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# **Right-first-time dyeing: A design of experiments approach for the optimisation of dyeing-processes using hard water**

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**Purpose** - Owing to the persistent water scarcity for more than two decades now, the textile industry in Pakistan is forced to rely on high-mineral-content ground water for use in textile wet processing. Furthermore, the limited amount of municipal water that is at the disposal of the textile industry is also high in mineral content. Thus, on the large scale, water hardness has become an acute problem for the textile processor. In particular, in the dyeing process, water hardness is known to

have crucial effects. However, to-date, no systematic study has been conducted on this aspect of textile dyeing.

**Design/methodology/approach** - In this study,  $3^2$  full factorial design was used to optimise the dyeing conditions to achieve right-first-time dyeing in hard water. Thus cotton fabric was dyed with Red Reactive dye (of dyebath concentration at 5, 10 and 15 g/L respectively) in prepared hard water (of hardness at 10, 40 and 70 °dH) respectively. Analysis of variance, coefficient of determination ( $R^2$ ) and  $p$ -values for the models were used to evaluate the adequacy of the predictive models. The surface plots of the effects were studied to further examine the interactions of two independent variables. Derringer's desirability function was used to determine the optimum levels of each variable.

**Findings** - Three levels for both independent variables generate second-order polynomial models to predict the colour strength, lightness, red/green, yellow/blue and total colour difference values of dyed cotton. The obtained predictive models point out the considerable influence of both water hardness and dye concentration on right-first-time dyeing.

**Originality/value** - Such a finding enabled the dye-mill to produce the correct shade at water hardness of 10°dH and 15g/L dye concentration, without the need for corrective reprocessing.

**Keywords:** Right-first-time dyeing; water hardness; reactive dyeing; colorimetric properties;  $3^2$  full factorial design; optimisation

## **Introduction**

Textile sector contributes to approximately 60% of Pakistan's total exports, making it the 8<sup>th</sup> largest exporter of textiles in Asia and 12<sup>th</sup> largest exporter globally (Shah et al., 2014). Correspondingly, this sector is also the biggest water consumer making up for about

70% of local industrial water consumption. Water consumption in Pakistan's textile sector is estimated to be 50 million m<sup>3</sup> per day which is significantly higher than the international average (Kapfensteiner and Azhar, 2017). Thus, it is evident that the textile sector of Pakistan needs to improve its water-use efficiency. In addition, a scientific understanding of the effects of hard water on water intensive processes such as dyeing will help in overcoming the stated problem in a sustainable manner.

Introduced in 1970, the concept of right-first-time production in textile dyeing sector means that every batch or lot of dyed fabric should be the correct shade, every time, as assessed against the target shade, without the need for corrective reprocessing or adjustment during the process (Park and Shore, 2009; Ammayappan et al., 2016; Rasel et al., 2018). The benefits of achieving the right-first-time are very significant including for instance, reduction in water and energy consumption, reduction in effluent generation along with time and colour saving (Parton, 1994). Right-first-time dyeing is a difficult and daunting task for a dyer and requires great control over dyeing parameters such as quality of dyestuff and auxiliaries, concentration of salt and alkali, time and temperature, and water quality (Park and Shore, 2009; Rasel et al., 2018; Roy Choudhury, 2013; Khatri et al., 2015).

The hardness of water (presence of Ca<sup>+</sup> and Mg<sup>+</sup> ions; expressed in terms of German Degree Hardness) is a particularly critical factor in shade matching and it can prevent dye-houses from achieving a high level of right-first-time production (Ahmed, 2005; Jain and Mehta, 1991; Ammayappan et al., 2016; Uddin and Atiquzzaman, 2014). It is reported that presence of calcium and magnesium ions in water can cause dye precipitation and aggregation which result in shade variation, faulty dyeing and loss in colour strength (Ahmed, 2005; Tullio, 1977). Further, carbonates and hydroxides of calcium and magnesium ions precipitate as whitish deposits on the fabric surface due to

interaction with alkalis and heating during dyeing and soaping. These precipitates are sticky, adhere readily to fabric surface and results in unlevelled dyeing, white patches and poor shade matching (Anis and Eren, 2002; Hossain, 2014).

Textile sector of Pakistan is facing an acute problem of water hardness because the two main sources of water, i.e., groundwater and municipal water, both contains large amounts of mineral content (Faisal et al., 2019). Therefore, the lack of right-first-time dyeing (target shade not achieved the first time after the first attempt of dyeing) are very frequent and leads to the necessity of corrective reprocessing such as re-dyeing or stripping which unnecessarily increases the additional consumption of resources such as dyestuff and auxiliaries, time, and also increases the cost of production (Dawson, 2012; Collishaw et al., 1993). Corrective reprocessing also consumes additional water, energy and generates more effluent (Ozturk and Cinperi, 2018; Roy Choudhury, 2013; Dawson, 2012; Collishaw et al., 1993).

Amongst many classes of textile dyestuffs, the reactive dyes contribute about 50% of the total market share due to their wide-ranging shade gamut, flexibility in application, and the outstanding fastness properties (Lewis, 2014). They are widely used in coloration of cotton, silk, wool and regenerated cellulosic fibres (Khatri et al., 2015; Khatri et al., 2011).

