Process Maximization of Salt Free Reactive Dyeing on Cotton using Taguchi Approach

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This study optimized the salt free reactive dyeing process using the Taguchi approach. Dyeing of cotton fabric with reactive dyes is popular because of its bright and brilliant color in various shade ranges. Cationization with ALBAFIX-WFF and the dyeing process on cotton fabric was carried out using the exhaust method. To determine the optimum process conditions, two types of multiple characteristic parameters, including the single characteristic value conversion method and the process maximization method, were used on the basis of color strength (*K*/*S*) and wash fastness. The single characteristic value conversion method confirmed that the optimum process condition was a cationization temperature of 40 °C and a dyeing pH of 11. Most importantly, the optimal conditions were confirmed by the process maximization method as a concentration of ALBAFIX-WFF 30 g/L, cationization temperature at 80 °C, dyeing pH 12, and material-to-liquor ratio (M:L) of 1:5. More suitable dyeing properties are also achieved by the process maximization method.

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INTRODUCTION

Reactive dyes are the most popular dyestuffs used for cotton dyeing. A large amount of salt is required to achieve higher exhaustion of the reactive dye from the dye bath onto the fiber (Broadbent 2001). To overcome the repulsion forces occurring between the negative charged fibers and the dye molecules, electrolytes are needed for the dyeing process. However, the resulting discharge of salt has a harmful effect on the environment (Shore 2002). Recently, there has been improvement of the dye-ability on cotton fabric without salt. Pre-treatment with a cationic agent on cotton fabric has been studied as an alternative approach instead of using an electrolyte (salt) (Buschle-Diller and Zeronian 1992; Montazer 2007). Primary and secondary hydroxyl groups of cotton can actively participate in the chemical modification (Chattopadhyay *et al.* 2007; Montazer 2007; Choudhury 2014). Cotton fabric was cationized using CIBAFIX-WFF (a polyamino chlorohydrin quaternary ammonium compound) (Kannan 2006; Sanjit 2014). Salt-free reactive dyeing was carried out by cationization of cotton (Hauser and Tabba 2001; EI-Shishtawy and Nassar 2002; Montazer 2007) and Ramie fiber with 3-chloro-2-hydroxypropyltrimethylammonium chloride (CHPTAC) (Liu *et al*. 2007). In addition, cationization of jute fabrics by ALBAFIX-WFF (poly-diallyl dimethyl ammonium chloride) and sodium hydroxide was found to improve the dye-ability of reactive dye (Arju *et al*. 2014). The cationization of cotton fabric using chitosan (Bhuiyan *et al*. 2014), Kemifix REA, Optifix F, and Optifix RSL (Mustafa Tutak *et al*. 2010), Solfix-E (polyaminochlorohydrin quaternary ammonium salt with epoxide functionality), and polyacryloxyethyltrimethylammonium chloride (PAOTAC) was used as a cationic agent for salt-free reactive dyeing (Lewis and McIlroy 1997; Teng *et al*. 2011). The cationized cotton substrate would be a suitable starting material of eco-friendly dyeing process for reducing the pollution load in terms of salinity and color discharge from the textile dyeing industries (Khatri *et al*. 2015; Arivithamani and Giri Dev 2017). However, the addition of NaOH for cationization caused the pH to rise to a very high level; subsequent neutralizing of the pH in wastewater treatments results in *in-situ* production of salt (Novak *et al*. 1998).

The Taguchi method was used to find out the optimum condition for Digital Textile Printing Process (Jung *et al*. 2016). The Taguchi method was used to find sewing conditions that minimize the seam pucker (Park and Young 2005) and maximize the delamination strength of fusible interlinings (Yoon *et al*. 2010). Dyeing process optimization and color strength prediction for viscose/Lycra blended knitted fabrics was measured by the Taguchi Method (Hossain *et al*. 2016). Optimization of the dyeing process of cotton knit fabric and reduction of the re-dyeing process was analyzed by the Taguchi method and ANOVA (Analysis of variance) (Wahyudin *et al*. 2017). Optimization of chemical coagulation of real textile wastewater was investigated by the Taguchi method (Gokkus *et al*. 2012; Swapnil *et al*. 2015). Kuo and Lin (2019) used the Taguchi method and fuzzy theory to find optimum processing parameters for sueding fabric comfort. Investigating the effect of temperature, heating time, concentration, and particle size on the improved gel spinning process of ultra-high molecular weight polyethylene (UHMWPE) was measured using the Taguchi method (Rajput *et al*. 2018). Analysis for optimization of coating process conditions for denim fabrics (Üstüntağ *et al*. 2020), the bursting strength of knitted fabrics (Mavruz and Ogulata 2010), and optimization of concrete strengthened with polymer after high temperature (Mavruz and Ogulata 2010) were also studied by the Taguchi method. The Taguchi method was used for design optimization of cutting parameters (Yang *et al*. 1998) and optimization of end milling parameters (Ghani *et al*. 2004).

The present studies were related to cotton fabric treated with cationizing agent in order to enhance the use of salt-free for reactive dyeing. The resulting performance was evaluated by color strength, exhaustion, fixation, and fastness properties. In the present optimized system, it might be assumed that NaOH is playing the role in increasing the ionic strength of the aqueous system as a means of suppressing electrostatic repulsions. However, there has been no research conduct to analyze the optimum condition by the help of Taguchi approach based on two characteristics, such as color strength and wash fastness. The multiple characteristic values are converted by statistical analysis into a single characteristic value and process maximization method. In addition, the most suitable statistical value was analyzed by the performance of dyeing properties.

