

Combined process for Reactive printing and crease resistance finishing for cotton fabric using an experimental design technique

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Experimental design is a standard statistical technique used to identify key factors and levels that influence the process. In the present investigation, the factors affecting the combined operation of reactive printing and crease resistance finishing were designed and analyzed using Design of Experiment (DOE). The influence of individual factors and their interactions on color yield (k/s) and Dry Crease Recovery Angle (DCR) have been critically examined using software Design Expert 7.0. The results showed that apart from the influence of individual factors, the final color yield and dry crease recovery angle also depended on the interaction effect of the factors. It has been observed from present analysis that the predicted values are in good agreement with experimental data, the correlation coefficients were found to 0.9802 & 0.9139

Keywords: DOE, Reactive Printing, Crease Resistance Finishing, k/s, DCR.

Introduction

In this age of fuel and energy crisis it is desirable to think of such processes and formulations which would result in lowering of fuel and energy consumption. From time to time immemorial man has been attempting to save time and energy. It is this urge that has led the industrial development and has given the technology in its present advanced shape. Textile printing process remains firmly rooted in traditional techniques and technologies that are continually being refined rather than revolutionized.

The same global pressure that affects all textile manufacturing is also applicable to textile printers who require speed and economy of production. All these demands mean that textile printers must be able to do right-first-time processing, just-in-time manufacturing, and dramatic reductions in lead times [1-2].

While simultaneous application of crease recovery finish and reactive dyeing has been reviewed previously [3-7], few studies have been reported on combined reactive printing and crease resistance finishing of cotton fabrics [8]. The present research work was undertaken with a view to study the factors influencing the final color yield (k/s) and dry crease recovery angle (DCR) for combo process of reactive printing and CR finishing.

Experimental Materials

Fabric

Commercially Singed, desized, scoured, bleached and mercerized cotton fabric with satin weave structure, 40x40 s, 130 ends/inch x 73 picks/inch, and an area density of approximately 136 g/m² was used in this research work.

Table 1: Factors and respective levels used in two-level fractional factorial design

Factor	Name	Level ^a	
		(-)	(+)
A	Hue	Red	Blue
B	Chroma	1%	3%
C	Drying conditions	7 min, 60°C	3 min, 100°C
D	concentration of Fixapert F-ECO	100 g/l	200 g/l
E	Concentration of MgCl ₂	15% of CR	25% of CR
F	Fixation conditions	Saturated steam 102°C-6 min	Hot air 180°C-3min

a⁻ and a⁺ refer to the lower and upper levels, respectively.

Chemical and Colorants

The Crease Recovery finishing agent used was Fixapert F-ECO (BASF), based on dimethyldihydroxy ethylene urea (DMDHEU). Magnesium chloride MgCl₂ was used to catalyse the CR finishing, Solusoft MW (Silicon softener), Ceranine-L (An ionic Softener) and Invadine PBN (Wetting Agent). The reactive dyes used were Drimarine Red P2B and Drimarine Blue P-3RL (Clariant).

Other chemicals used in this study were commercially available thickener Lamitex HP (sodium alginate), sodium bicarbonate, urea, Reduction Inhibitor (Lyoprint RG) and sodium hexa meta phosphate as a sequestrant.

Experimental Methods

Print-finish Paste Manufacture

The stock paste was prepared according to the following recipe: Urea 150g/kg, Lamitex HP (4%) 750 g/kg, sodium bi carbonate 30 g/kg, lyoprint RG 50 gm/kg and sodium hexa meta phosphate 5 gm/kg. The stock paste was adjusted to a constant viscosity of 65 dPa by adding the necessary amount of water. The printing pastes of two different levels were prepared with Drimarine P Hue and chroma as outlined in Table 1.

The CR finishing liquor was prepared by using Solusoft MW 20g/l, Ceranine-L 20g/l and Invadine PBN 5g/l. The final finish bath were prepared with Fixa pert F-ECO and MgCl₂ as outlined in Table 1.

