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Effective Reduction Clearing Parameters Involving Alternative Reducing Agent

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Abstract

Dyeing of polyester is done by using disperse dyes. Some of the disperse dyes remain unfixed during dyeing and create problems in shade and colorfastness properties. Reduction clearing (RC) is a process to remove these unfixed dyes and to enhance colorfastness properties. In the Pakistani textile industry, reduction clearing is being done by using sodium dithionite ($Na_2S_2O_4$), which has the best results in reducing dyestuff. The problem with sodium dithionite is that it is sensitive to air and also creates a lot of environmental issues. Its sensitivity causes inconsistent results of reduction clearing. Different dyers used different processes and recipes of reduction clearing. Thiourea Dioxide (TUD) is a green reducing agent and can be used as a replacement for sodium dithionite. TUD (CH_4N_2S) is a stable product with a comparable result with sodium dithionite. Interlock knitted fabric is used in this project whose composition has recycled polyester. The fabric was first dyed using black dyestuff and then reduction clearing was done on a lab-scale with different factors (Shade depth, the concentration of TUD, RC temperature). Three colorfastness tests were conducted to evaluate the different levels of each factor i.e. colorfastness to washing, fastness to perspiration, and fastness to water according to standard numbers ISO 105-C06, ISO 105-E04, and ISO 105-E01, respectively. It was observed that shade depth does not affect the results while concentration and RC temperature have a significant effect on the results. Moreover, the effective concentration of TUD was also determined.

Keywords: Shade Depth Dying, Colorfastness, Sodium Dithionite, Thiourea Dioxide.

1. Introduction:

Dyeing is one significant preparing venture in material creation, which increases the value of the product. The procedures for dyeing devour enormous volumes of energy, chemicals, and water. In the meantime, there is a hazard that the material is influenced by the dyeing procedure, which is probably going to decrease the life expectancy and most likely the recyclability of the material [1]. As a result of the low solubility of disperse dyes, some leftover dyes stay on the outside of the fiber. These unfixed colors badly affect the shading and fastness properties of the colored textures. A post-treatment procedure is done to expel these surface dyes from the fabric. This procedure is called ***Reduction clearing*** [2].

Reduction clearing is important to get the right shade and optimum fastness properties [3] and satisfy the customer by achieving given parameters[4]. The wet fastness property of a dyed fabric is enhanced by reduction clearing [5]. The reduction clearing process is used widely where dyeing of polyester and its blends is happening. If there is a blend of polyester and cotton, then

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disperse dyeing treatment is done in the start and reduction clearing is done before cotton dyeing [4]. The most common chemical used in reduction clearing is sodium dithionate in an alkaline medium in the presence of a non-ionic surfactant [2]. Reduction clearing of the disperse dyes kept superficially is the customary strategy to accomplish wanted wash-fastness properties. Leftover dyes molecules kept on surfaces are separated into littler, dismal, and all the more promptly water-solvent parts by reduction clearing. The most prominent chemical, which is used for reduction clearing, is sodium dithionite. Other chemicals like thiourea dioxide (TUD), inorganic salts and liquid reduction agents are utilized as a substitute of sodium dithionite [6]. In the event of unfixed colors not expelled, surface pollution can bother the splendor of shade and its fastness properties. Reduction clearing is done to colored polyester texture by utilizing caustic soda and sodium dithionite [7]. The viability of reduction clearing is subject to the concoction structure of the disperse dye. As most disperse dyes contain an azo gathering (N=N), they are delicate to treatment with a reducing agent. This obliterates the azo chromophore to lost its shade through the part of the azo gathering into two amino mixes with no color [8]. Sodium dithionite is very sensitive to air oxidation in an alkaline medium at high temperatures. Due to this reason, the consumption of sodium dithionate will be more to compensate for the loss due to air oxidation[9]. Sodium dithionite is a strong reducing agent because of its effective reducing power in the presence of an alkaline medium. It is used 2-3 g/l in a reduction clearing bath with 1-3 g/l caustic soda. The processing profile of reduction clearing is between 70°C to 80°C for 10 to 20 minutes [5]. More the temperature and quantity of chemicals in the reduction clearing bath, more deterioration of the fibers occurs [10]. A reducing agent is used in reduction clearing to reduce disperse dyes from the surface of the substrate and a non-ionic surfactant will remove it further from the bath [11].