EXPERIMENTAL

Materials

Cotton (1 × 1 single jersey, GSM 150, scoured and bleached) fabric was used for the experiment. AlBAFIX-WFF (supplied by HUNTSMAN) was applied as a cationizing agent for cationization of cotton fabric. Novacron Ruby S-3B (supplied by HUNTSMAN) reactive dye (2% shade) was used for dyeing the fabric.

Cationization

Cotton fabric was treated with ALBAFIX-WFF (20/30/40 g/L) along with NaOH (20 g/L) at a temperature of 40 °C/60 °C/80 °C for 20 min. The material-to-liquor ratio (M:L) (*w:v*) ratio was taken as 1:10, and the treatment was carried out in exhaust method. After cationization, fabric was washed and dried in open air for 24 h.

Dyeing

Cotton fabric was dyed with 2% (owf) Reactive dye (Novacron Ruby S-3B), 2 g/L wetting agent, 1 g/L leveling agent, and the required amount of soda wash (Na_2CO_3) to maintain the specified pH. Dyeing was carried out in the exhaust method at 60 °C for 60 min. The pH was set to either10, 11, or 12, and the M:L ratio (*w:v*) was 1:5, 1:10, and 1:15, respectively. After dyeing, the fabric was washed with cold water (room temperature) then hot water (70 °C) and again cold water. Finally, the sample was dried in an oven dryer.

Measurement of Color

The color strength (*k/s*) values of the dyed cotton fabrics were analyzed using a spectrophotometer (Datacolor 650, USA, standard light D_{65}; 10° standard observer, specular component included). The color strength (*k*/*s*) value was calculated from the sample reflectivity (*R*), as follows,

$$\frac{k}{s} = \frac{(1-R)^2}{2R} \tag{1}$$

where *k* is the absorption coefficient, *s* is the scattering coefficient, and *R* is the reflectivity (McDonald *et al* 1997).

Measurement of Color Fastness

Color fastness to wash was evaluated according to ISO 105-C06 (A2S). In addition, color staining was assessed with the help of Grey Scale and Light Box (D_{65} light source).

Experimental Design

Several factors are known to affect the color strength (*K*/*S*) value and wash fastness. Four important controllable factors were chosen in this study: ALBAFIX-WFF (A), cationization temperature (B), dyeing pH (C), and material and liquor ratio (M:L) (*w:v*) of dyeing (D). Their levels are as shown in Table 1. The orthogonal array selection rules are given in Table 2. The L^9 (3^4) orthogonal array is shown in Table 3. Each experiment was repeated three times.

Table 1. Different Factors and Levels

Factor		Concentration of ALBAFIX-WFF(g/L) A	Cationizing Temperature (°C) B	Dyeing pH C	Dyeing M:L Ratio D
Level	1	20	40℃	10	1:5
	2	30	60℃	11	1:10
	3	40	80℃	12	1:15

Table 2. Selection Process of Orthogonal Array

Factors		2	3	4	5	6	7	8	9	10
Levels	2	L_4	L_4	L_8	L_8	L_8	L_8	L_{12}	L_{12}	L_{12}
	3	L_9	L_9	L_9	L_{18}	L_{18}	L_{18}	L_{18}	L_{27}	L_{27}
	4	L_{16}	L_{16}	L_{16}	L_{16}	L_{32}	L_{32}	L_{32}	L_{32}	L_{32}
	5	L_{25}	L_{25}	L_{25}	L_{25}	L_{25}	L_{50}	L_{50}	L_{50}	L_{50}

Table 3. *L_9 (3^4)* Table of Orthogonal Array

Exp. No	Factors and Levels (Treatment Conditions)				Characteristics Value (Color strength/ Wash fastness)		
	A	B	C	D	y_1	y_2	y_3
1	1	1	1	1	y_{11}	y_{12}	y_{13}
2	1	2	2	2	y_{21}	y_{22}	y_{23}
3	1	3	3	3	y_{31}	y_{32}	y_{33}
4	2	1	2	3	y_{41}	y_{42}	y_{43}
5	2	2	3	1	y_{51}	y_{52}	y_{53}
6	2	3	1	2	y_{61}	y_{62}	y_{63}
7	3	1	3	2	y_{71}	y_{72}	y_{73}
8	3	2	1	3	y_{81}	y_{82}	y_{83}
9	3	3	2	1	y_{91}	y_{92}	y_{93}

Both color strength (*k*/*s*) and wash fastness values were determined. The signal to noise (S/N) ratio of each experiment was calculated using Eq. 2 (Jung *et al*. 2016).

$$\text{Signal to Noise Ratio} = -10 \log \frac{1}{n}\left(\sum_{i=0}^{n} \frac{1}{y_i^2}\right) \quad (2)$$

The number of repetitions for an experimental combination is *n*, the index number is *i*, and y_i is the characteristic value. In the Taguchi method, the S/N ratio is equivalent to the inverse of expected loss. Therefore, the expected loss is proportional to the S/N ratio. In this study, color strength (*k*/*s*) and wash fastness were chosen for characteristic value *y* and 9 S/N ratios were calculated using three characteristic values and measured three times per interval.