Print-finish Procedure

The combined process of reactive printing and CR finishing was carried out as follows: In the first stage the fabric was immersed in an aqueous solution of CR finish, and then squeezed to obtain a 70% wet pickup. The wet fabric was then dried. In the second stage the treated fabric was printed by the lab scale Rotary Printing machine (Zimmer). The printed fabric was again dried.

In the third stage, the print-finish fabric was fixed, and then washed according to washing-off procedure and finally dried. The preparation of finish bath, drying conditions, printing recipe and fixation conditions were employed in accordance with the experimental design arrangement as stated in Table 1 and 2.

Evaluation of fabric properties

Color yield Measurement

The printed fabrics were conditioned (at temperature 25±1° C and relative humidity 65 ± 1%) before color yield

measurement with a Tex-Flash spectrophotometer. The condition for measurement was set under specular excluded with large aperture. The fabric was folded twice to ensure opacity. The color yield (k/s value) was calculated for wavelengths 400-700nm at 20nm intervals within the visible spectrum. The k/s was calculated according to Eqn 1:

$$k/s = (1-R)^2 / 2R \quad (1)$$

Where, **k** is the absorption coefficient, **s** is the scattering coefficient and **R** is the reflectance of the colored samples. The higher the k/s value is, the greater the color yield and dye uptake.

Table 2: Two-level fractional factorial design with a 2⁶⁻¹ matrix (Run Order)

Run	Variable (Factors)					
	A	B	C	D	E	F
1	-	+	-	+	+	+
2	-	-	-	-	-	-
3	+	-	+	-	+	+
4	-	-	-	-	-	+
5	-	-	+	-	+	-
6	-	-	-	+	-	-
7	+	-	+	-	-	-
8	-	-	+	+	+	+
9	-	-	+	+	+	+
10	+	+	-	+	+	-
11	-	+	-	-	-	-
12	-	+	+	-	+	+
13	-	+	+	+	-	+
14	-	-	-	-	+	-
15	-	-	-	-	+	+
16	+	+	+	+	+	-
17	+	+	-	-	+	-
18	-	-	+	-	-	-
19	+	-	+	+	-	+
20	-	-	-	+	-	+
21	+	+	+	-	-	+
22	-	+	+	-	-	-
23	+	-	+	+	-	+
24	+	+	+	+	+	-
25	-	-	+	+	-	-
26	-	+	-	+	+	+
27	-	+	-	-	-	+
28	+	-	-	+	+	-
29	+	-	-	-	-	+
30	+	+	-	+	-	-
31	+	+	-	-	+	-
32	+	-	+	-	-	+

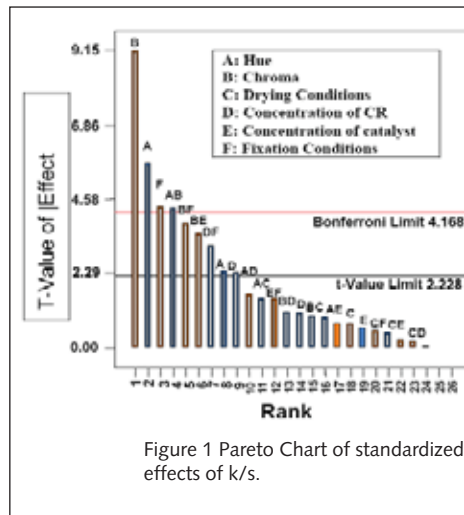


Figure 1 Pareto Chart of standardized effects of k/s.