Thiourea dioxide is used in the replacement of Sodium dithionite, which is a green reducing agent, comparatively. It is aminoiminomethane-sulfinic acid and during the process, it liberates sulfoxylic acid or sulfoxylate ions. These ions are the key which attacks and breaks C-S bond in alkaline medium at high temperature. The effectiveness of TUD increases with an increase in temperature [12] and it is more stable between 3 to 7 pH [13]. The instability of TUD is required to make it reactive and reductive. Its decomposition strongly depends on pH. Alkaline pH is required to make it unstable because it is stable in neutral to acidic medium [13]. It is used in many fields which involves chemistry, biochemistry, and chemical technology especially in the pulp, paper, and textile industry [14]. TUD is neutral at room temperature, it neither oxidized nor reduced and non-reactive to most chemicals. Besides, the resultant deterioration results of TUD have convincingly shown low defilement of ef?uent. Besides, as a strong or aqueous arrangement, TUD hardly creates an awful scent contrasted and traditional agents, for example, sodium hydrosul?te [15].

This study is carried out to replace sodium dithionite with Thiourea dioxide and to optimize its recipe and process profile, concerning the different concentration of dyestuff used to dye fabric. When a garment is washed, unfixed dyes from the surface of fabric will migrate to a nearby area, like labels. Nylon labels are used in all types of garments either 100% cotton, 100% polyester, or blends. The dyestuff, which was used to dye fabric, may cause discoloration of the Nylon fibers. Furthermore, if a garment has more than one fiber then there is a need to check discoloration on that fiber too. This study will be beneficial in identifying specific parameters of reduction clearing, to obtain the least discoloration on Nylon fibers. After the study, one will be able to select the right reduction clearing recipe and process by keeping in mind the requirement of the customer.

2. Materials and Methods:

2.1. Fabric:

The fabric used in this study was Interlock knitted fabric composed of recycled polyester and BCI cotton. Fabric is constructed in a way that on the front and in the middle of fabric there is 100%

recycle polyester yarn. On the backside of the fabric 80/20 (cotton/polyester), yarn was used. Knitting of fabric was done at Masood textile mills ltd. Faisalabad. Recycle polyester yarn was sourced from UNIFI America.

2.2. Dyestuff and Chemicals:

Black is the most common dyestuff used in textile industries. Deep black from "Yorkshire" (Chinese Dyestuff Company) was selected among a variety of dyestuffs available at Masood textile mills ltd. Faisalabad. In polyester dyeing, different auxiliaries were used before dyestuffs like leveling agent, dispersing agent, acetic acid, and acid buffer. 2 g/l of leveler was used for even dyeing, while 0.7 g/l of the dispersing agent was used to disperse the dyestuff in the liquor that helps in even migration. Acetic acid and acid buffer were used to maintain the required acidic pH for polyester dyeing, their quantities were 2 g/l and 0.5 g/l respectively.

In reduction clearing (RC), surfactant, caustic soda, and Thiourea dioxide (reducing agent) were used. Caustic soda was used to maintain an alkaline medium during reduction clearing. A strong surfactant was also used in a reduction clearing bath to remove the reduced compound from the surface of the fabric. Thiourea dioxide was used with 3 different concentrations, whereas, caustic soda and surfactant were used in 2g/l and 0.2 g/l, respectively.

2.3. Design of experiment

Three variables were used in this research i.e. Shade depth, RC temperature, and Thiourea dioxide concentration (Table 1). There were three levels of each factor and each test was conducted thrice, as displayed in Table 2.

Table 1: Factors and levels

Sr. #Factors	Level **1**	Level **2**	Level **3**
1 Shade depth (%)	3	4	5
2 Concentration (g/l)	0.5	1	1.5
3 RC Temperature c)	70	80	90

Table 2: Sample order table

Sr. #	Shade depth (%)	Concentration (g/l)	RC Temperature c
1	3	0.5	70
2	3	0.5	80
3	3	0.5	90
4	3	1	70
5	3	1	80
6	3	1	90
7	3	1.5	70
8	3	1.5	80
9	3	1.5	90
10	4	0.5	70
11	4	0.5	80
12	4	0.5	90
13	4	1	70
14	4	1	80
15	4	1	90
16	4	1.5	70
17	4	1.5	80
18	4	1.5	90
19	5	0.5	70
20	5	0.5	80
21	5	0.5	90
22	5	1	70
23	5	1	80
24	5	1	90
25	5	1.5	70
26	5	1.5	80
27	5	1.5	90

