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Effect of Corona Treatment Time on Surface Morphology and Dyeability Properties of Cotton Fabric with Reactive Dye

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Abstract

In this paper was investigated dyeability cotton fabric sample using surface modification to corona discharged method. At first. In accordance with the design of experiments. We exposed the samples to Corona conditions with power of 100 (w) and the main current was 4A applied, at 60 and 300 secs and then dyed with Reactive Yellow K-RN Dyes. Then, scanning electronic microscope(SEM) was used to determine the effects of this operation on the surface morphology and also, a reflective spectrophotometer was employed to evaluate K/S, %R and L* a* b* values. Experiments indicated that dye stability of the samples were good and but with increased time of Corona after washing it was decline. Also, results showed that greater time of Corona operation at a constant pressure, enhanced cracks and pores on the surface morphology of the samples. As, Fourier transform infrared (FTIR) spectroscopy test displayed the effects of the Corona operation on the determination of Surface changes as well as changes in the existing functional groups in fabric after being wash and finally, the optimal conditions of the experiment were determined using the statistical method of Central Composite Design. In fact, aim of this study was to investigate the dyeing properties of cotton fabric and surface morphology changes using corona operation and to obtain optimal dyeing conditions. The results obtained in this research illustrated that optimize condition of cotton fabric samples dyed with the Reactive Yelow3 dye for K/S1 and K/S2 at F-value respectively, equal to 29879.53 and 15940.10, and probe values greater than F is equal to < 0.0001.

Keywords: Cotton fabric, Corona discharge, Reactive Yellow KRN, FTIR, Reflective

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Spectrophotometer.

1. Introduction

The Cotton fiber that is known as one of the natural fibers enjoys a very complex structure and chemical processes have a widespread utilization to modify the fibers' surfaces for increase of dyeability. Moreover, researchers have shown the extensive application of Corona discharge for modifying the fibers' surfaces. Results indicated simpler treatment and higher practicality than any other physico-chemical techniques due to the quick treatment of the samples under atmosphere pressure [1]. Furthermore, researchers have demonstrated suitability of the corona operation as one of the dry methods. Therefore, they identified the plasma operation as one of the efficient techniques to promote the surface properties employed for various kinds of textile reform [2]. Numerous investigations have been done on the surface modified process of cotton as well as other fibers with corona discharge treatment, including wool and polyester, the impact of the treated time, treated voltages and powers on the features of cotton fibers, and the surface modified processes [3-6]. This method improves the surface with changing the bulk properties in a dry system without chemicals and water. The attention has been paid to improve the wettability, friction, adhesion, reflection of light, water repellency, soil release, printing, dyeing and other finishing process of textile fibres and fabrics by using corona technology [7]. Nourbakhsh et all [8] investigated the newly proposed corona discharge treatment of the cotton fabric with Cu and ZnO nanoparticles (NPs) and demonstrated the enhanced adsorption of copper NPs through pre-treatment method of corona discharge as well as greater self-cleaning effect and anti-bacterial function of the copper NPs than the post-treatment. Moreover, ZnO NPs exhibited the greatest anti-bacterial and self-cleaning effects using the pre-treatment technique. It is notable that the use of the post-treatment declined photo-catalyst activities of the ZnO NPs. Brzeziński et.al [9] founded that modifying the PET fabrics with corona discharge increased the fibers' surface energy and wettability of the activated samples. Research also has shown that greater ratio of oxygen to carbon indicates the formation of multiple functional polar groups in the top fibre layer of the PET fabrics, which have been activated with corona discharge. Because of development of the fibers' specific surfaces and removal of the direct contact between the negative charges accumulated on the surfaces of the activated PET fabric as well as the utilized bonding agents, adhesive ability of the fabrics remarkably enhanced, which has been revealed by the significant enhancement in the delamination force. The aim of this study was to investigate the dyeing properties of cotton fabric and surface morphology changes using corona operation and to obtain optimal dyeing conditions.

2. Experimental

2.1. Materials and Methods

At first, before Corona treatment, we selected 48 gr of the bleached cotton fabric in sizes of 50 ×50 cm2 from BABAKAN Textile Company and then washed the fabric with hot water and LCF-123 Jintery Eco anionic detergent at 60 ºC for 15 min for removing contamination from the fabric; finally dried the fabric at 80 ºC in a dryer for 1 hour.

2.2. Corona discharge treatment

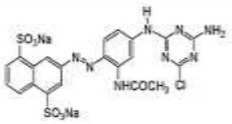
In this unit, the two sample of fabrics at sizes of 5 × 5 cm^2 were put at a vacuum and corona conditions for one min and the other 5 min. Corona conditions included a system power of 100 (w) and the main current was 4A applied. Figure 1 shows the schema of the above system.

