes on cotton and acid dyes/2:1 pre-metalized dyes on wool via either two baths or single bath-two stage process. Reactive dyes are also used for union dyeing of wool blends through either a single bath or two bath process with prior treatments of auxiliaries like sodium edate, bi-guanidine, etc [6-9]. The cationic pretreatment provided a strong attraction to anionic dyes like acid dye, reactive dye, and direct dye [10]. Generally dyeing of 100 % wool textiles with anionic dyes requires a leveling agent for retarded dye adsorption and evenness, which results in excellent penetration and level dyeing [11,12]. Dyeing of cotton textiles with direct dyes also requires an electrolyte at neutral pH, which enhances the dyeing rate as well as diffusion inside the fiber [13]. The research work on union dyeing of wool/cotton blends particularly on union fabric with direct dye in one bath is very rare.

It would be possible to develop a dyeing system for wool/cotton union fabric with a leveling agent, Lyogen SMKI (SMKI) for wool component and exhausting agent, sodium sulfate (SS) for both wool and cotton to achieve union shade. The aim of this research is to optimize the dyeing condition of wool/cotton union fabric in union shade with a direct dye at neutral pH and to investigate the effects of dye bath additives (SMKI and SS), dyeing temperature, and dyeing time. A Box-Behnken design was used to fit a second order model that would be required for optimal dyeing condition. The dyeing was characterized by dye bath exhaustion (%), color strength (*K/S*), washing and light fastness properties.

## Experimental

### Materials

Wool/cotton union fabric with woolen weft (4 Nm) and cotton warp (2/15's) having 34 EPI, 22 PPI, 292 g/m$^2$, 2/2 twill weave, and 1.09 mm thickness was chosen for present study. The blend ratio was estimated as per standard procedure and the ratio of wool:cotton 71:29 [14]. Indosol Red BA (CI Direct Red 110), leveling agent (Lyogen SMKI), and non-ionic detergent (Sandozin NIS Liq) supplied by Clariant Chemicals (India) Ltd., Mumbai, India were used in this present study. All other chemicals used elsewhere were AR grade.

### Preparation of Fabric for Dyeing

The wool/cotton union fabric was mild-scoured with Sandozin NISI (1 g/*l*), sodium carbonate (0.5 g/*l*) at 50 °C for 30 min, neutralized with acetic acid solution followed by rinsing with distilled water and dried at ambient condition.

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### Dyeing of Union Fabric

A four-factor, three-level Box-Behnken study design was used for experimental plan [15,16] and the variables are shown in Table 1. Dyeing was carried out in temperature controllable water bath. The sample (12×12 cm) was taken in a bath containing the required amount of Lyogen SKMI (0.0, 0.75, 1.5 % oww), sodium sulfate (0, 5, and 10 % owf) and kept for 15 min at 50 °C using a liquor ratio of 60:1. The required dye solution (1 % owf) was added in two portions at 50 °C over 15 min and the temperature was raised at the rate of 1.5 °C/min to specified temperature (85, 90, and 95 °C) and dyeing was carried out at that temperature for specified duration (15, 30, and 60 min). After dyeing, the dyeing bath was cooled to 60 °C, dyed sample was taken out, washed with distilled water, and dried at ambient condition.

### Measurement of Dye Bath Exhaustion (DBE)

A double beam UV-VIS spectrophotometer (ECIL, Hyderabad, India) was used to determine the optical density of dye solution at the wavelength of its maximum absorption ($\lambda_{\max}$). The percentage exhaustion of the dyestuff from the dye bath was calculated at 520 nm according to following formula.

$$\text{Percentage dye bath exhaustion (\%)} = \frac{A_1 - A_2}{A_1} \times 100$$

where $A_1$ and $A_2$ are the quantities of dye (mg) present in the dyeing bath, before and after dyeing, respectively.

### Measurement of Color Strength

The reflectance values of dyed samples were evaluated using a JAYPAK 4802 Color matching system (Jay Instruments Ltd., Mumbai, India) under D65/10° illumination/observer condition. The dyed samples were folded twice to give an opaque sample with four layers, measured in two different areas; refolded to expose the other side of the sample, and measured in two different areas at 520 nm ($\lambda_{\max}$ of the dye). The average of these four readings was taken and color strength values (*K/S*) were calculated according to AATCC Evaluation Procedure 6 [17]. Color difference ($\Delta E$) was calculated using the following equation as follows.

$$\Delta E = \sqrt{(L_1^* - L_2^*)^2 + (a_1^* - a_2^*)^2 + (b_1^* - b_2^*)^2}$$

where $L_1^*$, $a_1^*$, $b_1^*$ and $L_2^*$, $a_2^*$, $b_2^*$ were the corresponding colorimetric values of the control and dyed samples, respectively.