2.4. Testing methods and machines:

Staining on Nylon fibers was evaluated by colorfastness tests, i.e. Colorfastness (CF) to washing, fastness to perspiration, and fastness to water tests. Tests were conducted as per standard defined by ISO (International Organization for Standardization). Fabric samples were cut according to the ISO recommended sample size of 10 x 4 cm. Launder-Ometer machine (MODEL L0) from Atlas Material Testing Technology USA was used to conduct colorfastness to washing test. Perspiration tester (MODEL PR1, Atlas Material

Testing Technology, USA) was used to conduct colorfastness to water and perspiration. Samples were dried in an Oven from MEMMERT. Test methods for washing, perspiration, and water were ISO 105-C06, ISO 105-E04, and ISO 105-E01, respectively.

3. Results and Discussion:

Three replicate results are shown under each colorfastness test in Table 3. Statistical analysis was done against CF washing, CF water, and CF perspiration. R square value, P-value, main effect plot, Pareto chart, and 3D surface graph were plotted to explain results statistically. All the results were evaluated by using statistical software MINITAB. The R-square value was also checked against all tests. The value of R-square is always between 0 and 100%. If its value is 0% then it indicates that the model explains no inconsistency of the response data around its mean. 100% indicates that the model explains all the variability of the response data around its mean.

Table 3: Variables and colorfastness results

Sr. #	Shade Depth	Conc.	RC temp.	CF Washing 1	CF Washing 2	CF Washing 3	CF Water 1	CF Water 2	CF Water 3	CF Perspiration 1	CF Perspiration 2	CF Perspiration 3
1	3	0.5	70	4	4	4	3	3	3	3	3	3
2	3	0.5	80	4	4	4.5	4	4.5	4	4	4	4.5
3	3	0.5	90	4	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5
4	3	1	70	4.5	4.5	4.5	4.5	4.5	4	4.5	4	4.5
5	3	1	80	4.5	4.5	4	4	4.5	4.5	4.5	4	4
6	3	1	90	4.5	4.5	4	4.5	4.5	4.5	4.5	4.5	4.5
7	3	1.5	70	4.5	4	4	4.5	3	3	3	3	4.5
8	3	1.5	80	4.5	4.5	4.5	4.5	4	4.5	4.5	4.5	4.5
9	3	1.5	90	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5
10	4	0.5	70	4	3.5	4	3	3	3	3	3	3
11	4	0.5	80	4	4	4	4	4	4	4	4	4
12	4	0.5	90	4.5	4.5	4	4.5	4.5	4.5	4.5	4.5	4.5
13	4	1	70	4.5	4.5	4.5	4.5	4.5	4	4	4.5	4.5
14	4	1	80	4.5	4.5	4.5	4	4.5	4	4.5	4	4
15	4	1	90	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5
16	4	1.5	70	4.5	4	4	4	3.5	3	3	3	4
17	4	1.5	80	4.5	4.5	4.5	4	4.5	4	4.5	4	4
18	4	1.5	90	4	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5
19	5	0.5	70	4	3.5	3.5	3	3	3	3	3	3
20	5	0.5	80	4.5	4.5	4	4	4.5	4.5	4.5	4.5	4
21	5	0.5	90	4.5	4	4.5	4.5	4.5	4	4.5	4.5	4.5
22	5	1	70	4	4.5	4	4.5	4	4	4	4	4.5
23	5	1	80	4.5	4.5	4.5	4	4.5	4.5	4.5	4.5	4
24	5	1	90	4.5	4	4.5	4.5	4.5	4.5	4.5	4.5	4.5
25	5	1.5	70	3.5	4.5	4	4	3	3	3	3	4
26	5	1.5	80	4.5	4.5	4	4	4.5	4	4	4.5	4
27	5	1.5	90	4.5	4	4.5	4.5	4.5	4.5	4.5	4.5	4.5

3.1. CF Washing

The r square value of Nylon against colorfastness to washing was 55.82%. In Table 4, P-values are shown against each factor, their two-way and three-way interaction. We can see from the table that P-value for concentration and RC temperature is less than 0.05 that makes it highly significant, while P-values for shade depth are higher than 0.05, which means that it does not have any significant effect on results. P-value of two-way interaction between concentration and RC temperature is also significant.