Figure.1. Equipment employed for performing the corona treatment. 1) Power supply, 2) Container Operation, 3) Roller, 4) Electrode spark production corona, 5) Electrode distance regulator screws up roller, and 6) Electro motor [2].

2.3. Dyeing process

At this step, sample was taken through 2 % of Reactive dye; that is, yellow K-RN (Reactive yellow 3) (Table.1). Tehran Dye Research Center was chosen to supply the Reactive dyes. The pre-determined L:R was 20:1 and therefore we examined the procured samples for evaluating the effects of Corona condition on determining Lab, light reflection rate (R %) as well as K/S and determining the fabrics' washing fastness.

Table .1. Molecular structure and properties of the reactive dye known as the reactive yellow K-RN [13,14]

Name of Color Index	Molecular Structure	Molecular Formula	Image of Molecular structure	Molecular Weight	Ligth fastness
C.I.Reactive Yellow 3,C.I.13245 Reactive Yekkow K-RN	Single Azo Class	$C_{21}H_{15}ClN_8Na_2O_7S_2$		636.86	6 to 7

2.4. SEM

Pieces of experimented samples of 1 × 1 cm^2 size were cut and placed in the Sputter Coater machine for coating the samples with a gold layer. In the next step, we scanned the Corona-treated and untreated dyed samples for both samples with using of SEM instrument, model LEO440 made in England, for determining the effects of Corona condition on the fabrics' surface morphology.

2.5. Washing fastness of the samples

For this stage, we washed those samples with a weight of 5 g, which have been treated with Corona and dyed with the reactive dye at the exposure times of one and five minutes 5 times at 80 ºC in a soap bath consisting of 2 gr/L anionic soap and 4 gr/L sodium hydroxide (NaOH) with 40:1 ratio of bath mass on the fabric weight for 30min. Next, we dried the samples in a dryer for one hour and measured the Lab, % R rate as well as K/S of the washed samples with a reflective spectrophotometer machine for determining the fabrics' colour fastness.

2.6. Reflective spectro-photometer

As mentioned earlier, spectro-photometer ColourEye-7000A system with UV D65 standard light source and d/8º geometrical feature was employed for measuring the samples' Lab R % rate and K/S. In fact, the experiment was performed for determining the fabric's color fastness due to the exposure to nitrogen (N) gas cold plasma, prior to and following the washing process.

2.7. FTIR

All the samples were placed in the machine jaw for experimenting the entire level of the samples. After that, OMNIC software was employed for analyzing the samples and the experiment was operated in a machine known as Thermo Nicollet 807 fabricated in America for proving reliability of the reflective spectrophotometer test in order to specify the fabric's color and washing fastness.

2.8. Experimental design

In this step, Central Composite Design (CCD) was utilized for the experimental plan with 2 variables [10]. These two variables included the percentage of Reactive dye and amount of irradiation time(sec). Findings revealed the variables range for irradiation time between 55.78 and 300sec) and percentage of Reactive dye (0.12–0.98%), through the trial version of the Design Expert 8.0.1.0 software provided from the Stat-Ease, Inc.(USA). Table (2) (runs 1–8) completely reports the design process of the samples of Cotton fabric with the dye percentage and the irradiation time. Results reflected the influence of the variables on the Y_1 (K/S) and Y_2 (R) outputs adjusted by the third-order polynominal function (Equation 1). Though we chose the samples for 3 series of time under radiation, softness of the range of changes was randomly chosen for ranges between 4 and 12 min of radiation in 8 distinct runs. Put differently, samples with four iterations were used to perform the laboratory experiments:

$$Y = b_0 + \sum b_i X_i + \sum b_{ij} X_i X_j + \sum c_i X_i^2 \qquad i \geq j \qquad \text{Eq. 1}$$
$$i,j = 1,2,3$$

Here, b_0 stands for an independent term according to the mean value of the experimental plan. Bi represents the regression coefficient, explaining the effects of variables on the linear form. bij is the regression coefficients of the interaction terms between the variables. C_i refers to the coefficients of the variables quadratic form.

Table 2. CCD for K/S and R of the fabric samples treated with Reactive dye at various irradiation times.

Run	Factor 1:	Factor 2:	Response 1	Response 2	Response 3	Response 4
	A: Time treat (sec)	B: Reactive dye (%)	K/S1	K/S2	R1	R2
Control 1	60	0	Invisible	Invisible	Invisible	Invisible
Control 2	0	1	5.28	5.15	11.54	11.48
1	206.71	0.12	0.3	0.3	1.6	2.0
2	165.00	0.55	0.9	0.7	4.0	5.1
3	55.78	0.81	2.2	1.6	9.8	12.3
4	206.71	0.98	2.9	2.1	12.8	16.2
5	165.00	0.55	0.9	0.6	3.9	5.1
6	300.00	0.55	1.6	1.2	7.2	9.2

7	165.00	0.55	0.9	0.6	3.9	5.1
8	55.78	0.29	0.8	0.6	3.5	4.4

3. **Results and discussion**

With regard to Figure 4, Comparison of the curves of the raw sample dyed with reactive without Corona treatment with sample of dyed and Corona treated at time 1 and 5 min. The Corona conditions show that before washing rate of K/S decrease suddenly but after washing decrease gradually (a and c). Also, that before washing rate of %R decrease suddenly but after washing increase again (b and d). Table 3 indicated that comparison of K/S, %R and CIELab between samples of untreated and treated with Corona before and after washing, respectively.