The effects of water hardness on right-shade-first-time when dyeing with reactive dyes is well established and extensively studied (Jain and Mehta, 1991; Kan, 2008; Shinde et al., 2015; Rahman et al., 2016; Khalil and Sarkar, 2014; Chapatwala et al., 1994; Ishtiaque et al., 2000; Sampath, 2001; Faisal et al., 2019). However, to our knowledge, no previous work has reported systematic optimisation of dyeing-process for right-first-time dyeing in hard water. Thus, the main aim of the present study is to devise an approach for right-first-time dyeing in hard water – a situation which could

not be avoided in certain scenarios as discussed in the preceding text. In the present work, 3<sup>2</sup> full factorial design was employed to design experiments in an attempt to systematically understand the quantitative effects of water hardness and concentration of dye on shade variation of the dyed fabrics and optimise such dyeing process parameters.

## **Experimental**

### **Materials and reagents**

Magnesium sulphate heptahydrate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) and anhydrous calcium chloride ( $\text{CaCl}_2$ ) were purchased from Merck (Germany) and used as received. Drimaren Red HF-CD (CI number confidential, unknown), Solidokol NM and Revatol S (mild oxidising agent) were kindly provided by Archroma (Pakistan). Sodium bicarbonate, sodium hydroxide (36°Be), sodium chloride and soda ash of commercial grades were also provided by Archroma (Pakistan). Scoured, bleached and mercerised 100% cotton fabric (130 g/m<sup>2</sup>; plain weave) supplied by Lucky Textile Mills was used in this study.

### **Preparation of Stock Solution**

Stock Solution A was prepared by dissolving 39g of anhydrous calcium chloride ( $\text{CaCl}_2$ ) in distilled water and made up to a volume of 1000 mL to get 13554 ppm of stock solution of calcium. Likewise, Stock solution B was prepared by dissolving 43.93g of magnesium sulphate heptahydrate ( $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ ) in distilled water and made up to a volume of 1000 mL to get 4148 ppm of stock solution of magnesium.

### **Preparation of Standard Hard Water**

100g of solution A, 50g of solution B and 1.24g of sodium bicarbonate was dissolved in distilled water and to made up to a volume of 10 L. This provides water with a hardness

of 15°dH. The dilution to obtain water of various hardness was calculated by following Equation 1;

$$c_1 v_1 = c_2 v_2 \quad (1)$$

Where  $c_1$  is concentration of the prepared standard hard water;  $v_1$  is the volume of the standard hard water;  $c_2$  is the required concentration of hard water and  $v_2$  is the final volume of required hard water. The hardness of the prepared water was checked by titrating it against standard 0.01M EDTA solution and adjusted if required.

### **Dyeing**

Dyeing and rinsing of fabric samples was conducted with hard water dyebath and hard water respectively. Cotton fabric samples were padded with a pad liquor containing 5, 10 and 15 g/L of Drimarene Red HF-CD, respectively, and 10 g/L of solidokol NM. After drying at 120°C, the pre-padded fabric samples were padded again with pad liquor containing 250 g/L of sodium chloride, 20 mL/L of sodium hydroxide 36 °Be, 20 g/L of soda ash and 10 mL/L of Revatol S, followed by steaming at 102 °C for 60s. The fabric samples were then rinsed with cold water, soaped using 2 g/L Ladipur RSK at boil for 10 min, and finally rinsed with cold water until no further colour bleed and then dried under laboratory conditions.

### **Colour Measurements of Dyed Fabric**

The colorimetric data (%R, CIELAB coordinates) of the dyed fabric samples was measured using Datacolor spectrophotometer 650 using 10° observer with a D65 illuminant. Each dyed fabric sample was folded to four layers and measurements were made at five different locations. Total colour difference ( $\Delta E$ ) was calculated on the basis of measured CIELAB coordinates using Equation 2;

$$\Delta E = \sqrt{(L^* - L_0)^2 + (a^* - a_0)^2 + (b^* - b_0)^2} \quad (2)$$

Where  $L^*$  denotes lightness,  $a^*$  denotes the red/green value and  $b^*$  denotes the yellow/blue value of the measured fabric samples dyed in hard water and  $L_0$ ,  $a_0$  and  $b_0$  are the lightness, the red/green and the yellow/blue values of the target fabric samples dyed in distilled water using the same recipe.

The corresponding colour strength (K/S) value was calculated at  $\lambda_{\max}$  using Equation 3;

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

Where K is the absorption coefficient, S is the scattering coefficient and R is the reflectance of the dye fabric sample.

### Experimental Design

On the basis of the authors' previous work (Faisal et al., 2019), a two factors – three levels ( $3^2$ ) full factorial design was employed to study, predict and optimise the influence of process variables, such as water hardness (10, 40 and 70 g/L) and dye concentration (5, 10 and 15 g/L), on the colour strength and colorimetric properties of dyed cotton samples. The water hardness ( $x_1$ ) and dye concentration ( $x_2$ ) were chosen as the critical independent variables. The low, intermediate, and high levels of each variable were designated as -1, 0, and +1, and given in Table 1. The design of experiments and observed responses data are summarised in Table 2.