Table 4: Analysis of variance table for Nylon (CF Washing)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	28	3.90741	0.13955	2.35	0.004
Blocks	2	0.07407	0.03704	0.62	0.540
Linear	6	2.50000	0.41667	7.01	0.000
Shade depth	2	0.12963	0.06481	1.09	0.344
Conc.	2	1.24074	0.62037	10.43	0.000
RC Temp.	2	1.12963	0.56481	9.50	0.000
2-Way Interactions	12	1.11111	0.09259	1.56	0.134
Shade depth*Conc.	4	0.18519	0.04630	0.78	0.544
Shade depth*RC Temp.	4	0.29630	0.07407	1.25	0.303
Conc.*RC Temp.	4	0.62963	0.15741	2.65	0.044
3-Way Interactions	8	0.22222	0.02778	0.47	0.874
Shade depth*Conc.*RC Temp.	8	0.22222	0.02778	0.47	0.874

The individual effect of each level against each factor was also checked by plotting the main effect plot Figure 1. Staining results with 1g/l of thiourea dioxide are showing the best results in comparison with 0.5 g/l and 1 g/l. The effect of RC temperature is directly proportional to staining results. As we are increasing the temperature of RC, the staining value is increasing. To check the most significant factor among the three factors, the Pareto chart is drawn and is displayed in Figure 2. The factors which are crossing the reference line towards the right in the chart were the most significant. Concentration and RC temperature were crossing the reference line respectively, meaning that these factors were statistically significant. Interaction of RC temperature and concentration of Thiourea dioxide has also a significant effect on results. Correlation between significant factors was checked by plotting 3D surface graph displayed in Figure 3. 1g/l of thiourea dioxide is showing the best staining results in comparison with 0.5 g/l and 1.5 g/l even at low temperature. There is no need to increase the concentration of thiourea dioxide from 1g/l as it shows the stripping behavior above that concentration.

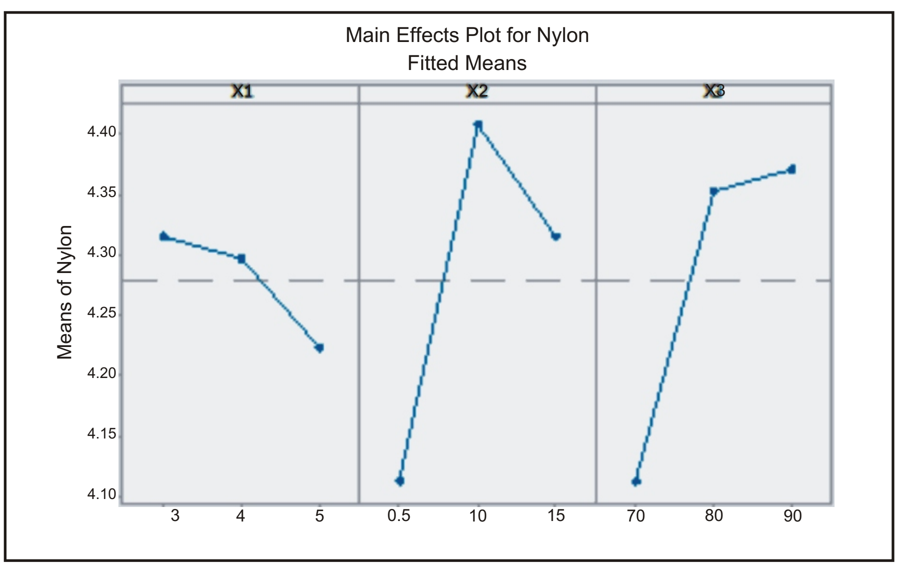

Figure. 1: Main effect plot for Nylon (CF Washing)