Table 3. Comparison of K/S, %R and CIELab between samples of untreated and treated with Corona before and after washing, respectively.

Sample	Untreated	Before washing		After washing	
Time of Corona		**1min**	**5min**	**1min**	**5min**
λ_{max}(nm)	425	375	375	425	425
K/S	5.28	2.92	2.9	2.16	2.1
%R	11.54	13.02	13.06	16.34	16.78
L*	80.51	49.27	49.45	79.47	78.34
a*	13.12	21.77	22.18	8.23	7.32
b*	67.59	9.96	10.32	48.93	46.23
c*	68.85	23.94	24.47	49.62	46.80
h°	79.01	24.59	24.95	80.45	81.00

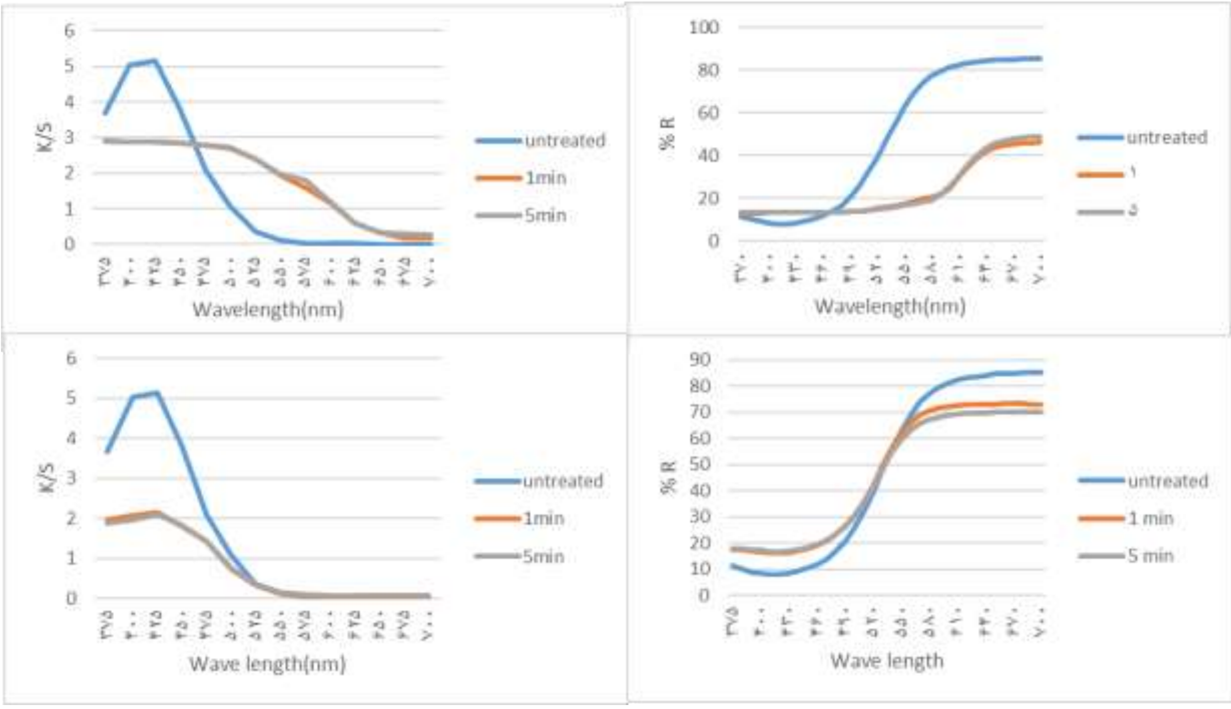


Figure 4. Comparison of graphs between :

a) Graph of K/S sample of untreated and treated with Corona before washing
b) Graph of %R sample of untreated and treated with Corona before washing
c) Graph of K/S sample of untreated and treated with Corona after washing
d) Graph of %R sample of untreated and treated with Corona after washing

As shown in Table 4, the value of the adsorption coefficient to the diffusion (K/S) of the dye raw sample compared to the sample treated with corona (1 and 5 min) and dyed, shows a sudden decrease, which for both before and after Washing is observed to be almost identical, while for values of the reflectance spectrum (%R) this value shows a gradual increase over the dyed raw sample before and after washing. This indicates that the corona operation and degradation of the surface morphology of cotton fibers cause less dye adsorption due to the plasticizer phenomenon and low dye penetration into the fibers, while the color wash stability is improved due to the hydrogen and covalent bonds between reactive dye and cotton fibers. Scheme.1, indicated the mechanism of Corona discharge and dyeing with Reactive K-RN treatments on cotton fiber.