Place Table 1 here.

Place Table 2 here.

The use of three levels for each independent variable allows the generation of a second-order polynomial equation to correlate the predictors and the responses, which encompass linear, quadratic and interactive effects of the process variables (Ishtiaque et al., 2000; Tronci, 2017; Tronci et al., 2018). All of the statistical analyses were performed



by means of Minitab 17 statistical software tool. The generalised form of the second-order polynomial equation that was employed is given in Equation 4 (Montgomery, 2017):

$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i < j} \beta_{ij} x_i x_j \quad (4)$$

Where  $y$  is the response variable;  $x_i$  and  $x_j$  are the independent variables affecting the response;  $\beta_0$ ,  $\beta_i$ ,  $\beta_{ii}$  and  $\beta_{ij}$  are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively.

## Results and Discussion

### Mathematical Modelling

The results obtained from the 3<sup>2</sup> full factorial experiments for dyeing process were evaluated by multiple regression analysis. A relationship between the response and predictors has been expressed by a second-order polynomial equation which will help to predict the colour strength and colorimetric properties ( $L^*a^*b^*$  and  $\Delta E$ ) of the dyed fabric in different sets of combinations. Five predictive models were generated to understand and predict the interactive correlation between each response and the predictors ( $x_1$  and  $x_2$ ). The final models obtained in terms of coded variables are given in Equations 5, 6, 7, 8 and 9;

$$K/S = 3.533 - 0.090x_1 + 1.719x_2 - 0.161x_1^2 + 0.211x_2^2 - 0.068x_1x_2 \quad (5)$$

$$L^* = 52.002 + 0.848x_1 - 6.260x_2 + 0.262x_1^2 + 1.217x_2^2 - 0.530x_1x_2 \quad (6)$$

$$a^* = 42.162 - 0.847x_1 + 4.958x_2 - 0.243x_1^2 - 0.888x_2^2 - 0.528x_1x_2 \quad (7)$$

$$b^* = -11.458 - 0.352x_1 + 0.214x_2 - 0.103x_1^2 + 0.444x_2^2 + 0.130x_1x_2 \quad (8)$$

$$\Delta E = 1.561 + 0.810x_1 - 0.843x_2 - 0.261x_1^2 + 0.875x_2^2 + 0.345x_1x_2 \quad (9)$$

### **Adequacy of Predictive Models**

The adequacy of the predictive models was assessed by analysis of variance (ANOVA), coefficient of determination ( $R^2$ ) and  $p$ -values for the model and the results are shown in Table 3. From Table 3, it is observed that, under 95 % confidence levels, the significant model terms for K/S was  $x_2$ ; for  $L^*$  value  $x_1$ ,  $x_2$  and  $x_2^2$ ; for  $a^*$  and  $\Delta E$  value  $x_1$  and  $x_2$ . In case of  $R^2$ , it was 0.9950, 0.9995, 0.9941 and 0.9410 for K/S,  $L^*$ ,  $a^*$  and  $\Delta E$ , respectively, which indicates that these models fit the experimental data very well. From Table 3, it is also evident that the  $p$ -value of predictive models was  $p < 0.001$  for K/S,  $L^*$  and  $a^*$  values and  $p < 0.05$  for  $\Delta E$ , which implied that these models were significant at the 95% confidence level. However, predictive model for  $b^*$  value was not statistically significant ( $p > 0.05$ , data not shown).

Place Table 3 here.

### **Effect of Independent Variables on Responses**

The regression coefficients of the predictive models and their statistical significance values are presented in Table 4. In addition, 3D surface plots were constructed using developed models (Equations 5–9) to examine the relationship between each response ( $K/S$ ,  $L^*$ ,  $a^*$ ,  $b^*$  and  $\Delta E$  respectively) and the two independent variables, which are shown in Figure 1.

#### *Effect on Colour Strength (K/S)*

Higher K/S value indicates greater depth of the colour of dyed fabric surface (Yuen et al., 2004). According to Table 4, water hardness had a negative effect on its linear and quadratic terms whereas the concentration of dye significantly ( $p < 0.05$ ) increased the colour strength in a linear manner. The interactive effects between water hardness and concentration of dye showed a negative effect on colour strength. It is clearly indicated (Figure 1a) that the colour strength decreases with the increasing hardness and increases

as the concentration of dye increases. The effect of the water hardness can be attributed to the presence of elevated amount of calcium and magnesium ions in the dye liquor that will increase the potential barrier at the interface between cotton fibre and dye liquor (Kan, 2008). This increase in potential barrier will resist dye anions passing the interface to enter the fabric surface, resulting in a decrease in dye absorption and hence decrease in colour strength. The decrease in colour strength at higher water hardness level is also attributed to the presence of precipitates of calcium carbonate and magnesium hydroxide. It was reported earlier (Jain and Mehta, 1991) that reactive dyes have strong affinity for these precipitates and are responsible for loss in colour strength.

Place Table 4 here.

Place Figure 1 here.