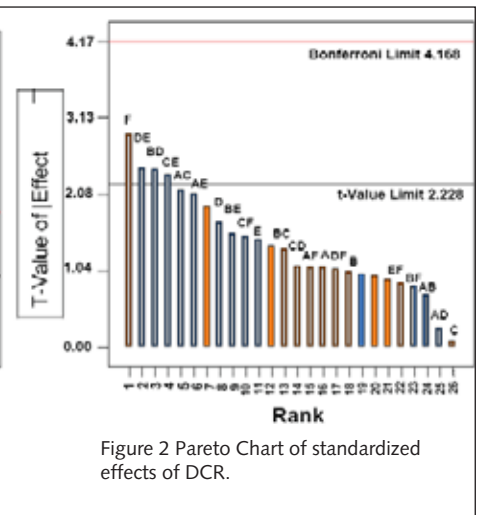


Figure 2 Pareto Chart of standardized effects of DCR.

Evaluation of crease recovery angle

The print-finish fabrics were conditioned (at temperature 25±1°C and relative humidity 65±1%) before the measurement of easy-care properties imparted by the CR finish. The dry crease recovery angle (DCR) of the fabric was measured using AATCC test method 66-1990, using Shirley Crease recovery tester. The wider the DCR is, the higher the crease recovery.

Experiment design

Two-level fractional factorial design was used to explore the effect of different factors namely: (i) hue, (ii) chroma, (iii) drying conditions, (iv) concentration of crease resistant, (v) concentration of catalyst, (vi) fixation conditions on the combined reactive printing and crease resistance finishing. A 2⁶⁻¹ two-level fractional factorial design of 32 trials with two repetitions were run according to the design matrix as shown in Table 2. The experiments were performed in random order. The results were analyzed with the Minitab program package software (Minitab Inc.). The details of the experimental design arrangement are shown in Tables 1 and 2.

Results and discussion

After processing the experimental trials, the k/s and DCR curves of different trials were plotted and the sum of k/s and DCR values of each trial were calculated with the help of Minitab software, the dominant factors of the combined print-finish process were assessed using Pareto Charts, main effect interactions and interaction plots of the six different factors.

Pareto Chart

The dominant factor can be determined using a Pareto Chart. A Pareto

Chart is the specialized version of a histogram that ranks the categories in the chart from most frequent to least frequent. The construction of the Pareto Chart places the highest cause of effect to the upper position and the rest of the effects, in descending order, to the lower position [9].

The Pareto Chart of the two response variables k/s and DCR are shown above in Figures 1 and 2.

Main Factors Interaction Plot

From the result shown in Figure 1, the factors Chroma (B), Hue (A) and Fixation Conditions (F) respectively were confirmed as the major factors influencing the final color yield of combined print-finish process. The interaction of these factors with other factors in affecting the final color yield could be demonstrated in the main factors interaction plot [9].

It was further confirmed that increasing the chroma could enhance the color yield, changing the hue would change the k/s value depending on the level of reflectance of the particular color, and Fixation by means of Hot air could enhance the color yield of combined print-finish process. Furthermore, it also interacted with one of the other factors to influence the final color yield of combined print-finish process, i.e. the two dominant factors interacting with each other would also influence the final color yield.

From the result shown in Figure 2, the factor of "Fixation Conditions (F)" was confirmed as the major factor influencing the crease resistance of combined print-finish process. It was further confirmed that hot air fixation gives higher DCR values. Furthermore, the interaction of two factors would influence the DCR values of the combined print-finish process. The two dominant factors effecting the k/s and DCR are summarized in Table 3.

Table 3: The two dominant factors effecting k/s and DCR

	Factor 1	Factor 2
k/s	Hue	Chroma
	Chroma	Fixation conditions
	Chroma	concentrationof catalyst
	concentrationof CR	Fixation conditions
	Hue	Fixation conditions
	concentrationof CR	concentrationof catalyst
DCR	concentrationof CR	concentrationof catalyst
	Chroma	concentrationof CR
	Drying Cond.	concentrationof catalyst

Interaction plot

Similar to the Pareto plots and the interaction of the main factors, the interaction calculations can be displayed graphically to measure the joint effect of two factors for k/s and DCR. The interaction plots display the average at each of the combinations of the two factors A and B [(-,-);(+,-);(-,+);(+,+)] using the B factor level as the horizontal and the average as the vertical axis; the averages having the same level of A, i.e. [(-,-);(+,-);(-,+);(+,+)] are joined by a line [9]. The interaction plots of k/s and DCR are shown in Figure 3 and Figure 4. The interaction effects between different factors that affected the final colour yield and crease resistance of combined print-finish fabrics were demonstrated in the interaction plots, and the results showed that different factors interacted with each other.