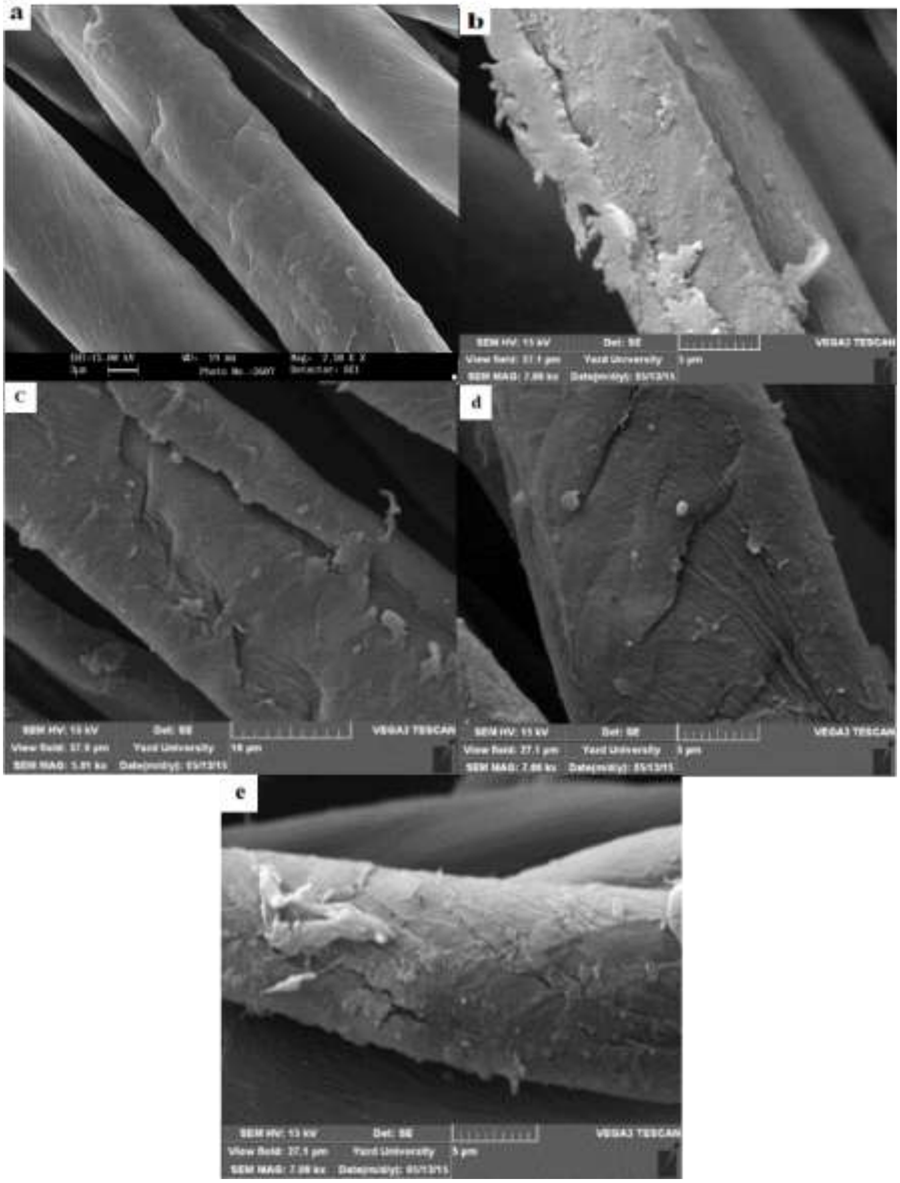
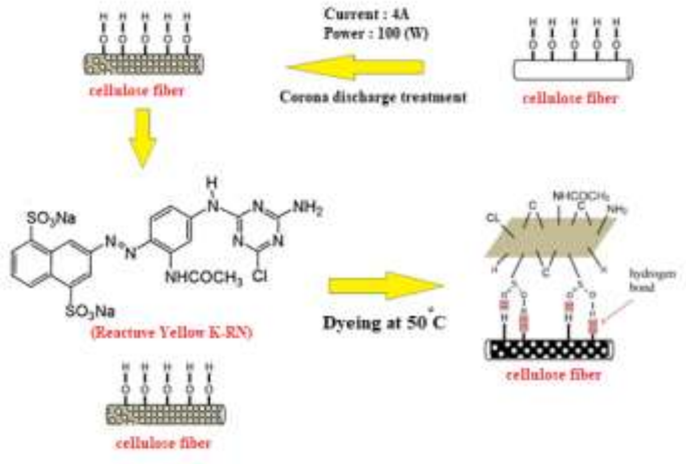


Figure 5. Image of SEM : a) Raw b) treated with corona at time 1 min, c) treated with corona at time five minutes, d) treated with corona at time 1 min and then dying e) treated with corona at time 5 min and then dying.