#### *Effect of Independent Variables on Colorimetric Properties*

In CIELAB colour space,  $L^*$  is an approximate measurement of lightness (Becerir, 2011). The higher the  $L^*$  value the lighter the shade. The results in Table 4 clearly show that both water hardness and concentration of dye significantly ( $p < 0.05$ ) affected the lightness value. Thus, water hardness showed a positive effect on its linear and quadratic terms and concentration of dye affected the lightness negatively in a linear and positively in quadratic manner showing a curvilinear decrease in  $L^*$  value. The interactive effects between water hardness and concentration of dye showed a negative effect. As shown in Figure 1b, it is clear that the  $L^*$  value of dyed samples increases with increasing water hardness and decreases with increasing dye concentration. The  $L^*$  values showed similar relationship to the colour strength indicating that as the water hardness increases the dyed samples become lighter. This increase in  $L^*$  value of dyed samples at higher water hardness could be attributed to the presence of whitish

precipitates or chalky stains of calcium carbonate and magnesium hydroxide on surface of dyed samples (Shinde et al., 2015; Saleem and Amin, 2017).

In CIELAB colour space, positive  $a^*$  value depicts shift towards red shade and negative value shows shift towards green shade (Becerir, 2011). It can be seen from Table 4 that water hardness exhibit strongly negative and concentration of dye exhibit strongly positive effect on the  $a^*$  value of dyed samples. It can be seen from Figure 1c that  $a^*$  value of dyed samples decreases (shifts toward green shade) with increasing water hardness indicating that the dyed samples become greener in shade. Moreover, with increasing dye concentration the  $a^*$  value increases which signifies that dyed samples become redder in shade.

In CIELAB colour space, positive  $b^*$  value illustrates shift towards yellow shade and negative value elucidates shift towards blue shade. It can be observed from Table 4 that neither water hardness and nor concentration of dye have a significant ( $p>0.05$ ) effect on the  $b^*$  value of the dyed samples. However, water hardness showed a negative effect on its linear and quadratic terms whereas concentration of dye showed a positive effect in its linear and quadratic terms. The interaction term of water hardness and concentration of dye showed a positive effect. From Figure 1d, it is apparent that  $b^*$  value decreases with increasing water hardness indicating that the samples become bluer or less yellow in shade. The results of CIELAB colour coordinates suggests that as the water hardness increases the dyed samples appeared lighter, greener (less red), and bluer (less yellower) because calcium and magnesium ions stimulates the formation of an insoluble complexes which causes shade variation.

The colour difference ( $\Delta E$ ) takes into account the  $L^*$ ,  $a^*$  and  $b^*$  values of the target and the dyed samples. It is generally used as a pass or fail criterion for assessing shade and a  $\Delta E$  value of 1 is considered to hinder the prospect of a guaranteed right-first-time

dyeing process (Parton, 1994). From Table 4 it can be seen that both water hardness and concentration of dye have significant ( $p < 0.05$ ) effect on the colour difference of the dyed samples. It was also observed that water hardness showed a positive effect on its linear term and a negative effect on its quadratic term whereas concentration of dye showed a negative effect in its linear term and a positive effect on its quadratic term. The interaction terms of both water hardness and concentration of dye showed a positive effect. Also,  $\Delta E$  was significant among the hardness levels and dye concentrations trailed. In addition, Figure 1e shows that higher  $\Delta E$  values were recorded for higher water hardness level and lower dye concentration, as the results of the changes in  $L^*$ ,  $a^*$  and  $b^*$  values of the dyed samples.

### **Simultaneous Optimization of Colour Strength and Total Colour Difference**

Attempts to obtain a set of an optimal conditions to achieve both maximum colour strength (K/S) and minimum total colour change ( $\Delta E$ ) followed. For usual practice, target values are identified based on literature or common industrial practice and used to set the optimisation process. However, in this study, the predictive models previously found for colour strength and  $\Delta E$  were employed to obtain specific optimum conditions. Simultaneous optimisations of the responses are commonly performed by means of Desirability Functions (Costa et al., 2011). Among all the proposed methods, the authors chose to carry out the optimisation using the Derringer's method (Derringer and Suich, 1980), due to its good reliability and the requirement of substantial mathematical and statistical expertise to implement. This method searches for the values of independent variables that simultaneously satisfy the pre-set requirements according to the chosen predictive models, through the evaluation of a specific (weighted) combination of colour strength and  $\Delta E$ 's equations. Finally, the optimal conditions will result from the pair of values of dye concentration and water hardness that maximises the Desirability

Function. Using the  $3^2$  factorial results, the optimum level of parameters was obtained which indicated that water hardness of -1 level i.e. 10 °dH and a dye concentration of +1 level i.e. 15 g/L giving a K/S value of 5.46 and a  $\Delta E$  value of 0.699 with an overall desirability value of 0.8709 (Figure 2). This combination of optimised values could be considered, in this specific case under investigation, as the optimum and feasible and taken in account in further studies to be validated as best conditions for right-first-time dyeing in hard water.