Process Evaluation by Dye Exhaustion, Fixation (%) and Levelness Parameter

The maximum process method was evaluated by dye exhaustion and fixation (%), which was determined by UV-Vis spectrophotometry (Cintra 2020, GBC, Australia). The percentage dye exhaustion (*E%*) and fixation (*F%)* were calculated according to the following equations

$$E\% = [1 - (A_1/A_o)] \times 100 \quad (3)$$

$$F\% = [(A_o - A_1 - A_2)/A_o)] \times 100 \quad (4)$$

Here, A_o *and* A_1 are the absorbance of the dye solution at λ_{max} before and after dyeing. A_2 is the absorbance of the dye-soaped solution with a non-ionic surfactant. The levelness of the dyed fabric was assessed using an instrumental method that was developed by Yang and Li (1993).

$$S_r(\lambda) = \sqrt{\frac{\sum_{i=1}^{n}[(\frac{k}{s})_{i,\lambda} - (\frac{\bar{k}}{s})_\lambda]^2}{(n-1)}} \quad (5)$$

$$(\frac{\bar{\boldsymbol{k}}}{\mathbf{s}})_\lambda = \frac{\mathbf{1}}{\mathbf{n}} \sum (\frac{\boldsymbol{k}}{\boldsymbol{s}})_{i,\lambda} \quad (6)$$

Here, λ is the wavelength for the measurement, *n* is the total number of measurements, and $(k/s)_{i,\lambda}$ is the *k*/*s* value of the i^{th} measurement at λ.

The levelness parameter was modified by Koh *et al.* (2001). Here, $S_r(\lambda)$ is the relative sample standard deviation of $(k/s)_\lambda$ and $V(\lambda)$ is the spectral luminous function. Thus, the unlevelness value is described as:

$$U = \sum_{400}^{700} S_r(\lambda) V(\lambda) \quad (7)$$

The levelness parameter L_{Lev} is varied to give different values, which are very similar to the gray-scale rating for color change if ($U \geq 0.3114$)

$$L_{Lev} = 1.20 \times [\,2.00 - \sum_{400}^{700} S_r(\lambda)\, V(\lambda)]\,] \quad (8)$$

And if $U < 0.3114$, then:

$$L_{Lev} = 5.0 - 1.2 \times \exp(\frac{7}{6}) \times U \quad (9)$$

RESULTS AND DISCUSSION

Optimum Condition by Taguchi Method

Color strength (*k*/*s*) and wash fastness were taken as characteristics values. Four factors and three levels were considered to find optimum conditions based on the two characteristic values of color strength (*k*/*s*) and wash fastness. In this study, optimum conditions were calculated by two methods, *i.e.* by means of single characteristics and when using the process maximization selection method.

Single Characteristics data Conversion Method

The average characteristic values and S/N ratios are shown in Tables 4, 5, and 6.

Table 4. Normalized Characteristics Value for Color Strength (*k*/*s*)

Exp. No	Characteristics Value (*k*/*s*)			Average of Characteristics value	Normalized Characteristics Value (*k*/*s*)		
	y_1	y_2	y_3		y_1	y_2	y_3
1	16.96	16.782	17.012	16.9180	1.0024	0.9919	1.0055
2	13.866	14.063	13.88	13.9363	0.9949	1.0090	0.9959
3	17.248	16.285	17.301	16.9446	1.0179	0.9610	1.0210
4	12.713	12.992	13.385	13.0300	0.9756	0.9970	1.0272
5	17.621	15.776	18.566	17.3210	1.0173	0.9108	1.0718
6	13.946	13.43	13.888	13.7546	1.0139	0.9763	1.0096
7	17.322	17.416	16.935	17.2243	1.0056	1.0111	0.9832
8	15.737	14.626	15.046	15.1363	1.0396	0.9662	0.9940
9	16.613	16.682	16.448	16.5810	1.0019	1.0060	0.9919

Normalized characteristics value was calculated using Eq. 10 (Jung *et al*. 2016).

$$\text{Normalized } y_i = \frac{y_i}{Average\ y_i} \qquad (10)$$

Likewise, the signal to noise ratio was calculated as follows:

$$S/N = -10 \log \frac{1}{n}\left(\sum_{i=0}^{n} \frac{1}{y_i^2}\right) \qquad (11)$$

Here, the number of repetitions for a multiple experiment is n, the index number is i, and y_i is the characteristic value of the i^{th} experiment.

Table 5. Normalized Characteristics Value for Wash Fastness (Staining on Cotton)

Exp. No	Characteristics Value (Wash Fastness)			Average of Characteristics value	Normalized Characteristics Value (Wash Fastness)		
	y_1	y_2	y_3		y_1	y_2	y_3
1	3	3	3	3.00	1.00	1.00	1.00
2	3.5	3	3	3.1666	1.1052	0.9473	0.9473
3	3.5	3	3.5	3.3333	1.05	0.90	1.05
4	3.5	3.5	3.5	3.50	1.00	1.00	1.00
5	3.5	3	3.5	3.3333	1.05	0.90	1.05
6	3.5	3.5	3.5	3.50	1.00	1.00	1.00
7	3	3	3	3.00	1.00	1.00	1.00
8	3.5	3.5	3	3.3333	1.05	1.05	0.90
9	3.5	3.5	3.5	3.50	1.00	1.00	1.00

Table 6. S/N Ratio of Average Characteristics Value

Exp. No	Factors and Levels (Treatment Conditions)				Characteristics Value (K/S +Wash fastness)			S/N Ratio
	A	B	C	D	y_1	y_2	y_3	
1	1	1	1	1	1.0012	0.9959	1.0027	-0.0006902
2	1	2	2	2	1.0501	0.9782	0.9716	-0.01624
3	1	3	3	3	1.0339	0.9305	1.0355	-0.03326
4	2	1	2	3	0.9878	0.9985	1.0136	-0.001745
5	2	2	3	1	1.0336	0.9054	1.0609	-0.06382
6	2	3	1	2	1.0069	0.9881	1.0048	-0.0012
7	3	1	3	2	1.0028	1.0055	0.9916	-0.00076
8	3	2	1	3	1.0448	1.0081	0.9470	-0.02186
9	3	3	2	1	1.0009	1.0030	0.9959	-0.000694

The sum of squares was calculated using Eq. 11.

$$\text{Sum of Square} = \sum_{i=1}^{n} \frac{(\text{Sum of characteristics value at level i})^2}{\text{Number of characteristics value at level } i} - \frac{\left(\sum_{i=1}^{p} y_i\right)^2}{N} \qquad (11)$$

where y_i is the characteristic value at level i, p is the number of levels, and N is the total number of characteristic values. The S/N Ratio was calculated using Eq. 12.