Fig.5, demonstrates SEM images obtained from the Raw samples' surfaces, which were treated with Corona at 1 and 5 min times through an electronic microscope, indicating differences in the surface morphology. As seen, if the samples are more exposed to the plasmatic condition, they would have more effects of destructive condition on them (b and c). The microscopic images of the fibers in figures (d) and (e) show well the uniform placement of the dye in the pores of the fibers created by the corona operation, and finally we see a smooth surface on the surface of the fibers, which is consistent with our previous results.



Scheme 1. The proposed mechanism of Corona discharge and dyeing with Reactive K-RN treatment on cotton fiber.

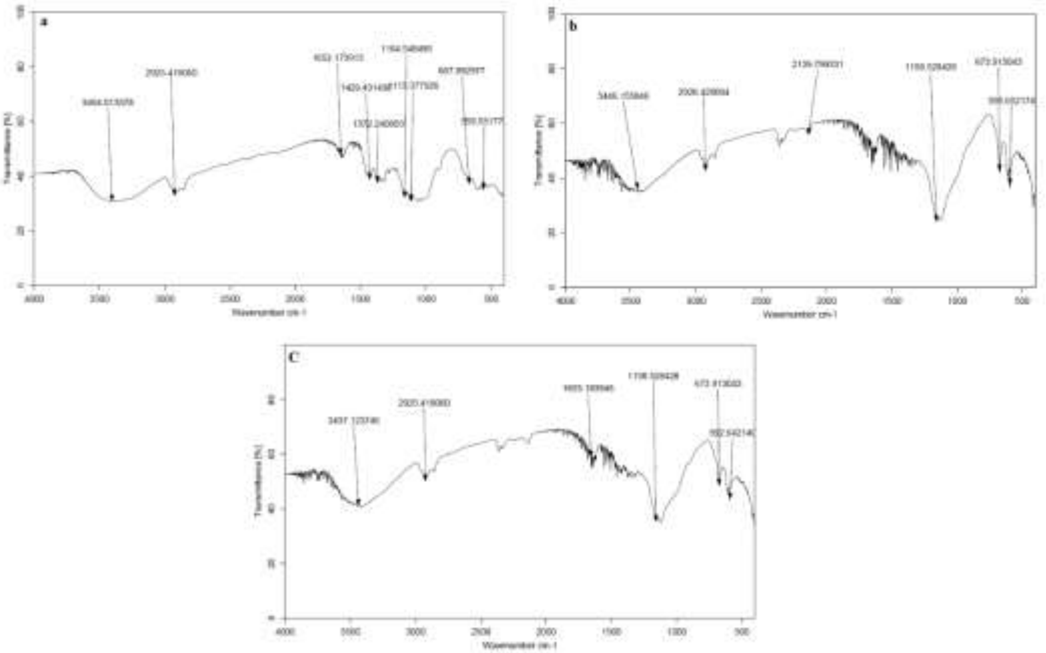


Figure 6. FTIR for : a) Raw b) treated with corona at time 1 min and then dying c) treated with corona at time 5 min and then dying

Studying Fig (6) as well as the peaks from the existing functional groups in the samples did not show any significant changes [14] so that CH stretching absorption in 3000 cm^{-1} 2850 cm^{-1} regions, ketone absorption in 1720 cm^{-1} region, and that of the sulphates in 3000 cm^{-1} and 2850 cm^{-1} regions are the same for each dyed sample of the treated and untreated samples with Corona. However, lower absorption was seen in 2350 cm^{-1} region on nitrile and in 650-720 cm^{-1} region of the stretching CH alkene or alcohol. With the enhanced operation time of Corona, peak also enhanced. Put differently, we found stronger absorption in 1150-1165 cm^{-1} region of sulphonic acid, indicating greater treatment time of Corona with increasing the peak. Hence, the fabrics dye fastness following the washing stage would be reasonable and for the taken samples. In fact, Corona condition was shown to not involve in the failed creation of washing fastness in the fabric in comparison with the untreated samples, reflecting minor effect of the operation on the K/S values and reflective spectrum.