Assessment of the significant factors in combo process**Influence of Chroma on Combo Process**

Chroma is the concentration of dye. It is a measure of saturation associated with color; degree of color purity; relative brightness of a hue when compared to another. Colour yield of the fabric is associated with the chroma value of dye. From the Pareto Chart shown in Figure 1, chroma was located at the highest frequency when compared with all other factors in k/s. This implied that it was a dominant factor with respect to the color yield.

The interaction plot of k/s Figure 3(a) showed that the final colour yield of the combo process attained the maximum value in case of high chroma of reactive dye. This is due to the fact that more number of dye sites are available for covalent bonding in high concentration of reactive dyes thus increasing the colour yield of the fabric.

Influence of Hue on Combo Process

Hue is the colour of a particular dye. Daylight (white light) is made up of numerous waves or impulses each having different dimensions or wavelengths. When separated, any single wavelength will produce a specific color impression to the human eye. When light fall on fabric, the dye absorbs certain waves and reflects others, this determines the hue of the dye. The light actually generates the color. What we see as color is the reflection of specific wavelength of light rays from fabric. the hue red means the chromophore of the dye reflects only red light and absorbs all other light.

From the Pareto Chart Fig 1, Hue was found to be the second most frequent factor when compared with all the other factors for k/s. this implied that it was another dominant factor with respect to color yield.

The interaction plots of k/s showed that when the Hue red was used in the print paste, the color yield of the printed fabrics was enhanced correspondingly Figure 3(a). This is due to the reason that hue Red gives reflection in broader region of spectrum than blue therefore increasing the k/s value.

Influence of Fixation Conditions on Combo Process

Fixation is the most important part of combo process. When the fabric is printed and finished through the combo process and dried, the dye and finishing chemical are not actually transferred into the cloth, only a thin dried film of thickner containing the dye and other chemicals are mechanically deposited.

The interaction plot of k/s and DCR Figure 3(a) and 4(a) showed that the final colour yield and DCR of the combo process attained the maximum k/s and DCR at 180°C-3 min, hot air fixation but the samples were slightly yellow and pale. The reason being that high temperature of curing aids the concurrent fixation of reactive dye and CR finish, but at the same time causes the paling of shade and yellowing of fabric due to the scorching of cotton fiber. This indicates that Hot Air Fixation with reduced temperature and increased time may be tested in further experimental work of combined print-finish process to avoid this problem.

Assessment of the significant interactions in combo process**Influence of Hue and Chroma on Combo Process**

The interaction of hue and chroma found to be significant in k/s model. In interaction plot Figure 3(b) increasing the

chroma from 1% to 3% of red hue significantly increases the colour yield but when compared with blue hue the increment is not that much significant as in case of red hue. The reason behind this is the fact that blue is more sensitive than red, as its region of reflection is very small compared to red.

Influence of Chroma and Fixation Condition on Combo Process

The interaction of chroma and fixation condition was found to be significant in k/s model. The interaction plot figure 3(c) showed at lower level of chroma changing the fixation condition from steaming to curing increases the colour yeild of combo process but at higher level of chroma this enhancement of colour yeild increase drastically. Such an increment in colour yield at higher level of chroma is due to the lower extent of reactive dye interaction with CR.