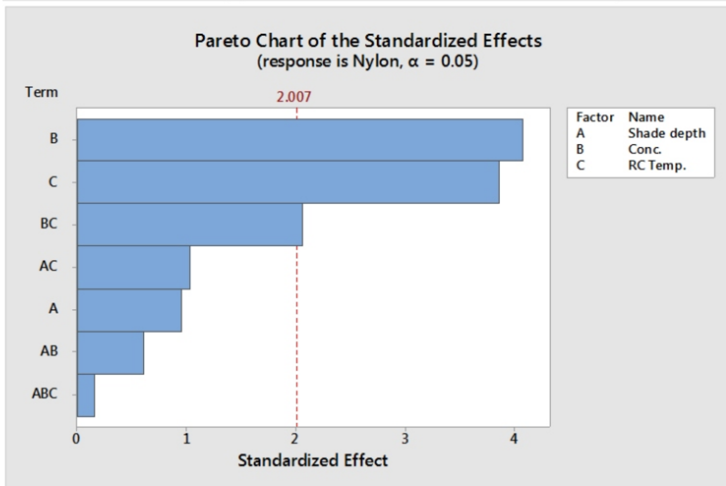

Figure 2: Pareto chart for Nylon (CF Washing)

3.2. CF Water:

The r square value of Nylon against colorfastness to washing was 82.43%. In Table 5, P-values are shown against each factor, their two-way and three-way interaction. We can see from the table that P-value for concentration and RC temperature was less than 0.05 that makes it highly significant while P-values for shade depth were higher than 0.05, which means that it does not have any significant effect on results. P-value of two-way interaction between concentration and RC temperature is also significant.

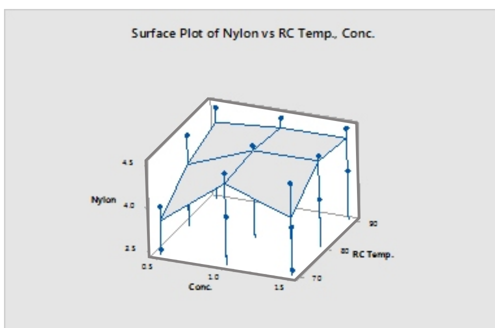

Figure 3:
3D Surface plot for Nylon (CF Washing)

Table 5: Analysis of variance table for Nylon (CF Water)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	28	20.2407	0.72288	8.71	0.000
Blocks	2	0.3519	0.17593	2.12	0.130
Linear	6	15.0370	2.50617	30.20	0.000
Shade depth	2	0.0556	0.02778	0.33	0.717
Conc.	2	3.1852	1.59259	19.19	0.000
RC Temp.	2	11.7963	5.89815	71.08	0.000
2-Way Interactions	12	4.7037	0.39198	4.72	0.000
Shade depth*Conc.	4	0.0370	0.00926	0.11	0.978
Shade depth*RC Temp.	4	0.2037	0.05093	0.61	0.655
Conc.*RC Temp.	4	4.4630	1.11574	13.45	0.000
3-Way Interactions	8	0.1481	0.01852	0.22	0.985
Shade depth*Conc.*RC Temp.	8	0.1481	0.01852	0.22	0.985

The individual effect of each level against each factor was also checked by plotting the main effect plot (Figure 4). Staining results with 1g/l of thiourea dioxide have shown the best results in comparison with 0.5 g/l and 1 g/l. The effect of RC temperature was again directly proportional to staining results. As we are increasing the temperature of RC, the staining value is increasing. To check the most significant factor among the three factors, the Pareto chart is drawn again (Figure 5). The factors which are crossing the reference line towards the right in the Pareto chart were the most significant. RC temperature, the interaction of concentration with RC temperature, and concentration alone were crossing the reference line respectively means that these factors are statistically significant. Interaction of RC temperature and concentration of Thiourea dioxide had also a significant effect on results. Correlation between significant factors was checked by plotting a 3D surface graph (Figure 6). 1g/l of thiourea dioxide showed the best staining results in comparison with 0.5 g/l and 1.5 g/l even at low temperatures. As before, there is no need to increase the concentration of thiourea dioxide from 1g/l as it shows the stripping behavior above that concentration.