3.1. Statistical ***analysis***

Optimization of the application of the NPs reflected considerable involvement in the better function of the textile fabrics. In contrast to the conventional optimization, it is possible to consider statistical optimization interactions and processes between the variables to create a procedure response. In addition, RSM was demonstrated to be a strong statistical method to analyze numerous variables because it involves fewer crucial experimental trials as compared to the "one-factor-at-a-time" technique. On the one hand, RSM was considered as an efficient mathematical approach for optimizing the complex processes with the capability of producing an empirical pattern for evaluating correlations of several controlled experimental parameters with the observed results. On the other hand, RSM has a widespread utilization in various bio-chemical, nanochemical as well as chemical methods to examine the effect(s) of independent variables and optimize the process responses using proper values of the parameters [10–12]. It could be said that this study was performed according to RSM and CCD. Therefore, 8 experimental CCD runs were designed (see Table 2). We also addressed the evaluation of, the impact(s) of independent variables such as treat time and Reactive dye percentage on the response surface and showed %R and K/S features of the treated fabric samples. At the end, Equations (2), (3), (4), and (5) give actual factors of the K/S and %R of the fabric samples, which were treated with Corona treatment and Reactive dye:

$$K/S_1 = +1.40565 - 0.012097\ \text{Time treat} - 1.31523\ \text{Reactive Yellow 3} + 0.001825\ \text{Time treat} * \text{Reactive Yellow 3} + 0.000035\ \text{Time treat}^2 + 3.50813\ \text{Reactive Yellow 3}^2 \quad \text{Eq(2)}$$

$$K/S_2 = +1.03158 -- 0.008928\ \text{Time treat} - 0.965332\ \text{Reactive Yellow 3} + 0.001521\text{Time treat} * \text{Reactive Yellow 3} + 0.000026\text{Time treat}^2 + 2.53548\ \text{Reactive Yellow 3}^2 \quad \text{Eq(3)}$$

$$R_1 = +6.27415 - 0.054369\ \text{Time treat} - 5.68116\ \text{Reactive Yellow 3} + 0.008027\ \text{Time treat} * \text{Reactive Yellow 3} + 0.000159\ \text{Time treat}^2 + 15.56296\ \text{Reactive Yellow 3}^2 \quad \text{Eq(4)}$$

$$R_2 = +7.79478 - 0.067927\ \text{Time treat} - 6.87782\ \text{Reactive Yellow 3} + 0.011527\ \text{Time treat} * \text{Reactive Yellow 3} + 0.000199\ \text{Time treat}^2 + 19.22187\ \text{Reactive Yellow 3}^2 \quad \text{Eq(5)}$$

Therefore, we employed the final statistical model for drawing the response surface (Equations 2–5), and in this way determined the relationship of each independent variable with K/S and %R of the fabric samples. Figures 7 and 8 represent the response surface of the samples of fabric.

Moreover, we applied Design-Expert software with the Corona time equal to 5 minutes or 300 sec for Reactive dye for obtaining optimum condition of the features of K/S and %R of the samples. In the next step, we used analysis of variance (ANOVA) for the process of data analysis in order to establish interaction of independent variables with the responses. Then, this test was employed for analyzing results for evaluating K/S and %R of the mentioned samples (Tables 4, 5, 6, 7).

Results reflected significance of this new model of the fabric samples with the Reactive Yelow3 for K/S_1 and K/S_2 at F-value equal to 29879.53 and 15940.10, and prob values greater than F, < 0.0001, (Tables 4, 5). Consequently, the model of the treated fabric samples with the Reactive Yellow 3 for $\%R_1$ and $\%R_2$ was shown to be significant at F-value equal to $\mathbf{6.006\times10^5}$ and $\mathbf{9.593\times10^5}$ with the probability values more than F< 0.0001, respectively (Tables 6, 7).

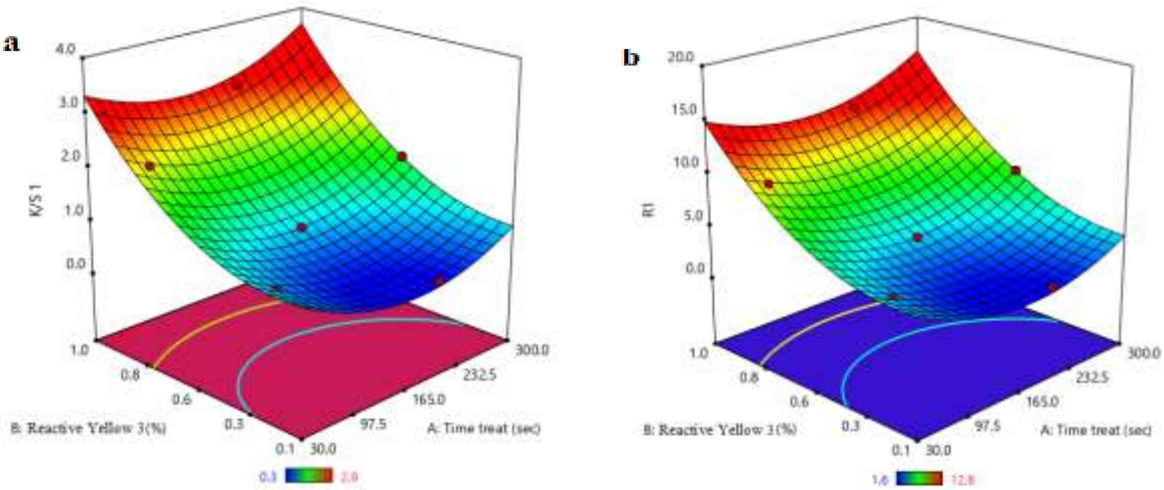


Figure 7. Response surface for a) K/S_1 and b) $\%R_1$ as the Corona time function and Reactive dye for the fabric samples.