Place Figure 2 here

## **Conclusion**

In this research, the influence of water hardness and the dye concentration of the dyebath on right-first-time production have been studied through a set of  $3^2$  full factorial design. As a result of the analysis and interpretation of the experimental data obtained, second-order equations as mathematical models were developed with a particular attention to their fitting performances. These models were later employed in the prediction and simultaneous optimisation of the conditions of dyeing-process in hard water by means of the Desirability Function. By focusing on  $R^2$ , which were found to exhibit more than 95% confidence level of the models, the efficiency of the second-order equations in the description of the relationships between the independent variables and the responses has been demonstrated. In particular, the analysis of variance presented extremely high  $R^2$  of 0.9950, 0.9995, 0.9941 and 0.9410 for colour strength,  $*L$ ,  $a^*$  and  $\Delta E$  respectively, confirming an adequate fit of the developed equations with the experimental data. Such models were used to obtain a set of optimal dyeing conditions that result in highly accurate colour matching of the dyed fabric samples with the target fabric samples. The optimum conditions were found to be 10 °dH and 15g/L for water hardness and dye concentration, respectively. The predicted values of colour strength

and  $\Delta E$ , which resulted from the combination of the optima of independent variables, were calculated to be 5.46 and 0.699 respectively. Further studies and consistent experimental campaigns will be necessary to validate this assumption, which will surely be a future development of this work.

## References

- AHMED, N. S. E. 2005. The use of sodium edate in the dyeing of cotton with reactive dyes. *Dyes and Pigments*, 65, 221-225.
- AMMAYAPPAN, L., JOSE, S. & RAJ, A. A. 2016. Sustainable production processes in textile dyeing. In: MUTHU, S. & GARDETTI, M. (eds.) *Green Fashion: Environmental Footprints and Eco-design of Products and Processes*. Singapore: Springer.
- ANIS, P. & EREN, H. A. 2002. Comparison of alkaline scouring of cotton vs. alkaline pectinase preparation. *AATCC Review*, 2, 22-26.
- BECERIR, B. 2011. Assessment of the results of different color difference formulae under different illuminants by wash fastness tests. *Fibers and Polymers*, 12, 946-956.
- CHAPATWALA, M. N., KAPADIA, P. J. & GANDHI, R. S. 1994. Effect of water hardness and total dissolved solids on dyeing of polyester and cationic dyeable polyester. *Colourage*, 41, 29-41.
- COLLISHAW, P. S., PHILLIPS, D. A. S. & BRADBURY, M. J. 1993. Controlled coloration: a success strategy for the dyeing of cellulosic fibres with reactive dyes. *Journal of the Society of Dyers and Colourists*, 109, 284-292.
- COSTA, N. R., LOURENÇO, J. & PEREIRA, Z. L. 2011. Desirability function approach: a review and performance evaluation in adverse conditions. *Chemometrics and Intelligent Laboratory System*, 107, 234-244.
- DAWSON, T. 2012. Progress towards a greener textile industry. *Coloration Technology*, 128, 1-8.
- DERRINGER, G. & SUICH, R. 1980. Simultaneous optimization of several response variables. *Journal of Quality Technology*, 12, 214-219.
- FAISAL, S., FAROOQ, S., HUSSAIN, G. & BASHIR, E. 2019. Influence of Hard Water on Solubility and Colorimetric Properties of Reactive Dyes. *AATCC Journal of Research*, 6, 1-6.
- HOSSAIN, M. 2014. Investigation into cotton knit dyeing with reactive dyes to achieve right first time (RFT) shade.

- ISHTIAQUE, S., PARMAR, M. & CHAKRABORTY, M. 2000. To study the structural behaviour of natural coloured cotton and its interaction with different chemicals. *Colourage*, 47, 15-24.
- JAIN, A. K. & MEHTA, K. S. 1991. Control water hardness to minimize colour matching variations. *ATIRA Communication on Textiles*, 25, 105-111.
- KAN, C. W. 2008. Influence of water hardness on acid dyeing with silk. *Fibers and Polymers*, 9, 317-322.
- KAPFENSTEINER, C. & AZHAR, M. 2017. Water Efficiency in the Textile Industry. Faisalabad: Ministry of Textile Industry
- KHALIL, E. & SARKAR, J. 2014. Effect of hardness of water on fixation and total wash off percentage of reactive dyes when applied to cellulosic fiber. *International Journal of Scientific and Research Publications*, 4, 73-76.
- KHATRI, A., PEERZADA, M. H., MOHSIN, M. & WHITE, M. 2015. A review on developments in dyeing cotton fabrics with reactive dyes for reducing effluent pollution. *Journal of Cleaner Production*, 87, 50-57.
- KHATRI, Z., MEMON, M. H., KHATRI, A. & TANWARI, A. 2011. Cold Pad-Batch dyeing method for cotton fabric dyeing with reactive dyes using ultrasonic energy. *Ultrasonics Sonochemistry*, 18, 1301-1307.
- LEWIS, D. M. 2014. Developments in the chemistry of reactive dyes and their application processes. *Coloration Technology*, 130, 382-412.
- MONTGOMERY, D. C. 2017. *Design and analysis of experiments*, New Jersey, John Wiley & Sons.
- OZTURK, E. & CINPERI, N. C. 2018. Water efficiency and wastewater reduction in an integrated woolen textile mill. *Journal of Cleaner Production*, 201, 686-696.
- PARK, J. & SHORE, J. 2009. Evolution of right-first-time dyeing production. *Coloration Technology*, 125, 133-140.
- PARTON, K. 1994. Right-first-time dyeing - the dye manufacturer's role. *Journal of the Society of Dyers and Colourists*, 110, 4-5.
- RAHMAN, M., ISLAM, A. & BISWAS, J. 2016. Effects of Water Hardness on Dyeing of Cotton Fabrics with Different Types of Reactive Dyes and Shade Percentages. *International Journal of Materials Science and Applications*, 5, 254-260.
- RASEL, M., AHMED, M. T., HASAN, M., ULLAH, A., ABIR, H. R., HAQUE, K. M. H. & BHUIYAN, M. N. H. 2018. A Sustainable Approach to Meeting the Quality of Product in Textile Dyeing Industry; Right First Time (RFT). *American Journal of Chemistry and Material Science*, 5, 78-84.
- ROY CHOUDHURY, A. K. 2013. Green chemistry and the textile industry. *Textile Progress*, 45, 3-143.