Influence of Chroma and Concentration of Catalyst on Combo Process

The interaction of chroma and concentration of catalyst has been found to be significant and crossed in k/s model as shown in Figure 3(c). At low level of chroma, the low concentration of catalyst provides high colour yield, however when chroma increased to 3% the order reversed, high concentration of catalyst gave high colour yield. This is due to the reason that high level of catalyst of CR quickly deactivates without accelerating the CR to the fabric. Fewer CR linkages are being formed with the cotton fiber resulting in more reactive dye being fixed on the fabric thus increasing the k/s.

Influence of Concentration of CR and Fixation Conditions on Combo Process

The interaction of concentration of CR and fixation condition was found to be significant and interesting in k/s model. In interaction plot Figure 3(e) when concentration of CR increases from 100g/l to 200g/l in steaming mode of fixation k/s increases. This is due to the fact that cross linking of CR is not favorably established in steaming environment. Instead, the steaming aids the fixation of reactive dyes increasing the color yield. However in interaction plot Figure 3(e) when the concentration of crease resistant increased to 200g/l, the color yield of combo process decreased in hot air fixation. Such a drop in color yield with respect to increased amount of crease resistant is due to an increase in cross linking of crease resistant in curing condition.

Influence of Hue and Fixation Conditions on Combo Process

The interaction is found to be significant in k/s model. Both fixation conditions showed same trend Figure 3(b) with respect to hue. Irrespective of fixation condition, colour yield

of red is higher than blue as justified earlier, whereas in the same hue curing provides higher colour yield as compared to steaming.

Influence of Concentration of CR and Concentration of Catalyst on Combo Process

The interaction of concentration of crease resistant and concentration of catalyst was found to be significant and crossed in both k/s and DCR models Figure 3(e) & 4(e). When concentration of CR was low, high concentration of catalyst gave high k/s and DCR, but when concentration of CR increased the trend reversed. This is due to the reason that there is a limiting point of catalyst concentration, after which it will quickly deactivate without performing its task. In case of CR it is added as 15-25 % of CR, whereas limiting point is 30g/l. When concentration of CR increases to 200 g/l 25% of CR is 50g/l which is > 30g/l. So the concentration of catalyst should increase till its limiting point i.e. 30g/l.

Influence of Chroma and Concentration of CR on Combo Process

The interaction is found to be significant and crossed in DCR model. When chroma of reactive dye is low, high concentration of CR gives high DCR Figure 4(c) but as concentration of reactive dye increases the trend reverses. This is due to the reason that reactive dye is highly reactive and at high concentration it doesn't allow the CR to fix on cotton.

Influence of Drying Conditions and Concentration of Catalyst on Combo Process

The interaction is found to be significant and crossed in DCR model. When drying condition is low i.e. 60°C-7min, high concentration of catalyst gives high DCR, whereas when drying condition is high 100°C-3min, the trend reverses Figure 4(d). The reason is that catalyst activates with temperature when drying temperature is increased to 100°C,

high concentration of catalyst activates rapidly and without performing its task deactivates. The drying conditions should be optimized at 85°C-5min.

Conclusions

Through the experimental design method it was found that hue, chroma and fixation conditions had major contribution to the final color yield and dry crease recovery angle of combined print-finish process. Based on the experimental design study, it was further confirmed that each factor had an interaction effect with each other and other factors such as maximum color yield and dry crease recovery angle were the compromise effect of these factors. Relatively satisfactory properties of printed and finished cotton fabric can be obtained with appropriate adjustment of the treating conditions. An important thing was identified through this experimental work that the factor "concentration of crease resist-