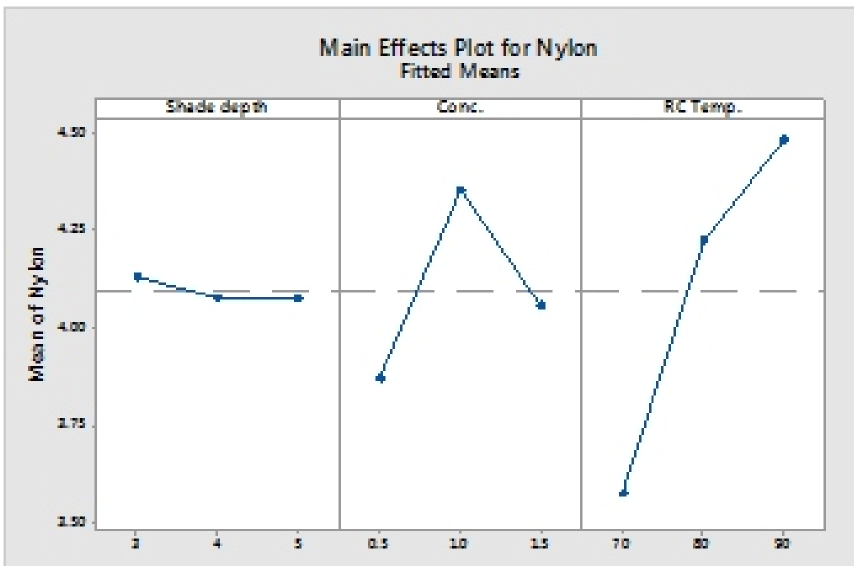

Figure 4: Main effect plot for nylon (CF Water)

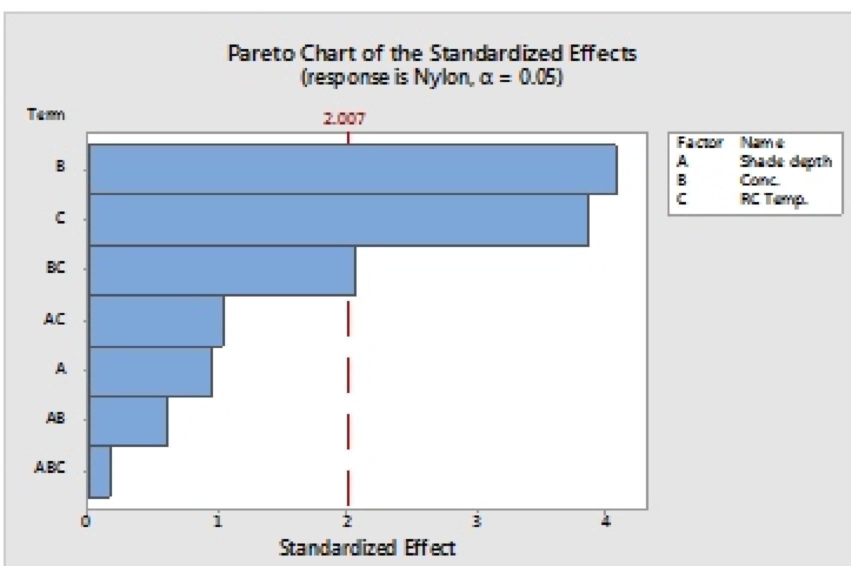

Figure 5: Pareto chart for Nylon (CF Water)

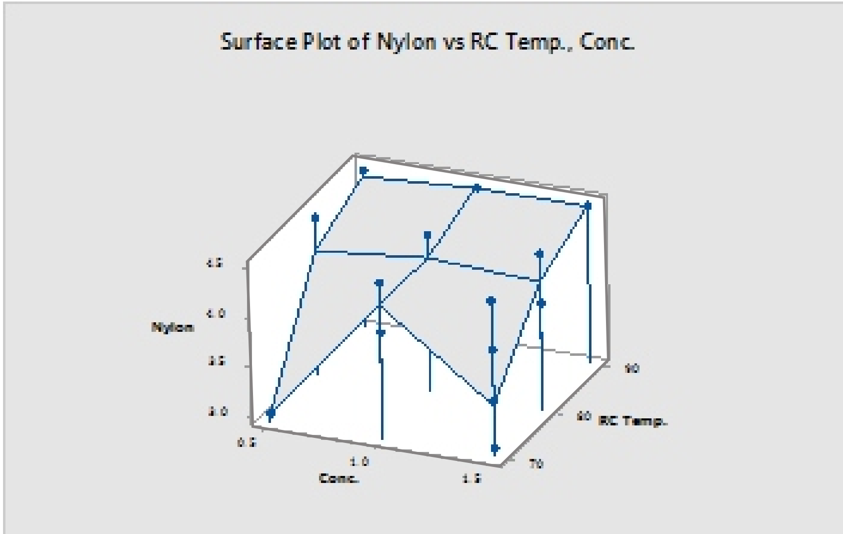

Figure 6: 3D Surface plot for Nylon (CF Water)