Each level= Average S/N Ratio at each level-Total average S/N Ratio (12)

The total average S/N Ratio was calculated using Eq. 13.

$$\text{Total average S/N Ratio} = \frac{\text{Total}\,\frac{S}{N}\,\text{Ratio}}{9} \quad (13)$$

The results of the analysis of the S/N ratio are shown in Table 7.

Table 7. Analysis of S/N Ratio for Single Characteristic Value

Factor	Level	Sum	Sum of Square	Average of S/N Ratio	Contribution	Pooling
A	1	-0.0501902	0.000331	-0.16730066	0.00005	Yes
	2	-0.066765		-0.022255	-0.00547	
	3	-0.02263		-0.00075433	0.01602	
B	1	-0.0031952	0.001692	-0.00106506	0.0157	No
	2	-0.10192		-0.0339733	-0.01719	
	3	-0.035154		-0.011718	-0.00506	
C	1	-0.0237502	0.001309	-0.00791673	0.00886	No
	2	-0.018679		-0.00622633	0.01055	
	3	-0.09784		-0.032613	-0.01583	
D	1	-0.0652042	0.00042	-0.0217347	-0.00499	Yes
	2	-0.0182		-0.006067	0.01071	
	3	-0.056865		-0.018955	-0.00217	

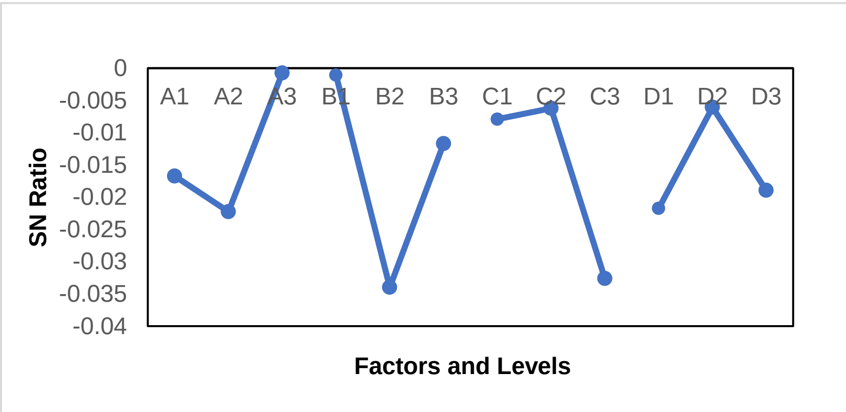

Fig. 1. Cause and effect diagram of single characteristic value for color strength and wash fastness

Factors B and C had the largest values of 0.001692 and 0.001309 by sum of squares. Factors A and D, with relatively small sum of square error, were pooled for error. The results of the F-Test are shown in Table 8. The degrees of freedom were calculated using Eq. 14.

$$\text{Degrees of freedom} = (\text{Level} - 1) \quad (14)$$

Table 8. ANOVA Results

Factor	Sum of Square (S)	Degrees of Freedom (ϕ)	Mean Square ($V=S/\phi$)	$F_0=V/V_e$	F(2,4,0.95)
B	0.0016917	2	0.0008458	4.50852	6.94
C	0.001309	2	0.0006545	3.4888	6.94
Total	0.0007506	4	0.0001876 (V_e)		

Cationization temperature and dyeing pH were considered to be the factors affecting color strength (k/s) and wash fastness. The optimum condition was B1 C2 where the S/N Ratio of each factors became the highest, with a cationization temperature of 40 ℃ and dyeing at pH 11.

Average and Confidence Interval under Optimum Condition:

The optimal S/N Ratio (SN_o) can be predicted as follows,

$$SN_o = \hat{u} + b_1 + c_2 \quad (15)$$

$$= -0.01678 + 0.0157 + 0.01055$$

$$= 0.00947$$

where û is the average S/N ratio, b_1 is the contribution of B1, and c_2 is the contribution of C2.

Difference of Expected Loss

The current condition was experiment 2 in Table 8 and its S/N ratio (SN_c) was -0.01624. The predicted S/N ratio at maximum condition (SN_o) was 0.00947. The difference of expected losses can be done using equation (16).

$$SN = -10 \log L$$

$$SN_o - SN_c = d = 0.02571$$

$$-10 \log L_0 - -10 \log L_c = d = 0.02571$$

$$\frac{L_c}{L_0} = 10^{\frac{d}{10}} \quad (16)$$

$$= 10^{\frac{0.02571}{10}}$$

$$= 1.0059$$

where L is the expected loss of characteristics value and it means the variance of characteristics value. In this case, the expected loss is 1.0059 times larger than that of optimum condition, which means that color strength (K/S) and fastness have been improved.

Process Maximization Method

Single parameter design for each characteristic value

The optimum two characteristic values have been chosen according to the single parameter design method.

Color Strength Results

S/N Ratio of Color Strength (k/s) are as represented in Table 9.