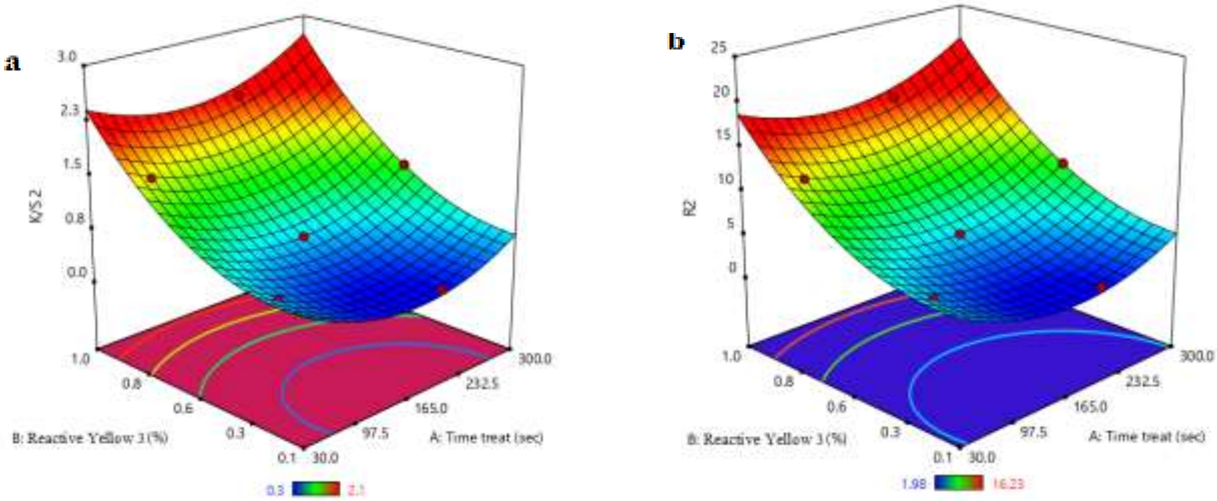


Figure 8. Response surface for a) K/S_2 and b) $\%R_2$ as the Corona time function and Reactive dye for the fabric samples.

Table 4. ANOVA result of K/S_1 for the fabric samples with Reactive yellow 3 at different Corona times

Source	Sum of Squares	df	Mean Square	F-value	p-value	prob > F
Model	4.98	5	0.9960	29879.53	< 0.0001	significant
A-Time treat	0.0136	1	0.0136	408.38	0.0024	
B-Cold dye	4.10	1	4.10	1.229×10^5	< 0.0001	
AB	0.0077	1	0.0077	230.49	0.0043	
A²	0.4428	1	0.4428	13284.57	< 0.0001	
B²	0.5406	1	0.5406	16218.81	< 0.0001	
Pure Error	0.0001	2	0.0000			
Cor Total	4.98	7				

Table 5. ANOVA Result of K/S_2 for the samples of fabric with Reactive yellow 3 at different Corona times

Source	Sum of Squares	df	Mean Square	F-value	p-value	prob > F
Model	2.66	5	0.5313	15940.10	< 0.0001	significant
A-Time treat	0.0125	1	0.0125	376.07	0.0026	
B-Cold dye	2.18	1	2.18	65377.40	< 0.0001	
AB	0.0053	1	0.0053	160.15	0.0062	
A²	0.2426	1	0.2426	7278.50	0.0001	
B²	0.2824	1	0.2824	8472.04	0.0001	
Pure Error	0.0001	2	0.0000			
Cor Total	2.66	7				

Table 6. ANOVA result of $\%R_1$ for samples of the fabric with Reactive yellow 3 at various Corona times.

