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Reactive Printing and Crease Resistance Finishing of Cotton Fabrics
Part I - Study of Influential Factors by an Experimental Design Approach

Fareha Asim
Department of Textile Engineering
NED University of Engineering & Technology
Karachi, Pakistan

Muzzaffar Mahmood
Department of Mechanical Engineering,
NED University of Engineering & Technology
Karachi, Pakistan

ABSTRACT

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 was 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) has 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 shortages, it is desirable to think of processes and formulations which would result in lowering fuel and energy consumptions. 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 shaped the technology to the present advanced form.

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 also affects textile printers, requiring a higher speed and economic production method. All these demands mean that textile printers must be able to do right-the-first-time processing, just-in-time manufacturing, and a dramatic reduction in lead time [1-2]. Reports in literature reveal that simultaneous application of crease recovery finish and reactive dyeing has been reviewed previously [3-7]. But 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 of studying 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, 40x40s, 130 ends/inch x73 picks/inch, and an area density of approximately 136 g/m^2 were used in this research work.

Chemical and Colorants
The crease recovery finishing agent used was Fixapert F-ECO (BASF), based on dimethlodihydroxy ethylene urea (DMDHEU). Magnesium chloride $MgCl_2$ was used to catalyze the CR finishing, Solusoft MW (Silicon softener), Ceranine-L (anionic 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.

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.

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

		Level^a	
Factor	**Name**	(-)	(+)
A	Hue	Red	Blue
B	Chroma	1%	3%
C	Drying Conditions	7min,60˚C	3 min, 100˚C
D	Conc. Of Fixapert F-ECO	100g/l	200g/l
E	Concentration of MgCl2	15%of CR	25% of CR
F	Fixation Conditions	Saturated Steam 102˚C-6min	Hot Air 180˚C-3min

a '-'and '+'refer to the lower and upper levels, respectively

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:

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

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.

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.

Table 2: Two-level fractional factorial design with a 2^{6-1} matrix (Run Order)

	Variable					
Run	**Factor A**	**Factor B**	**Factor C**	**Factor D**	**Factor E**	**Factor 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	+	-	+	-	-	+

Pareto Chart

The dominant factor can be determined using a Pareto chart. A Pareto chart is 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 in Figure 1and 2.

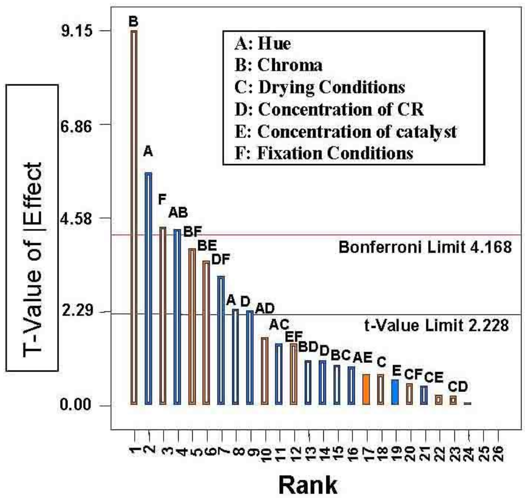

Figure 1 Pareto chart of standardized effects of k/s

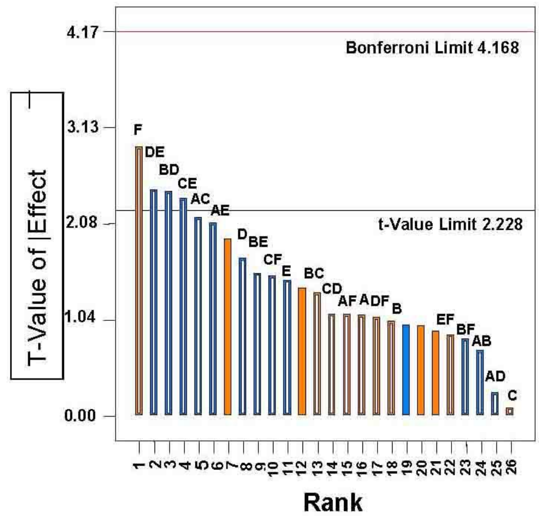

Figure 2 Pareto chart of standardized effects of DCR

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 is summarized in Table 3.

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

k/s	***Factor 1***	***Factor 2***
	Hue	Chroma
	Chroma	Fixation Conditions
	Chroma	Conc. Of Catalyst
	Conc. Of CR	Fixation Conditions
	Hue	Fixation Conditions
	Conc. Of CR	Conc. Of catalyst
DCR		
	Conc. of CR	Conc. Of Catalyst
	Chroma	Conc. Of CR
	Drying Cond.	Conc. Of 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 color 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. Color 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 color 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 is available for covalent bonding in high concentration of reactive dyes thus increasing the color yield of the fabric.

Influence of Hue on Combo Process
Hue is the color of 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 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 is not actually transferred into the cloth, and only a thin dried film of thickener containing the dye and other chemicals were mechanically deposited. The interaction plot of k/s and DCR Figure 3(a) and 4(a) showed that the final color yield and DCR of the combo process attained the maximum k/s and DCR at 180˚C-3min, Hot Air fixation but the samples are slightly yellow and pale. The reason behind that is the high temperature of curing, which 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 color 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 as compared to red.