**Table 1.** Variables in the box-behnken design

| Variable | Level used | | |
|---|---|---|---|
| | Low (-1) | Medium (0) | High (+1) |
| *Independent variables* | | | |
| Lyogen SMKI : $x_1$ (% oww) | 0.0 | 0.75 | 1.50 |
| Sodium sulphate : $x_2$ (% owf) | 0.0 | 5.0 | 10.0 |
| Dyeing time : $x_3$ (minutes) | 15 | 30 | 60 |
| Dyeing temperature : $x_4$ (°C) | 85 | 90 | 95 |
| *Dependent variables* | | | |
| Dye bath exhaustion : $y_1$ (%) | | | |
| Colour strength (*K/S*) : $y_2$ | | | |
| Washing fastness : $y_3$ (grade) | | | |
| Light fastness : $y_4$ (grade) | | | |

### Evaluation of Fastness Properties

The fastness properties of the dyed samples were tested according to Indian standard. The specific tests were color fastness to daylight IS: 686-1985 and color fastness to washing IS: 687-1979 [14].

### Experimental Design

A four-factor, three-level Box-Behnken design was used in this study. This design is suitable for exploration of

**Table 2.** Experimental data and the observed response values used in the Box-Behnken for the response surface methodology[a]

| Run | Factor level | | | | Experimental data | | | |
|---|---|---|---|---|---|---|---|---|
| | $x_1$ | $x_2$ | $x_3$ | $x_4$ | $y_1$ | $y_2$ | $y_3$ | $y_4$ |
| 1 | 1.50 | 0 | 30 | 90 | 48.1 | 1.727 | 2-3 | 6-7 |
| 2 | 0.00 | 5 | 15 | 90 | 41.9 | 1.828 | 2-3 | 6-7 |
| 3 | 0.75 | 0 | 30 | 85 | 38.6 | 1.553 | 2-3 | 6 |
| 4 | 0.75 | 0 | 30 | 95 | 48.7 | 2.003 | 2-3 | 7 |
| 5 | 0.75 | 5 | 30 | 90 | 52.8 | 1.881 | 2-3 | 7 |
| 6 | 1.00 | 5 | 15 | 90 | 46.5 | 1.832 | 2-3 | 7 |
| 7 | 0.75 | 5 | 15 | 85 | 40.9 | 1.632 | 2-3 | 6-7 |
| 8 | 0.75 | 0 | 60 | 90 | 49.9 | 2.047 | 2-3 | 7 |
| 9 | 0.75 | 10 | 30 | 95 | 50.6 | 1.811 | 2-3 | 6-7 |
| 10 | 0.75 | 5 | 15 | 95 | 42.5 | 1.913 | 2-3 | 7 |
| 11 | 0.75 | 0 | 15 | 90 | 36.4 | 1.544 | 2-3 | 6-7 |
| 12 | 0.75 | 10 | 30 | 85 | 59.8 | 2.149 | 2 | 7 |
| 13 | 0.75 | 5 | 30 | 90 | 50.6 | 1.786 | 2-3 | 6-7 |
| 14 | 0.75 | 5 | 60 | 85 | 52.3 | 2.063 | 2 | 7 |
| 15 | 1.00 | 10 | 30 | 90 | 60.2 | 2.024 | 2 | 7 |
| 16 | 1.00 | 5 | 30 | 85 | 49.8 | 1.682 | 2-3 | 6-7 |
| 17 | 0.75 | 10 | 60 | 90 | 68.4 | 2.238 | 2 | 7 |
| 18 | 1.00 | 5 | 60 | 90 | 59.5 | 2.107 | 2 | 7 |
| 19 | 0.75 | 5 | 30 | 90 | 53.1 | 1.913 | 2-3 | 7 |
| 20 | 1.00 | 5 | 30 | 95 | 56.3 | 2.132 | 2 | 7 |
| 21 | 0.75 | 5 | 30 | 90 | 53.4 | 1.924 | 2-3 | 7 |
| 22 | 0.00 | 0 | 30 | 90 | 37.9 | 1.578 | 2-3 | 6 |
| 23 | 0.00 | 10 | 30 | 90 | 42.5 | 1.913 | 2-3 | 7 |
| 24 | 0.75 | 5 | 30 | 90 | 51.8 | 1.875 | 2-3 | 6-7 |
| 25 | 0.00 | 5 | 60 | 90 | 53.6 | 2.069 | 2-3 | 6-7 |
| 26 | 0.75 | 10 | 15 | 90 | 50.3 | 1.905 | 2-3 | 7 |
| 27 | 0.75 | 5 | 60 | 95 | 63.3 | 2.272 | 2 | 7-8 |
| 28 | 0.00 | 5 | 30 | 95 | 40.7 | 1.614 | 2-3 | 6 |
| 29 | 0.00 | 5 | 30 | 85 | 50.7 | 2.092 | 2 | 6-7 |