Source	Sum of Squares	df	Mean Square	F-value	p-value	prob > F
Model	100.09	5	20.02	6.006×10^5	< 0.0001	significant
A-Time treat	0.2875	1	0.2875	8624.45	0.0001	
B-Cold dye	82.47	1	82.47	2.474×10^6	< 0.0001	
AB	0.1487	1	0.1487	4460.15	0.0002	
A²	9.00	1	9.00	2.699×10^5	< 0.0001	
B²	10.64	1	10.64	3.192×10^5	< 0.0001	
Pure Error	0.0001	2	0.0000			
Cor Total	100.09	7				

Table 7. ANOVA result of $\%R_2$ for samples of fabric with Reactive yellow 3 at various Corona times

Source	Sum of Squares	df	Mean Square	F-value	p-value	prob > F
Model	159.88	5	31.98	9.593×10^5	< 0.0001	significant
A-Time treat	0.7185	1	0.7185	21554.81	< 0.0001	
B-Cold dye	132.36	1	132.36	3.971×10^6	< 0.0001	
AB	0.3066	1	0.3066	9199.25	0.0001	
A²	14.05	1	14.05	4.214×10^5	< 0.0001	
B²	16.23	1	16.23	4.869×10^5	< 0.0001	
Pure Error	0.0001	2	0.0000			
Cor Total	159.88	7				

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4. Conclusion

In this research and according to the design of experiments by the statistical software of the Central Composite Design(CCD), sample of the bleached cotton fabric was exposed to Corona condition at times of 60 and 300 sec and then dyed using Reactive yellow 3. Then, we examined the effect(s) of Corona on the degradation/destruction of the surface morphology and employed SEM to assess them. In the next step, reflective spectrophotometer machine was used to show the outputs resulting from Corona condition in determination of K/S, b*, a*, L* and R % rates. According to the results, those samples dyed with the reactive dyes had dye fastness, though washing fastness partly declined following the Corona operation that may be neglected because ATR-FTIR test proved it. Moreover, images from the Corona effect on the surface morphology with the use of SEM showed the enhanced destruction with the greater Corona treatment time. Also, Design-Expert software with a Corona time equal to 300 sec for Reactive Yellow 3 dye was employed for obtaining an optimum condition of the K/S features and %R of the fabric samples.

References

[1] Pibo Ma, Juan Huang, Genyang Cao, and Weilin Xu, Influence of Temperature on Corona Discharge Treatment of Cotton Fibers, Fibers and Polymers, Vol.11, No.6, 941-945(2010).
[2] M. Mirjalili1, S.Sharif Nasirian and L.Karimi, Effects of corona discharge treatment on some properties of wool fabrics, African Journal of Biotechnology Vol.10(83), pp.19436-19443, 21 December,(2011).
[3] W. Xu, X. Shen, X. Wang, and G. Ke, Sen'I Gakkaishi, 62,111 (2006).
[4] W. Xu and X. Liu, Eur. Polym. J., 39, 199 (2003).
[5] D. Sun and G. K. Stylios, J. Mater. Process. Tech., 173, 172 (2006).
[6] H. A. Karahan and E. Ozdogan, Fiber. Polym., 9, 21(2008).
[7] Peiman Valipour, Ramin Khajavi, Sahar Belaj , Shirin Nourbakhsh, PHYSICAL AND MECHANICAL PROPERTIES OF CORONA DISCHARGE TREATED POLYESTER FABRIC, XIIIth International Izmir Textile and Apparel Symposium April 2-5, (2014).
[8] Sh.Nourbakhsh, H.Sepehrnia and E.Akbari, Novel corona discharge treatment of cotton fabric with Cu and ZnO nanoparticles, The journal of textile institute Vol 111,(2020).
[9] Stefan Brzeziński, Stefan Połowiński, Dorota Kowalczyk, Iwona Karbownik, Grażyna Malinowska. Effect of the Corona Discharge Treatment of Polyester Fabrics on their Adhesive Properties, FIBRES & TEXTILES in Eastern Europe, Vol. 17, No. 4 (75) pp. 98-102(2009).
[10] Nazari, A., Montazer, M., Mirjalili, M., Nazari, S.: Polyester with durable uv protection properties through using nano TiO2 and polysiloxane softener optimized by RSM. J. Text. Inst. 104, 511–520 (2013).
[11] Zarrinabadi Ehsan, Abghari Ramin, Nazari Ali, Mirjalili Mohammad: Environmental effects of enhancement of mechanical and hydrophobic properties of polyester fabrics using silica/kaolinite/silver nanocomposite: a facile technique for synthesis and RSM optimization. Eurasia J Biosci 12, 1–14 (2018).

[12]H.Akbarpour, A.Rashidi, M.Mirjalili, A. Nazari, Comparison of the conductive properties of polyester/viscose fabric treated with Cu nanoparticle and MWCNTs; Journal of Nanostructure in Chemistry,(2019).
[13] Reactive yellow dye.www.worlddyevariety.com.
[14] H.Akbarpour, A.Rashidi, M.E Yazdanshenas and H.Tayebi; Effect of Nitrogen Gas Cold Plasma on Cotton Fabric Dyed with Reactive Dyes, Asian Journal of Chemistry; Vol. 25, No. 2, 695-700,(2013).