3.3. CF Perspiration:

The r square value of Nylon against colorfastness to washing was 83.33%. In Table 6, P-values are shown against each factor, their two-way and three-way interaction. The P-value for concentration and RC temperature was less than 0.05, while P-values for shade depth was higher than 0.05, which means that it did not have any significant effect on results. As before, the P-value of two-way interaction between concentration and RC temperature was also significant.

Table 6: Analysis of variance table for Nylon (CF Water)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	28	21.2963	0.76058	9.29	0.000
Blocks	2	0.2407	0.12037	1.47	0.239
Linear	6	15.5185	2.58642	31.58	0.000
Shade depth	2	0.0741	0.03704	0.45	0.639
Conc.	2	2.7222	1.36111	16.62	0.000
RC Temp.	2	12.7222	6.36111	77.66	0.000
2-Way Interactions	12	5.3519	0.44599	5.44	0.000
Shade depth*Conc.	4	0.1481	0.03704	0.45	0.770
Shade depth*RC Temp.	4	0.1481	0.03704	0.45	0.770
Conc.*RC Temp.	4	5.0556	1.26389	15.43	0.000
3-Way Interactions	8	0.1852	0.02315	0.28	0.969
Shade depth*Conc.*RC Temp.	8	0.1852	0.02315	0.28	0.969

The individual effect of each level against each factor was again checked by plotting the main effect plot and is displayed in Figure 7. Staining results with 1g/l of thiourea dioxide again showed the best results. The effect of RC temperature was again directly proportional to staining results. The factors which crossed the reference line towards the right in the Pareto chart (Figure 8) were the most significant. RC temperature, the interaction of concentration with RC temperature, and concentration were crossing the reference line respectively, meaning that these factors were statistically significant. Interaction of RC temperature and concentration of Thiourea dioxide had also a significant effect on results. Correlations between significant factors were checked by plotting a 3D surface graph (Figure 9). 1g/l of thiourea dioxide showed the best staining results once again, as compared to 0.5 g/l and 1.5 g/l even at low temperature. There is technically no need to increase the concentration of thiourea dioxide from 1g/l, as it showed the stripping behavior above that concentration. From the above figures, the most significant factor in enhancing the CF washing of Nylon was Concentration. RC temperature is the second most significant factor, whereas, the most significant factor in enhancing the CF Water and CF Perspiration of Nylon was RC temperature. Interaction of RC temperature with concentration and concentration itself were the second and third most significant factors, respectively.

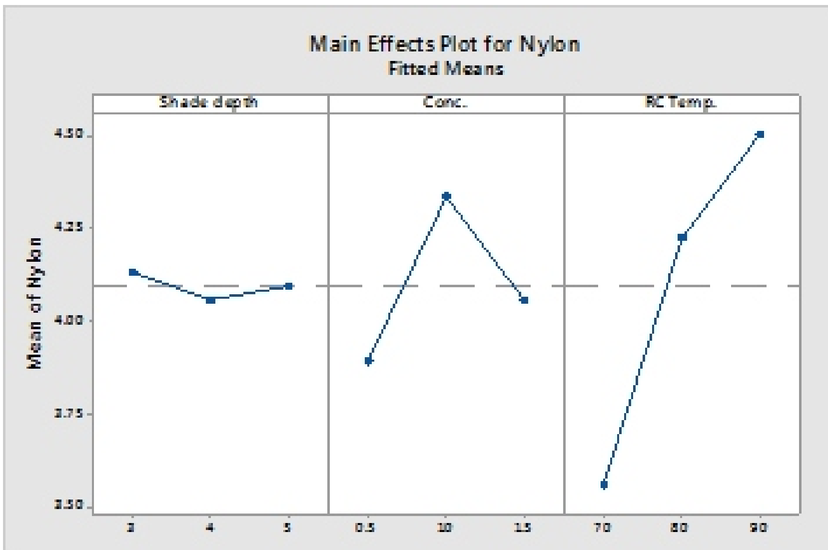

Figure 7: Main effect plot for nylon (CF Perspiration)