Influence of Chroma and fixation condition on Combo Process

The interaction of chroma and fixation condition 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 color yield of combo process but at higher level of chroma this enhancement of color yield increase drastically. Such an increment in color yield is at higher level of chroma is a due to the lower extent of reactive dye interaction with CR.

Influence of Chroma and Conc. of catalyst on Combo Process

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

Influence of Conc. of CR and Fixation Conditions on Combo Process

The interaction of concentration of CR and fixation condition found to be significant and interesting in k/s model. In interaction plot Figure 3(e) when conc. 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 that's why color yield increases. However In interaction plot Figure 3e when the concentration of crease resistant increase 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 condition showed same trend Figure 3(b) with respect to hue, irrespective of fixation condition color yield of red is higher than blue as justified earlier, whereas in same hue curing provides higher color yield as compared to steaming.

Influence of Conc. of CR Conc. of Catalyst on Combo Process

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

Influence of Chroma and Conc. 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 reversed. This is due to the reason that reactive dye is highly reactive than CR. At high concentration of reactive dye it doesn't allow the CR to fix on cotton.

Influence of Drying Conditions and Conc. 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 reversed Figure 4(d). The reason is that catalyst activates with temperature when drying temperature 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 an experimental design, it was found that hue, chroma and fixation conditions had major contributions 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 among them and with other remaining factors such that the maximum color yield and dry crease recovery angle were the combined effects of these factors. Properties of printed and finished cotton fabrics were reasonably satisfactory when the treatment conditions were appropriately adjusted. One important finding was from this experimental was that "Concentration of Crease Resistant" was not identified as a significant factor for dry crease recovery angle. It is possible that the reactive dye fixation on the cotton fiber competes with the cross linking of crease resistant with the fiber. Thus, an increase in the dye concentration in the print paste may make more dye molecules to be available for absorption for reaction with the cotton, whilst the crease resistant finish will have a less chance to be absorbed and cross-linked with cotton. Thus, the need for a further experimental work is evident for fixing concentration of reactive dyes with a broader range of crease resistant finish.

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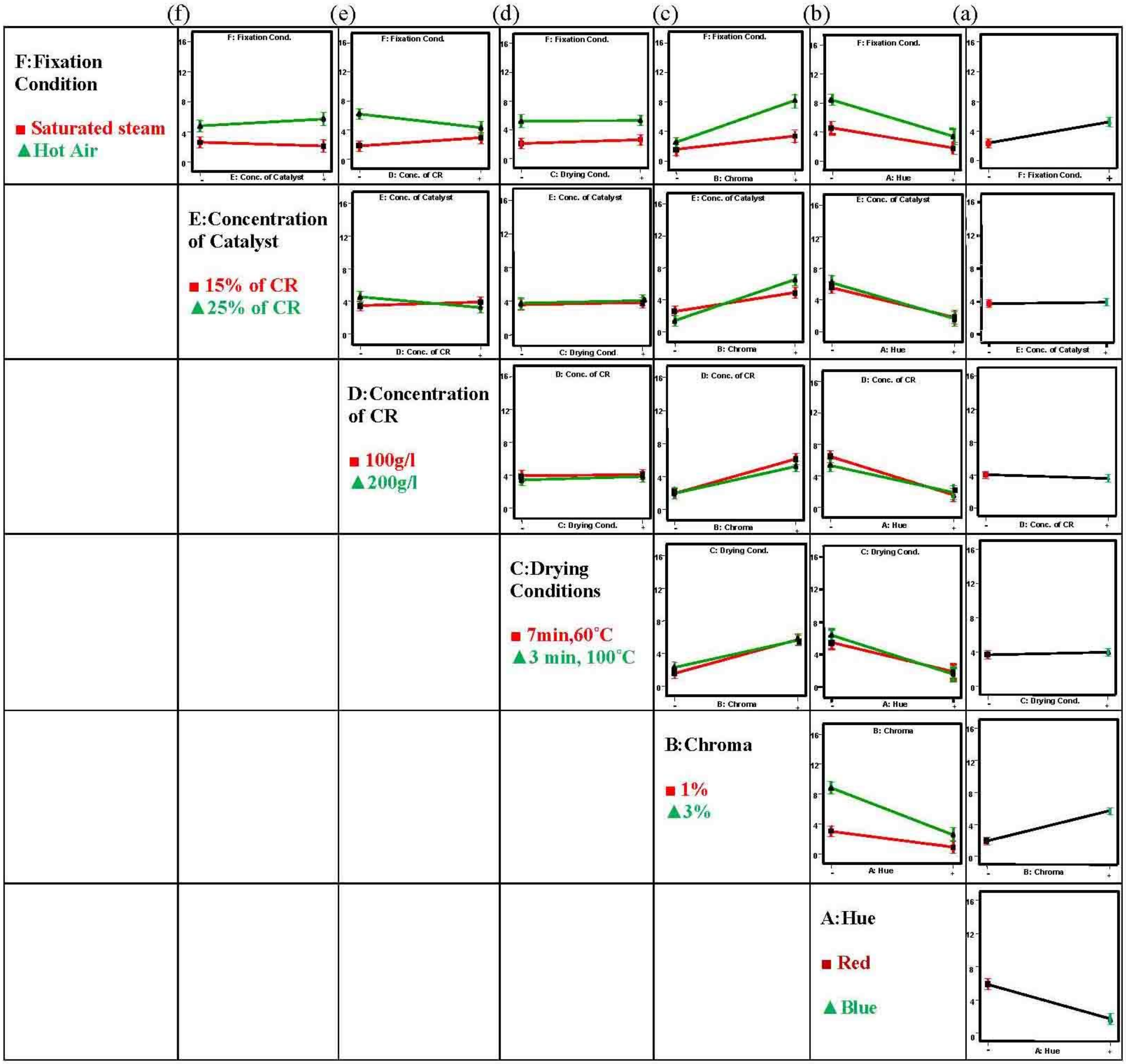