Table 9. S/N Ratio (k/s value) for Process Maximization Method

Exp. No	Factors and Levels (Treatment Conditions)				Characteristics Value (K/S Value)			S/N Ratio
	A	B	C	D	y_1	y_2	y_3	
1	1	1	1	1	16.960	16.782	17.012	24.5665
2	1	2	2	2	13.866	14.063	13.880	22.8824
3	1	3	3	3	17.248	16.285	17.301	24.5705
4	2	1	2	3	12.713	12.992	13.385	22.2930

5	2	2	3	1	17.621	15.776	18.566	24.7109
6	2	3	1	2	13.946	13.430	13.888	22.7652
7	3	1	3	2	17.322	17.416	16.935	24.7209
8	3	2	1	3	15.737	14.626	15.046	23.5886
9	3	3	2	1	16.613	16.682	16.448	24.3917

Table 10. Evaluation of S/N Ratio (K/S Value) for Process Maximization Method

Factor	Level	Sum	Sum of Square	Average of S/N Ratio	Contribution	Pooling
A	1	72.0194	1.5695	24.0064	0.1743	No
	2	69.7691		23.2563	-0.5758	
	3	72.7012		24.2337	0.4016	
B	1	71.5804	0.0531	23.8601	0.028	Yes
	2	71.1819		23.7273	-0.1048	
	3	71.7274		23.9091	0.077	
C	1	70.9203	3.4445	23.6401	-0.192	No
	2	69.5671		23.1890	-0.6431	
	3	74.0023		24.6674	0.8353	
D	1	73.6691	2.3611	24.5563	0.7242	No
	2	70.3685		23.4561	-0.376	
	3	70.4521		23.4840	-0.3481	

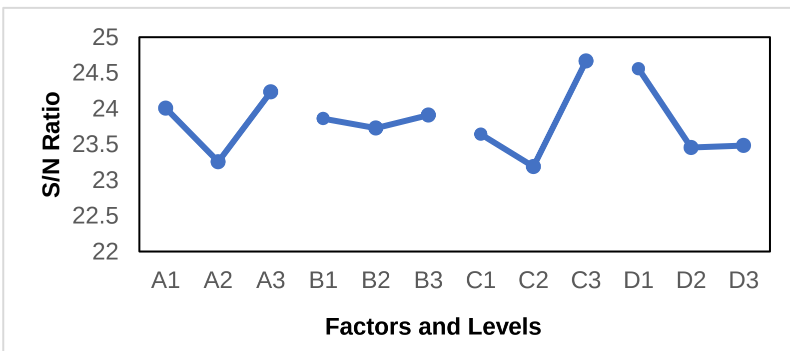

Fig. 2. Cause and effect diagram of process maximization value for color strength and wash fastness

The sum of square of factors A, C, and D had the largest value of 1.5695, 3.4445, and 2.3611, respectively. F-tests are shown in Table 11.

Table 11. ANOVA Results (K/S value)

Factor	Sum of Square (S)	Degree of Freedom (ϕ)	Mean Square (V=S/ϕ)	$F_{0=V/Ve}$	F(2,2,0.95)
A	1.5695	2	0.78475	29.5524	19
C	3.4445	2	1.72225	64.8571	19
D	2.3611	2	1.18055	44.4576	19
Error	0.0531	2	0.02655 (V_e)		
Total	7.4282	8			

Table 11 factors A, C and D could be considered to be meaningful. Concentration of ALBAFIX-WFF, dyeing pH and M: L Ratio considered to be the factors affecting highest color strength (K/S) and wash fastness. The optimum condition was A3 C3 D1 where S/N ratio of each factors becomes largest, such as Concentration of ALBAFIX-WFF 40 g/l, Dyeing pH of 12 and M: L Ratio of 1:5.

Wash Fastness Results

S/N Ratio for Wash Fastness are illustrated in Table 12.

Table 12. S/N Ratio (Wash Fastness**)**

S.L No	Factors and Levels (Treatment Conditions)				Characteristics Value (Wash Fastness)			S/N Ratio
	A	B	C	D	y_1	y_2	y_3	
1	1	1	1	1	3	3	3	9.5424
2	1	2	2	2	3.5	3	3	9.9445
3	1	3	3	3	3.5	3	3.5	10.38
4	2	1	2	3	3.5	3.5	3.5	10.8813
5	2	2	3	1	3.5	3	3.5	10.38
6	2	3	1	2	3.5	3.5	3.5	10.8813
7	3	1	3	2	3	3	3	9.5424
8	3	2	1	3	3.5	3.5	3	10.38
9	3	3	2	1	3.5	3.5	3.5	10.8813

Table 13. Results of S/N Ratio (Wash Fastness)

Factor	Level	Sum	Sum of Square	Average of S/N Ratio	Contribution	Pooling
A	1	29.86	0.875	9.9533	-0.3592	No
	2	32.14		10.7133	0.4008	
	3	30.80		10.2666	-0.0459	
B	1	29.96	0.797	9.9866	-0.3259	No
	2	31.20		10.400	0.0875	
	3	32.14		10.7133	0.4008	
C	1	30.80	0.33	10.2666	-0.0459	Yes
	2	31.70		10.5666	0.2541	
	3	30.30		10.100	-0.2125	
D	1	30.80	0.28	10.2666	-0.0459	Yes
	2	30.36		10.120	-0.1925	
	3	31.64		10.5466	0.2341	