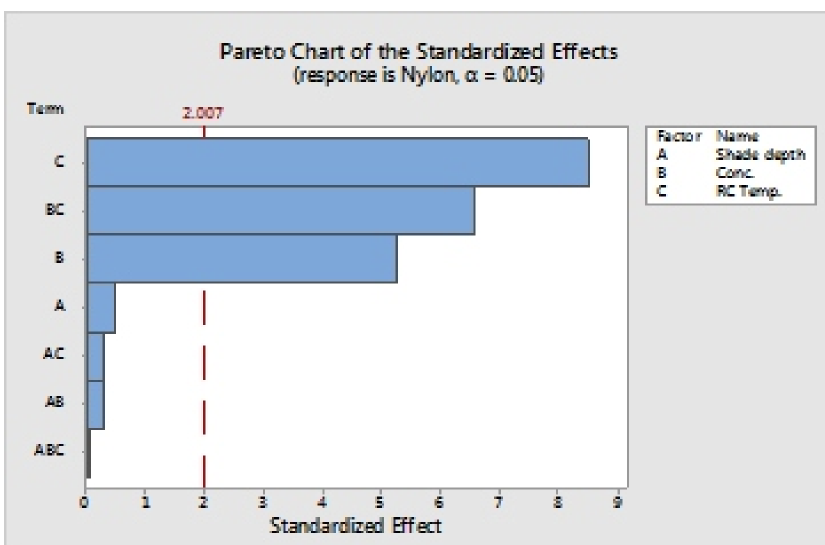

Figure 8: Pareto chart for Nylon (CF Perspiration)

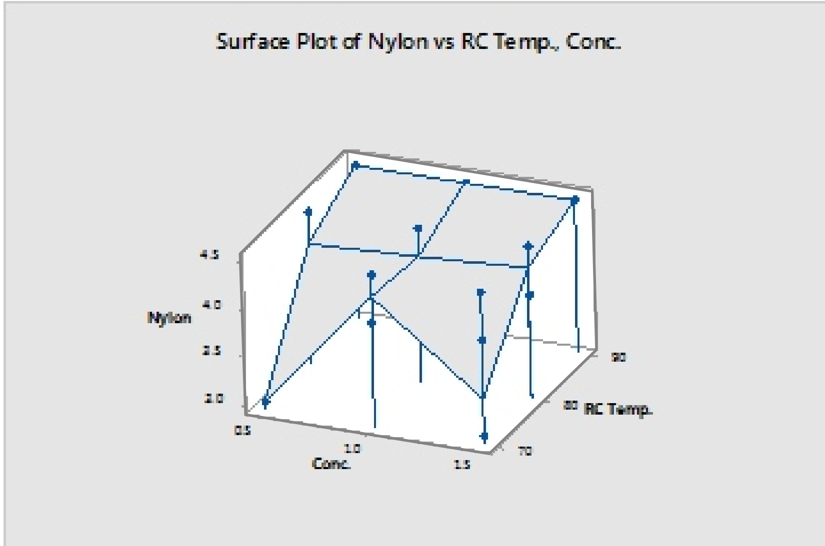

Figure 9: 3D Surface plot for Nylon (CF Perspiration)

4. Conclusion:

Staining on Nylon fibers was observed by CF washing, CF water, and CF perspiration through greyscale rating. All three factors were evaluated against each test, so it is easy for processing managers in the industry to choose the most significant factor to enhance fastness properties. Shade depth was the only factor among all three factors whose P-value was not significant. Concentration and RC temperature were the significant factors in all three tests. Stripping action was observed when the concentration of Thiourea dioxide was increased from 1 g/l to 1.5 g/l because staining results with 1g/l were better than at 1.5g/l. The effect of RC temperature was directly proportional to staining. As we increased the temperature of RC, staining increased. Conclusively, the most significant factor in enhancing the CF washing of Nylon was concentration. RC temperature was the second most significant factor. The most significant factor in enhancing the CF water and CF perspiration for Nylon was RC temperature. Interaction of RC temperature with concentration and concentration alone were the second and third most significant factors, respectively.

5. Acknowledgment

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