- SALEEM, F. & AMIN, S. 2017. Study of Cotton Fabric Dyeing by Reactive Dyes in Various Water Hardness Systems. *Journal of Chemical Society of Pakistan*, 39, 6-10.
- SAMPATH, M. R. 2001. Frequently encountered problems in textile wet processing and a diagnostic approach for prevention/solutions. *Colourage*, 48, 58-62.
- SHAH, S. A. S., SYED, A. & SHAIKH, F. M. 2014. Impact of Textile Industry on Pakistan Economy. *Romanian Statistical Review Supplement*, 62, 43-59.
- SHINDE, T., MARATHE, R. & DORUGADE, V. A. 2015. Effect of water hardness on reactive dyeing of cotton. *International Journal on Textile Engineering and Processes*, 1, 27-34.
- TRONCI, A. 2017. Optimization of dyeing fixing process on silk fabrics through DOE analysis. *Journal of Natural Fibers*, 14, 736-746.
- TRONCI, A., ORRÙ, P. F. & BUONADONNA, P. 2018. Product quality and energy consumption optimisation of dyeing fixing process by steaming through DOE analysis: a cotton case study. *International Journal of Management and Decision Making*, 17, 467-487.
- TULLIO, V. 1977. *Hard water-tolerant dye solutions*.
- UDDIN, M. G. & ATIQUZZAMAN, A. S. M. 2014. Estimation of Total Hardness of Bath Water in Knit Dye Houses in Bangladesh and Study of Its Effects. *International Journal of Textile Science*, 3, 59-63.
- YUEN, C. W. M., KU, S. K. A., CHOI, P. S. R. & KAN, C. W. 2004. Study of the factors influencing colour yield of an ink-jet printed cotton fabric. *Coloration Technology*, 120, 320-325.

**Table 1** Independent Variables and Levels (coded and uncoded) used for 3<sup>2</sup> Full Factorial Design

Symbol	Independent Variables	Level		
		Low (-1)	Intermediate (0)	High (1)
$x_1$	Hardness of Water (g/L)	10	40	70
$x_2$	Dye Concentration (g/L)	5	10	15

**Table 2** 3<sup>2</sup> Full Factorial Design Matrix and Observed Responses

<b>Run</b>	<b><math>x_1</math></b>	<b><math>x_2</math></b>	<b>K/S</b>	<b>L*</b>	<b>a*</b>	<b>b*</b>	<b><math>\Delta E</math></b>
<b>1</b>	-1	-1	1.94	58.29	37.31	-10.92	2.47
<b>2</b>	-1	0	3.37	51.56	43.01	-10.81	0.89
<b>3</b>	-1	1	5.50	46.83	46.20	-11.01	1.43
<b>4</b>	0	-1	2.06	59.43	36.66	-11.25	2.98
<b>5</b>	0	0	3.43	52.10	41.53	-11.93	2.07
<b>6</b>	0	1	5.53	46.91	46.52	-10.31	1.38
<b>7</b>	1	-1	1.75	61.24	34.49	-11.72	4.91
<b>8</b>	1	0	3.48	52.87	41.46	-11.84	2.25
<b>9</b>	1	1	5.04	47.66	45.49	-11.29	2.49