[a]: Abbreviations: see Table 1.

quadratic response surfaces and construction of a second order polynomial model, thus helping in optimizing a process using a small number of experimental runs [15,16]. A total of 29 samples were dyed as per conditions given in Table 2. The design consists of three replicated center points, and a set of six points lying at the midpoints of each edge of the multidimensional cube.

### Statistical Analysis

The nonlinear computer generated quadratic model given below was used for analysis of the data.

$$y = b_0 + \sum_i b_i x_i + \sum_i b_{ii} x_i^2 + \sum_j \sum_{k(k>j)} b_{jk} x_j x_k$$

with $i=x_1-x_4$, $j=x_1-x_3$, and $k=x_2-x_4$, where $y$ is the response variables, $b$ is the regression coefficients of the model, and $x$ is the coded level of an independent variable. We applied regression analysis using Design Expert software Version 7.1.2 to arrive at the statistical significance between variables and its effect on the outputs. Further, the effects of interaction among the variables on the outputs also were studied. The regression equation above was optimized using Design Expert software Version 7.1.2 (Stat-Ease Inc., Minneapolis, USA) [18]. The statistical significance of the second order model equation was determined by $F$- value, and the multiple coefficient of determination ($R^2$).

## Results and Discussion

### Selection of Shade

A preliminary trial was undertaken by taking the warp cotton yarn and weft wool yarn of experimental wool/cotton union fabric separately and dyed with the direct dye in the following condition (dye : 0.25, 0.5, 1.0, 1.5, and 2.0 % (owf); SMKI : 0.5 % (oww); SS : 5 % (owf); 95 °C; 30 min; LR=60:1; pH 7) in order to select the suitable shade for the experiment. The dye bath exhaustion, *K/S* value, and $\Delta E$ (color strength difference between respective wool and cotton dyed samples at each shade) values are tabulated in Table 3. It is observed that the difference between wool and cotton component in DBE (1.6 %) as well as *K/S* (0.056) is lower in 1 % shade than any other shade and it is confirmed by their corresponding $\Delta E$ value (1.125). There is a possibility of non-uniform adsorption of direct dye at dark shade between wool and cotton substrate, the 1 % shade was selected for dyeing of union fabric in this study [19].

**Table 3.** Dye bath exhaustion and colorimetric value of cotton warp yarn and wool weft yarn dyed with direct dye at different shade

| %owf shade | Dye bath exhaustion (%) | | *K/S* value | | $\Delta E$ |
|---|---|---|---|---|---|
| | Cotton | Wool | Cotton | Wool | |
| 0.25 | 58.2 | 62.4 | 1.250 | 1.395 | 2.804 |
| 0.50 | 57.8 | 60.8 | 1.507 | 1.777 | 1.901 |
| 1.00 | 56.8 | 58.4 | 2.090 | 2.146 | 1.125 |
| 1.50 | 55.6 | 58.2 | 2.210 | 2.555 | 1.578 |
| 2.00 | 54.9 | 57.3 | 2.423 | 2.614 | 2.322 |