Figure 3: MAIN FACTOR AND INTERACTION PLOTS OF K/S

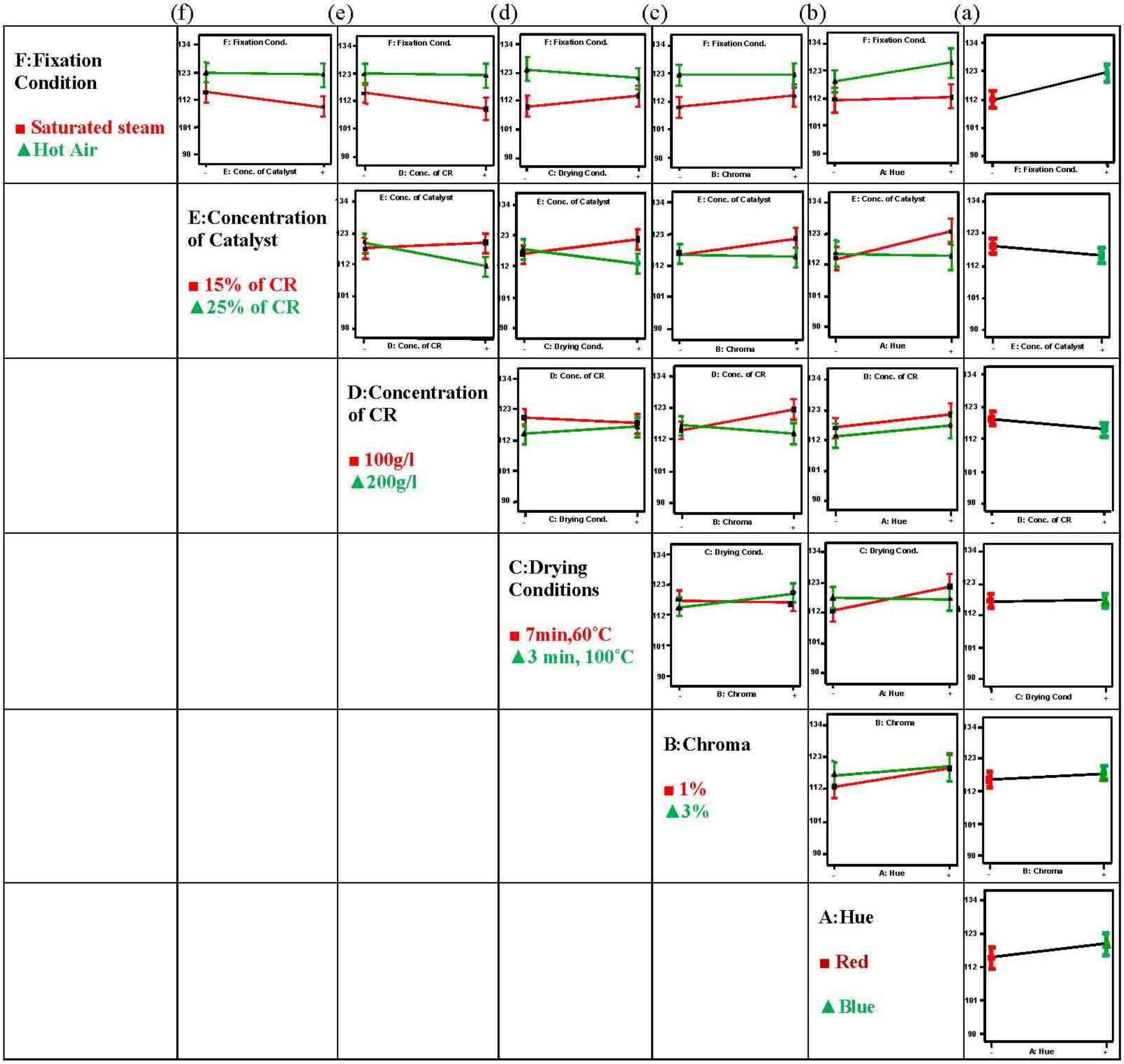

Figure 4: MAIN FACTOR AND INTERACTION PLOTS OF DCR