**Table 3** Analysis of variance (ANOVA) of the predictive models

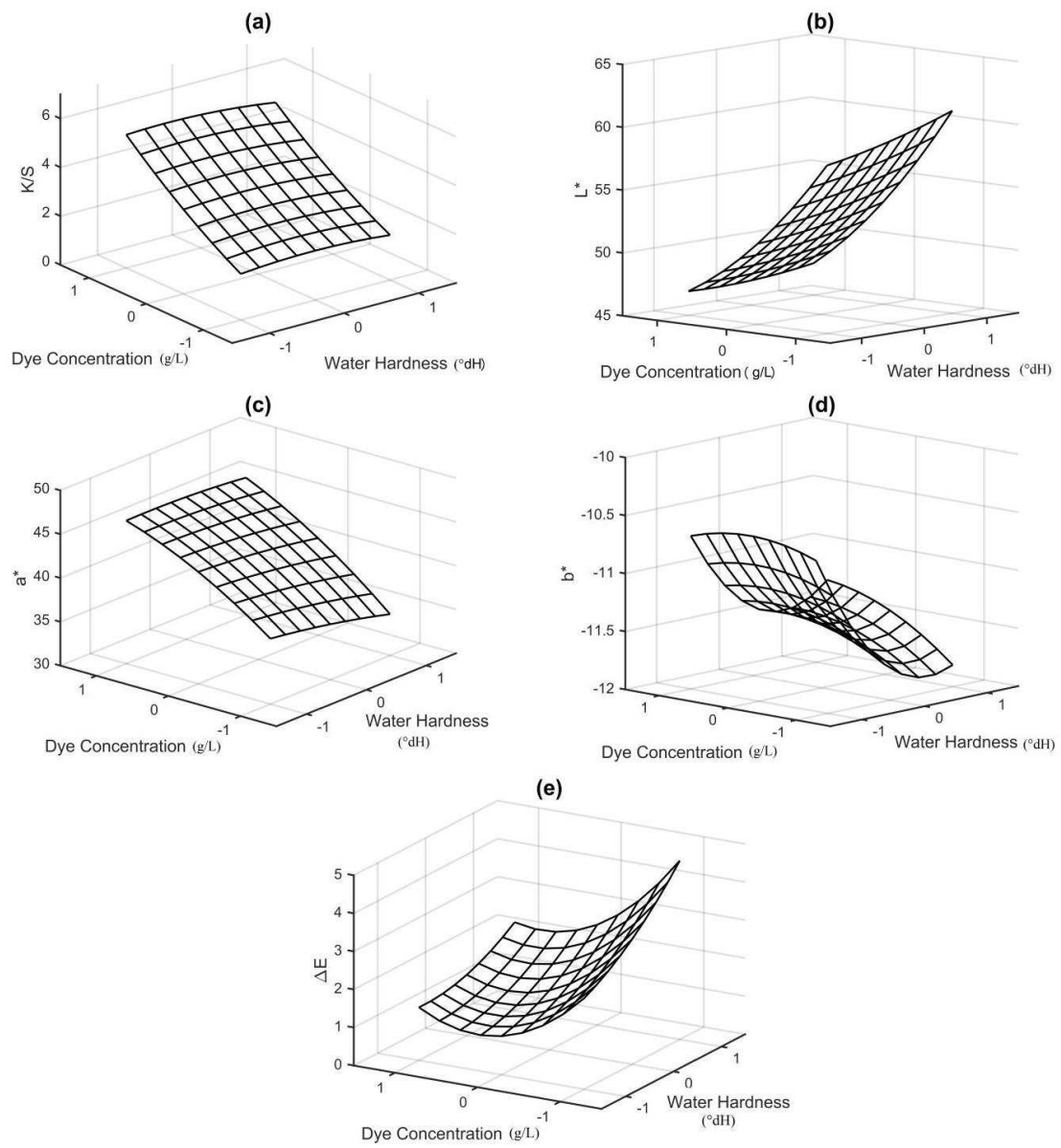
Source	DF	Adj SS	Adj MS	F-Value	P-Value	Adj SS	Adj MS	F-Value	P-Value
		<b>Colour Strength (K/S)</b>				<b>Lightness (L*) value</b>			
Regression	5	17.93	3.59	119.28	0.001 <sup>a</sup>	243.67	48.73	1093.94	0.000 <sup>a</sup>
$X_1$	1	0.05	0.05	1.62	0.292	4.32	4.32	96.93	0.002 <sup>a</sup>
$X_2$	1	17.72	17.72	589.48	0.000 <sup>a</sup>	235.13	235.13	5278.01	0.000 <sup>a</sup>
$X_{12}$	1	0.05	0.05	1.72	0.281	0.14	0.14	3.07	0.178
$X_{22}$	1	0.09	0.09	2.97	0.183	2.96	2.96	66.46	0.004 <sup>a</sup>
$X_1X_2$	1	0.02	0.02	0.60	0.494	1.12	1.12	25.22	0.015
Error	3	0.09	0.03			0.13	0.05		
Total	8								
Summary		R <sup>2</sup> = 0.9950; R <sup>2</sup> (adj) = 0.9867				R <sup>2</sup> = 0.9995; R <sup>2</sup> (adj) = 0.9985			
		<b>Red/green (a*)</b>				<b>Total Colour Difference (ΔE)</b>			
Regression	5	154.62	30.92	101.15	0.002 <sup>a</sup>	10.34	2.07	9.57	0.046 <sup>a</sup>
$X_1$	1	4.30	4.30	14.07	0.033 <sup>a</sup>	3.93	3.93	18.20	0.024 <sup>a</sup>
$X_2$	1	147.51	147.51	482.51	0.000 <sup>a</sup>	4.26	4.26	19.71	0.021 <sup>a</sup>
$X_{12}$	1	0.12	0.12	0.39	0.578	0.14	0.14	0.63	0.485
$X_{22}$	1	1.58	1.58	5.16	0.108	1.53	1.53	7.09	0.076 <sup>aa</sup>
$X_1X_2$	1	1.11	1.11	3.64	0.152	0.48	0.48	2.21	0.234
Error	3	0.92	0.31			0.65	0.22		
Total	8								
Summary		R <sup>2</sup> = 0.9941; R <sup>2</sup> (adj) = 0.9843				R <sup>2</sup> = 0.9410; R <sup>2</sup> (adj) = 0.8426			