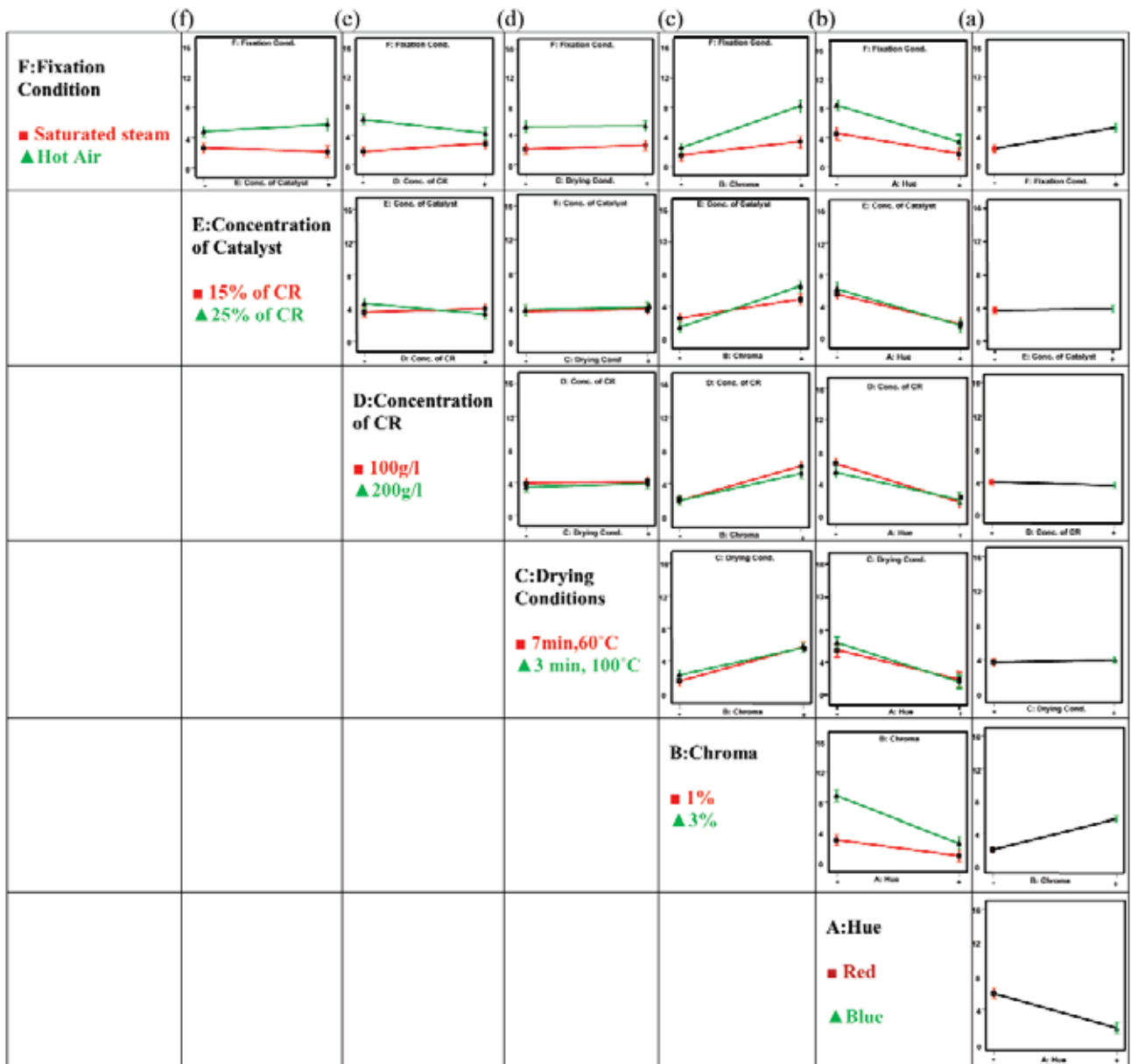


Figure 3: Main factor and interaction plots of K/S.

ant" was not identified as significant factor for dry crease recovery angle. It is possible that the reactive dye fixation on the cotton fiber will compete with the cross linking of crease resistant with the fiber. Thus, increasing the dye concentration in the print paste will result in more dye molecules being available to be absorbed and react with the cotton, whilst the crease resistant will have a lower chance to be absorbed and cross linked to the cotton. Thus, the need for further experimental work having a fixed concentration of reactive dyes with a broader range of crease resistant is evident.

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Figure 4: Main factor and interaction plots of DCR.