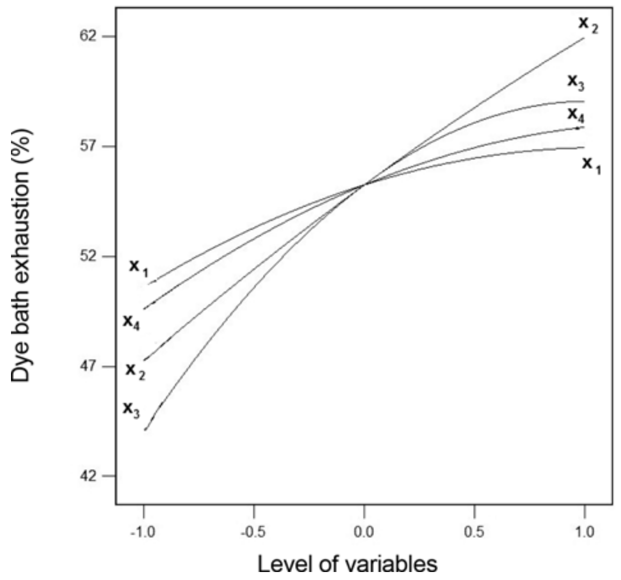

**Figure 1.** Perturbation plot the dye bath exhaustion versus the factor in code values; ($x_1$) lyogen SMKI, ($x_2$) sodium sulfate, ($x_3$) dyeing time, and ($x_4$) dyeing temperature.

### Dye Bath Exhaustion of Union Fabric

The dye bath exhaustion (DBE) is ranging between 36.4 and 68.4 %, which is similar to those usually observed in dyeing of wool/cotton blended textiles at neutral pH. The dye bath exhaustion increased linearly with increasing concentration of SS, dyeing time, and dyeing temperature (Figure 1), whereas it changed slightly with increasing SMKI. The statistical analysis indicated that DBE changed significantly ($P<0.0001$) with all factors and interaction of dyeing temperature and dyeing time. The addition of electrolyte in the dye bath facilitates the diffusion of sodium cation by mass action and consequently the amount of dye anion adsorbed by the fiber increased. The increase in dyeing time and temperature improves the migration of direct dye on both fibers, which leads to higher exhaustion [20,21].

### Color Strength of Dyed Union Fabric

The *K/S* ranged between 1.554 and 2.272 and it changed significantly with all factors ($P<0.0001$) and interaction of SMKI and dyeing time. The color strength increased with increasing concentration of SS, dyeing time, and dyeing temperature (Figure 2), whereas it slightly declined with increasing SMKI. The addition of leveling agent in this dyeing system controls the migration of direct dyestuff in wool component and so levels the dye molecules aggregation

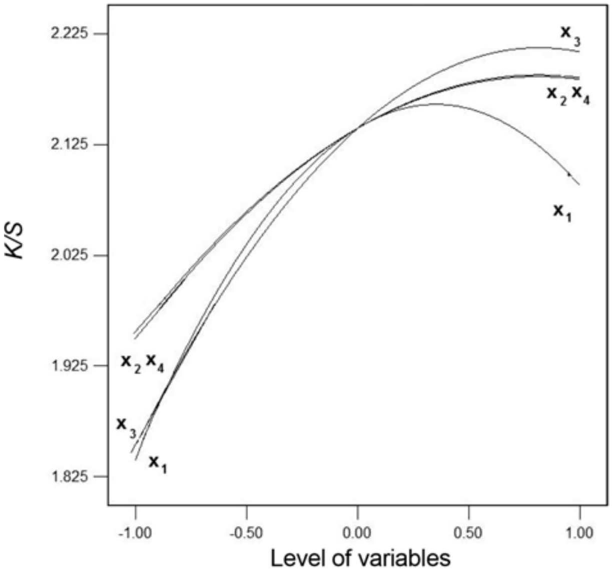

**Figure 2.** Perturbation plot the *K/S* value versus the factor in code values; ($x_1$) lyogen SMKI, ($x_2$) sodium sulfate, ($x_3$) dyeing time, and ($x_4$) dyeing temperature.

both in wool and cotton fiber matrix [22], which leads to union, shade of the fabric. The aggregation of dyestuff is increased with interaction of SMKI and dyeing temperature. Moreover, the *K/S* value is significant with quadratic relationship of $x_1$, $x_2$, $x_3$, and $x_4$.

### Fastness Properties of Dyed Fabrics

The washing fastness of dyed samples ranged between 2 and 2-3 and samples that have greater than 50 % dye bath exhaustion showed washing fastness of 2. It is inferred that this dye requires after-treatment with a cationic fixative, which might prevent stripping of direct dye. The light fastness ranged between 6 and 7-8. Generally fading of a dye due to light energy depends on the amount of aggregated dye inside the fiber [23,24]. The presence of leveling agent in the dyeing system improves the leveling as well as the aggregation of dye inside the fiber matrix, while electrolyte facilitates the diffusion and exhaustion [25]. The synergistic effect of both additives in the dyeing system improves the aggregation of dyestuff (color strength) as well as light fastness property. Since fastness property of dyed sample depends on the chemical nature of dye, these four factors show little significant effect on improving this property.