<sup>a</sup> significant at p<0.05, <sup>aa</sup> significant at p<0.1

**Table 4** Statistical parameters of predictive models

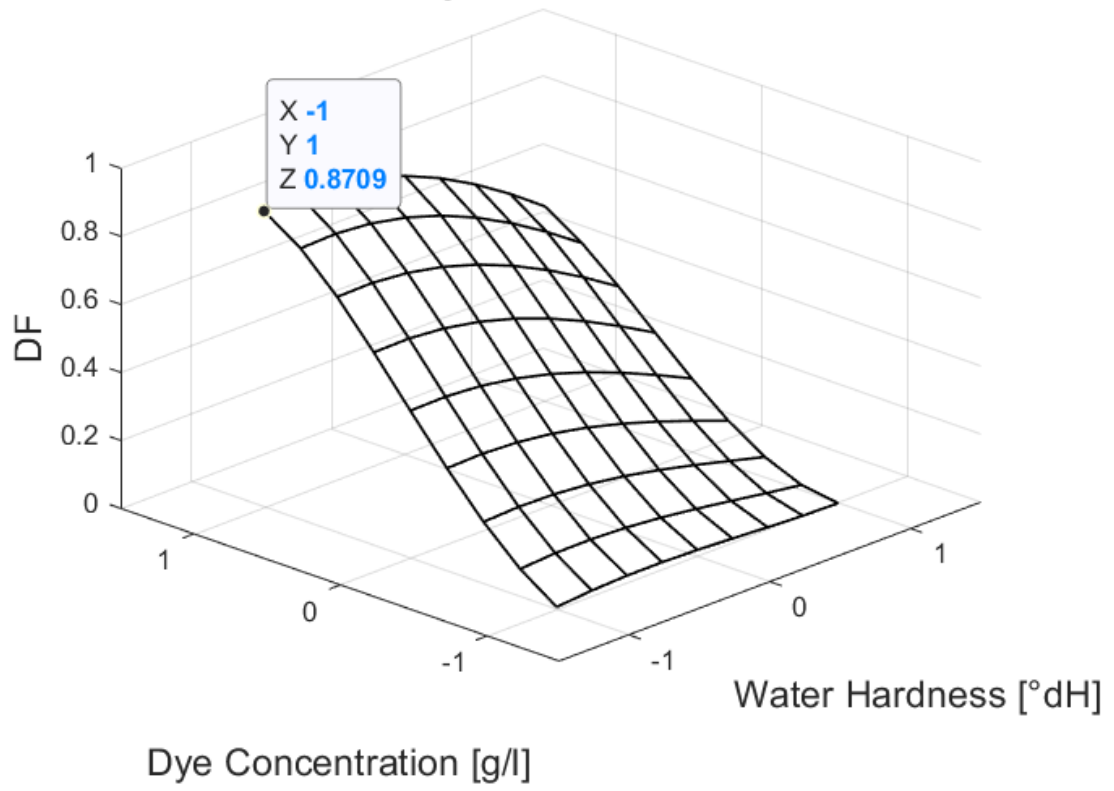
Coefficients	K/S		L*		a*		b*		ΔE	
	estimate	p-value	estimate	p-value	estimate	p-value	estimate	p-value	estimate	p-value
$\beta_0$	3.533	0.000 <sup>a</sup>	52.002	0.000 <sup>a</sup>	42.162	0.000 <sup>a</sup>	-11.458	0.484	1.561	0.020 <sup>a</sup>
$\beta_1$	-0.090	0.292	0.848	0.002 <sup>a</sup>	-0.847	0.033 <sup>a</sup>	-0.352	0.190	0.810	0.024 <sup>a</sup>
$\beta_2$	1.719	0.000 <sup>a</sup>	-6.260	0.000 <sup>a</sup>	4.958	0.000 <sup>a</sup>	0.214	0.380	-0.843	0.021 <sup>a</sup>
$\beta_{11}$	-0.161	0.281	0.262	0.178	-0.243	0.578	-0.103	0.793	0.261	0.485
$\beta_{22}$	0.211	0.183	1.217	0.004 <sup>a</sup>	-0.888	0.108	0.444	0.306	0.875	0.076 <sup>aa</sup>
$\beta_{12}$	-0.067	0.494	-0.530	0.015	0.528	0.152	0.130	0.646	-0.345	0.234

<sup>a</sup> significant at  $p < 0.05$ , <sup>aa</sup> significant at  $p < 0.1$ ;  $\beta_0$  is intercept;  $\beta_1$  and  $\beta_2$  are linear regression coefficients for  $x_1$  and  $x_2$ ;  $\beta_{11}$  and  $\beta_{22}$  are quadratic regression coefficients for  $x_1^2$  and  $x_2^2$ ;  $\beta_{12}$  is regression coefficient between  $x_1x_2$



**Figure 1**

## Desirability Function - Overall



**Figure 2**