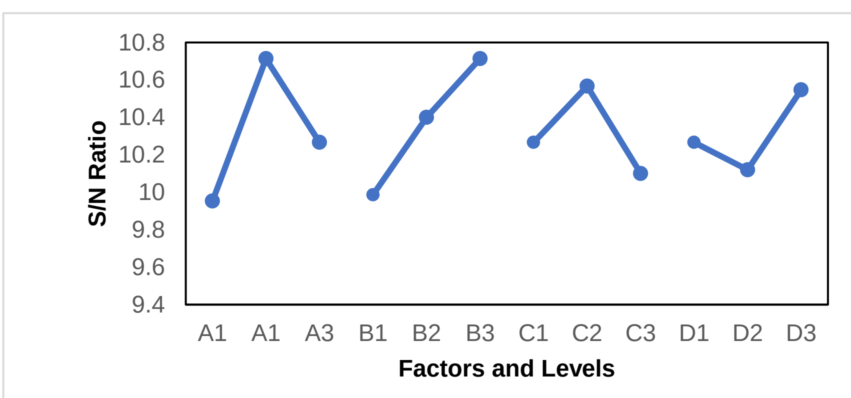

Fig. 3. Cause and effect diagram for wash fastness

The sum of square of factors A, and B had the largest value of 0.875 and 0.797. The results of the F-test are shown in Table 14.

Table 14. ANOVA Results (Wash Fastness)

Factor	Sum of Square (S)	Degree of Freedom (ϕ)	Mean Square (V=S/ϕ)	$F_{0=V/Ve}$	F(2,4,0.95)
A	0.875	2	0.4375	2.8688	6.94
B	0.797	2	0.3985	2.6131	6.94
Error	0.61	4	0.1525(V_e)		
	2.282	8			

Table 14 factors A, and B could be considered to be meaningful. The optimum condition was A2 B3 where S/N Ratio of each factors becomes largest, in other words, Concentration of ALBAFIX-WFF 30 g/l, Cationization temperature at 80. Process maximization levels of characteristics values are shown in Table 15.

Table 15. Optimum Levels of Characteristics Values

Characteristics Value	Optimum Levels
Color Strength (K/S)	A3C3D1
Wash Fastness	A2B3

As can be seen in Table 15, A is the only conflicting factor in this experiment, which had different optimum level between color strength (*k*/*s*) and wash fastness. Factors B, C, D were low conflicting factors and the optimum levels of each factor were B3, C3 and D1. S/N ratios of conflicting factor A are shown in Table 16.

Table 16. Average and Normalized S/N Ratio of Conflicting Factor

Factors	SN Ratio (K/S Value)	Average (K/S value)	S/N Ratio Wash fastness value	Average (Wash fastness)	Normalized SN Ratio	
					k/s value	Wash fastness
A1	24.0064	23.8321	9.9533	10.3110	1.0073	0.9653
A2	23.2563		10.7133		0.9758	1.0390
A3	24.2337		10.2666		1.0168	0.9956

In this case, level 2 with the maximum value of 1.0390 was selected. So, the selected optimum condition after compromise was A2 B3 C3 D1.

Comparison of Progress Effect

The progress effect of the determination of the process maximization is as shown in Table 17. The single characteristics value conversion method showed more progressive than the process maximization method.

Table 17. Comparison of Progress Effect

Method	Optimum Level	Progress Effect
Single characteristics value Conversion method	B1 C2	1.005937
Process maximization method	A2 B3 C3 D1.	0.9949

Evaluation of Dyeing quality and Dyeing Properties

The dye exhaustion, fixation, and levelness of the cationized cotton fabric dyed with Reactive Red are shown in Table 18. The process maximization method revealed higher exhaustion (80.25%) and fixation (97.98%) than the single characteristic value conversion method exhaustion (77.65%) and fixation (81.29%). This difference arises because the concentration of ALBAFIX-WFF of 30 g/l, cationization temperature at 80 °C, dyeing pH of 12, and M: L ratio of 1:5 enhanced the swelling of the fiber, which could be introduced dyestuff into the interior of the cellulosic material. However, the single characteristic value method levelness value was quite a bit higher than for the process maximization method. This can be explained based on the fact that the levelness value ensures that low dye uptake reduces dye desorption from the fabric surfaces (Kabir *et al.* 2020).

Table 18. Dyeing Properties of Different Methods

Method	Optimum Level	Exhaustion (%)	Fixation (%)	Levelness (L)
Single characteristics value Conversion method	A2B1 C2D1	77.65	81.29	4.93
Process maximization method	A2 B3 C3 D1.	80.25	97.98	4.92

Dyeing Mechanism of Cationized Cotton Fabric

The dyeing mechanism of reactive dye in a cationized cotton is illustrated in Fig. 4. It was clearly found that most of the reactive dyes were easily absorbed and diffused into the cationized cotton due to the presence of nucleophilic cationic sites, which attract the oppositely charged anionic reactive dyes by electrostatic attractions in the exhaust dyeing process. The cationic sites of the cationized cotton fabric could also restrict the movement of dye anions, resulting in decreased hydrolysis of reactive dyes (Arivithamani and Giri Dev 2017). The longitudinal morphology of cotton fibers was examined under bright field illumination using an optical microscope (Leitz Dialux, UK). It is clearly apparent that raw cotton has natural convolutions. However, after alkali treatment at pH 11 and pH 12 conditions, fibers became more rod-like cylinders, and the convolutions were completely removed. Most importantly, alkaline pH 12 conditions gave rise to a rounder shape can be formed which influenced more dye exhaustion and fixation (Remadevi *et al.* 2016, Wicker and Hallam 1970).