### Regression Coefficients of Analysis

The estimated regression coefficients for the quadratic model fitted to the experimental results and their significances together with the corresponding $R^2$ values are summarized in Table 4. The coefficient of determination inferred from the models for the dye bath exhaustion and *K/S* value responses are 0.98 and 0.94, respectively. Figure 3(A) and 3(B) show a parity plot of dye bath exhaustion and *K/S* value obtained with the empirical model. The actual and the predicted dye bath exhaustion and *K/S* are in good agreement and the results are always greater than zero. The deviation between the actual and predicted values for dye bath exhaustion is smaller than that for the *K/S* value.

**Table 4.** Results for the regression coefficients of the quadratic models

| Regression equation coefficients | Response variables | |
|---|---|---|
| | Dye bath exhaustion | *K/S* |
| $b_0$ | 55.24 | 2.14 |
| Linear | | |
| $b_1$ | $3.168^{***}$ | $0.12^{***}$ |
| $b_2$ | $7.33^{***}$ | $0.12^{***}$ |
| $b_3$ | $7.48^{***}$ | $0.18^{***}$ |
| $b_4$ | $4.13^{***}$ | $0.11^{***}$ |
| Quadratic | | |
| $b_{11}$ | $-1.47^{*}$ | $-0.17^{***}$ |
| $b_{22}$ | -0.64 | $-0.072^{*}$ |
| $b_{33}$ | $-3.67^{***}$ | $-0.11^{**}$ |
| $b_{44}$ | $-1.51^{*}$ | $-1.51^{*}$ |
| Interaction | | |
| $b_{12}$ | -0.87 | 0.016 |
| $b_{13}$ | -0.054 | $9.647\text{E-}003^{*}$ |
| $b_{14}$ | -0.58 | 0.087 |
| $b_{23}$ | 1.41 | -0.042 |
| $b_{24}$ | 0.52 | 0.022 |
| $b_{34}$ | $2.02^{*}$ | -0.013 |
| P-value | <0.0001 | <0.001 |
| $R^2$ | 0.9755 | 0.9351 |

$^{*}$Significance at the 0.05 level ($P<0.05$), $^{**}$significance at the 0.01 level ($P<0.01$), $^{***}$significance at the 0.001 level ($P<0.001$), $b_0$: constant, $b_1$: coefficient of SMKI, $b_2$: coefficient of SS, $b_3$: coefficient of time, and $b_4$: coefficient of temperature.

The relationship of the regression coefficients of the dye bath exhaustion is significant with linear model for all factors and a quadratic model for $x_1$, $x_3$, and $x_4$ with an interaction between $x_3$ and $x_4$. Dyeing time (7.48) gives the largest value for the coefficient of dye bath exhaustion. It infers that by increasing the dyeing time, the number of adsorbed dye molecule at equilibrium reduced [26]. The *K/S* value is significant in both linear and quadratic models for all factors with an interaction between $x_1$ and $x_3$. The coefficient of dyeing time has good significance and the largest value (0.18) that increases the *K/S* value most. In direct dye, production of level and well-aggregated dyeing is usually favored by the increased dyeing time and so dyeing time has significant influence on the improvement of both dye bath exhaustion and *K/S* value.

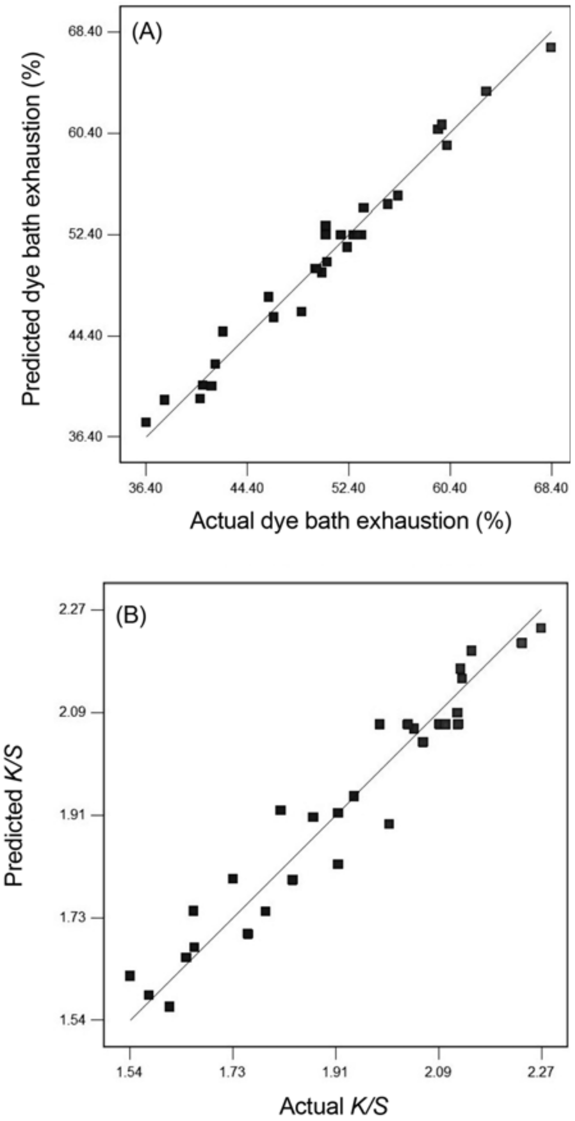

**Figure 3.** Actual values of dye bath exhaustion (A) and colour strength (B) versus predicted values given by the model.

**Table 5.** Optimum conditions for the union dyeing of wool/cotton union fabric with 60 % dye bath exhaustion and 2.200 *K/S* value

| Response | Required | Optimum |
|---|---|---|
| Dye bath exhaustion (%) | 60.00 | 60.01 |
| Colour strength (*K/S* value) | 2.200 | 2.200 |
| Washing fastness (grade) | 2 to 2-3 (in range) | 2-3 |
| Light fastness (grade) | 6-7 to 7 (in range) | 7 |

| Factor | Code | Level |
|---|---|---|
| SMKI (% oww) | $x_1$ | 1.48 |
| SS (% owm) | $x_2$ | 5.00 |
| Dyeing time (min) | $x_3$ | 47 |
| Dyeing temperature ($^{o}$C) | $x_4$ | 92 |

### Optimization of Dyeing Condition

The main objective of this work has to develop union shade at neutral pH with optimum dye bath exhaustion, *K/S*, and fastness properties on wool/cotton union fabric. The norms for optimized dyeing condition are set as follows: 60 % dye bath exhaustion; 2.2 *K/S* value; 2 to 2-3 washing fastness; and 6-7 to 7 light fastness. The optimized conditions could be determined with response surface strategy and numerical optimization solution by Design Expert software Version 7.1.2. Out of 55 solutions, 10 solutions have shown desirability of 1.000 and the following conditions have been selected as optimized conditions: SMKI-1.48 %; SS-4.5 %; Time 47 min; Temperature 92 $^{o}$C (Table 5).

Dyeing is carried out at this optimized condition and it showed 61.6 % dye bath exhaustion, 2.175 *K/S* value, 2-3 washing fastness, and 6-7 light fastness. It infers that the actual and the predicted dye bath exhaustion, *K/S* value, and fastness properties at optimized condition are in good agreement at more than 95 % level. The washing fastness of the direct dye could be improved by cationic fixative and/or application of a glyoxal resin finishing treatment.

## Conclusion

Generally, the study pertaining to coloration of two fibers has been achieved by using two classes of dyes as well as by two baths. The results obtained from this work inferred that wool/cotton union fabric could be dyed in union shade with direct dye at 92 $^{o}$C in the presence of Lyogen SMKI (1.5 % oww), sodium sulfate (4-5 % owf) for 45 min at neutral pH in one bath, which gave 61.60 % dye bath exhaustion, 2.175 *K/S* value with moderate washing fastness (2-3) and good light fastness (6-7). The Lyogen SMKI influences the *K/S* value and light fastness; while sodium sulfate improves the exhaustion of dye with an interaction dyeing time and dyeing temperature. It could be concluded that direct dye can be used for dyeing of wool/cotton union fabric at the above optimized condition for medium shade (1 %) along with after-treatment with a cationic dye fixing agent in order to achieve good washing (4-5) and light fastness (6-7).

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