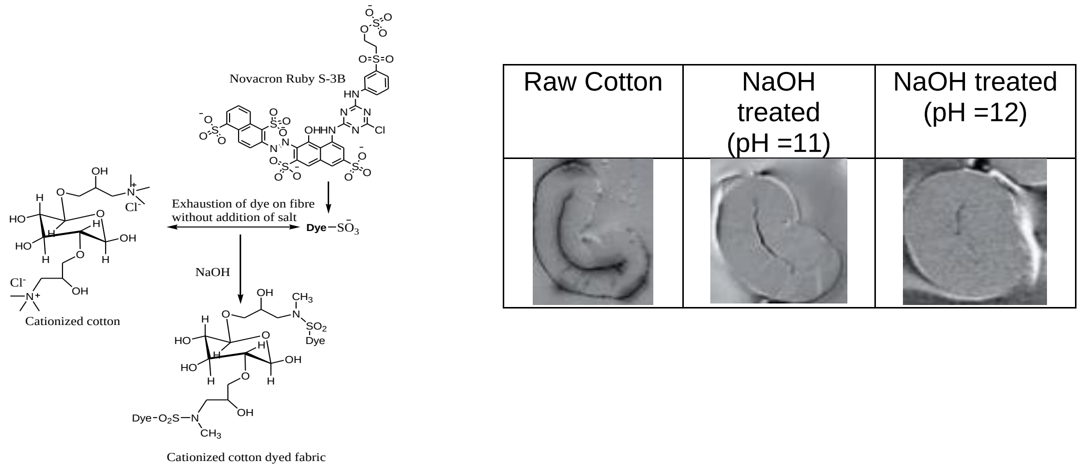

Fig. 4. Dyeing mechanism of cationized cotton fabric and optical images of cotton fibers cross-sections

CONCLUSIONS

The Taguchi approach was used in this study to optimize the process for salt-free reactive dyeing of cotton. Color strength (*k*/*s*) and wash fastness were selected as characteristics values and two kinds of multiple characteristics value analyses were performed. According to the single characteristics value, the maximum process condition was with a cationization temperature at 40 °C and dyeing pH at 11. According to the process maximization method, the optimum condition was with a concentration of ALBAFIX-WFF of 30 g/l, cationization temperature at 80 °C, dyeing pH 12, and material-to-liquor ratio (M: L) of 1:5. The process maximization method showed better dyeing properties than single characteristic value.

REFERENCES

Arivithamani, N., and Giri Dev, V. R. (2017). " Cationization of cotton for industrial scale salt-free reactive dyeing of garments" *Clean Tech Environ Policy* 19, 2317-2326. DOI: 10.1007/s10098-017-1425-y

Arju, S. N., Afsar, A. M., Das, D. K., and Khan, M. A. (2014). "Role of reactive dye and chemicals on mechanical properties of jute fabrics polypropylene composites," *Procedia Engineering* 90, 199-205. DOI: 10.1016/j.proeng.2014.11.837

Bhuiyan, M. A. R., Shaid, A., and Khan, M. A. (2014). "Cationization of cotton fiber by chitosan and its dyeing with reactive dye without salt," *Chemical and Materials Engineering* 24, 96-100. DOI: 10.13189/cme.2014.020402

Broadbent, A. D. (2001). *Basic Principles of Textile Coloration*, Society of Dyers and Colourists, Bradford, UK.

Buschle-Diller, G., and Zeronian, S. H. (1992). "Enhancing the reactivity and strength of cotton fibers, " *J Appl. Polym. Sci.* 45, 967-979. DOI: 10.1002/app.1992.070450604

Chattopadhyay, D. P., Chavan, R. B., and Sharma, J. K. (2007). "Salt-free reactive dyeing of cotton" *Int. J. Cloth. Sci. Technol.* 19, 99-108. DOI: 10.1108/09556220710725702

Choudhury, A. K. R. (2014). "Coloration of cationized cellulosic fibers – A review," *AATCC J. Res.* 1, 11-19.

EI-Shishtawy, R. M., and Nassar, S. H. (2002). "Cationic pretreatment of cotton fabric for anionic dye and pigment printing with better fastness properties," *Coloration Technology* 118, 115-120. DOI: 10.1111/j.1478-4408.2002.tb00367.x

Ghani, J. A., Choudhury, I. A., and Hassan, H. H. (2004). "Application of Taguchi method in the optimization of end milling parameters," *Journal of Materials Processing Technology* 145, 84-92. DOI: 10.1016/S0924-0136(03)00865-3

Gökkus, Ö., Yildiz, Y., and Yavuz, B. (2012). "Optimization of chemical coagulation of real textile wastewater using Taguchi experimental design method," *Desalination and Water Treatment* 49, 263-271. DOI: 10.1080/19443994.2012.719334

Hauser, P. J., and Tabba, A. H. (2001). "Improving the environmental and economic aspects of cotton dyeing using a cationized cotton," *Color Technol* 117, 282-288. DOI: 10.1111/j.1478-4408.2001.tb00076.x

Hossain, I., Hossain, A., and Choudhury, I. A. (2016). "Dyeing process parameters optimisation and colour strength prediction for viscose/lycra blended knitted fabrics using Taguchi method," *The Journal of The Textile Institute* 107(2), 1-11. DOI: 10.1080/00405000.2015.1018669

Jung, J. J., Kim, S., and Park, C. K. (2016). "Optimization of digital textile printing process using Taguchi method," *Journal of Engineered Fibers and Fabrics* 11(2), 51-59. DOI: 10.1177/155892501601100207

Kabir, S. M. M., Salauddin, S., and Koh, J. (2020). "Sustainable low liquor ratio dyeing of cotton with C. I. Reactive Blue 21 using dioctyl sodium sulfosuccinate," *Textile Research Journal* 91(9-10), 1-11. DOI: 10.1177/0040517520971363

Kannan, S. S. (2006). "Influence of cationization of cotton on reactive dyeing," *Journal of Textile and Apparel Technology and Management* 5(2), 1-16.

Khatri, A., Hussain, M., Mohsin, M., and White, M. (2015). "A review on developments in dyeing cotton fabrics with reactive dye for reducing effluent pollution," *J. Clean. Prod.* 87, 50-57. DOI:10.1016/j.jclepro.2014.09.017

Koh, J., Sim, G., and Kim, J. (2001). "pH control in the dyeing of polyamide with acid dyes" *Color Technol* 117, 156-160. DOI:10.1111/j.1478-4408. 2001.tb00055.x

Kuo, C. F. J, and Lin, W. T. (2019). "Optimum processing parameters of sueding fabric comfort by applying the Taguchi method and fuzzy theory," *Textile Research Journal* 89 (23-24), 5165-5176. DOI: 10.1177/0040517519849465

Lewis, D. M., and McIlroy, K. A. (1997). "The chemical modification of cellulosic fibres to enhance dyeability," *Rev. Prog. Color.* 27, 5-17. DOI: 10.1111/j.1478-4408.1997.tb03770.x

Liu, Z. T., Yang, Y., Zhang, L., Liu, Z.W., and Xiong, H. (2007). "Study on the cationic modification and dyeing of ramie fiber," *Cellulose* 14, 337-345. DOI: 10.1007/s10570-007-9117-0

Mavruz, S., and Ogulata, R. (2010). "Taguchi approach for the optimisation of the bursting strength of knitted fabrics," *Fibres and Textiles in Eastern Europe* 18, 78-83.

McDonald, R. (1997). *Colour Physics for Industry* (2^{nd} ed.), Society of Dyers and Colourists, Bradford, UK.

Montazer, A. R. M. (2007). "Salt free reactive dyeing of cationized cotton," *Fibers and Polymers* 8, 608-612. DOI: 10.1007/BF02875997

Mustafa Tutak, A. O. O. (2010). "Reactive dyeing of cationized cotton: Effects on the dyeing yield and the fastness properties," *Journal of Applied Polymer Science* 119, 500-504. DOI: 10.1002/app.32648

Novak, J. T., Love, N. G., Smith, M. L., and Wheeler, E. R. (1998). "The effect of cationic salt addition on the settling and dewatering properties of an industrial activated sludge," *Water Environ. Res.* 70(5), 984-996. https://www.jstor.org/stable/25045109

Park, C. K., and Young, H. J. (2005). "A process for optimizing sewing conditions to minimize seam pucker using the Taguchi method," *Textile Research Journal* 75 (3), 245-252. DOI: 10.1177/004051750507500310

Rajput, A. W., Ali, U., Zahid, B., Abbas, A., Jamshaid, H., and Qureshi, R. F. (2018). "Application of Taguchi method to investigate the effect of temperature, heating time, concentration and particle size on improved gel spinning process of UHMWPE," *Industria Textila* 19, 347-351.

Remadevi, R., Gordon, S., Wang, X., and Rajkhowa, R. (2016). "Investigation of the swelling of cotton fibers using aqueous glycine solutions," *Textile Research Journal* 87(18), 1-10. DOI: 10.1177/0040517516665267

Sanjit Acharya, F. M. (2014). "Chemical cationization of cotton fabric for improved dye uptake," *Cellulose* 21, 4693-4706. DOI: 10.1007/s10570-014-0457-2

Shore, J. (2002). *Colorants and Auxiliaries* (2nd ed.), Society of Dyers and Colourists, Bradford, UK.

Swapnil, G. (2015). "Application of Taguchi method for optimization of concrete strengthened with polymer after high temperature," *Construction and Building Materials* 79, 97-103.

Teng, X., Zhang, S., and Ma, W. (2011). "Application of a hydrolyzable cationic agent, poly (acryloxyethyl trimethylammonium chloride), in salt-free reactive dyeing for good dyeing properties," *Journal of Applied Polymer Science* 122, 2741-2748. DOI: 10.1002/app.34023

Üstüntağ, S., Şenyiğit, E., Mezarcıöz, S., and Türksoy, H. G. (2020). "Optimization of coating process conditions for denim fabrics by Taguchi method and grey relational analysis," *Journal of Natural Fibers* 19(2), 1-15. DOI: 10.1080/15440478.2020.1758866

Wahyudin, Kharisma, A., Murphiyanto, R. D., Perdana, M. K., and Kasih, T. (2017). "Application of Taguchi method and ANOVA in the optimization of dyeing process on cotton knit fabric to reduce re-dyeing process," *IOP Conf. Ser.: Earth Environ. Sci*. 109, 012023. DOI: 10.1088/1755-1315/109/1/012023

War Wicker, J. O., and Hallam, P. (1970). " The effect of alkaline and acid swelling agents on the mechanical properties of cotton fibres. *Text. Inst.* 61, 61-76. DOI:10.1080/00405007008629967

Yang, W. H., and Tarng, Y. S. (1998). "Design optimization of cutting parameters for turning operations based on the Taguchi method," *Journal of Materials Processing Technology* 84,122-129. DOI: 10.1016/S0924-0136(98)00079-X

Yang, Y., and Li, S. (1993). " Instrumental measurement of the levelness of textile coloration," *Text Chem Colorist* 25, 75-78.

Yoon, S. Y., Park, C. K., Kim, H., and Kim, S. (2010). "Optimization of fusing process conditions using the Taguchi method," *Textile Research Journal* 80(11), 1016-1026. DOI: 10.1177/0040517509349784

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