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Optimization of dyeing process of wool with extract of saffron petals using response surface methodology

ABSTRACT

Recently the awareness of the demand on eco-friendly dyes in textile applications is increased, since the natural dyes can reveal better biodegradability and generally have a higher compatibility with the environment. There are ongoing attempts to overcome some disadvantages related to natural dyeing such as prolonged dyeing time, consumption of energy, and chemicals used. In this point of view, the optimization of process parameters is a good alternative. In this work, the dyeing of wool fibers with extract of saffron petals is presented. The response surface methodology is used to optimize the process parameters and to investigate the relationship between different factors taken into consideration, such as dyeing time, dye concentration, mordant amount, dyeing temperature and pH. The dye uptake was evaluated by absorbance measurements, using UV-Vis spectrophotometer and was used as response. The optimal dyeing conditions for obtaining the highest dye uptake value were as follows: dyeing time 69.89 minutes, dye concentration 3.7%, mordant amount 1.02g (0.56%) , dyebath pH 2.08, dyeing temperature 110^{o}C.

Key words: *natural dye, optimization, RSM, saffron extract, wool*

1. INTRODUCTION

Synthetic dyes are commonly used for the coloration of textiles because of wide variety of colors available with good fastness properties at a low cost and easy applications [1,2]. However, the synthetic dyes present in dyeing wastewater discharged into the environment cause health risks, and toxic and allergic reactions [3-5] to humans as well as threaten the ecosystems [1,2]. Global concern for environmental pollution has put rigorous rules forward for high level pollutant industries [6]. Textile industry with huge amount of hazardous wastewater production is one of such industries [7-9]. Germany has completely banned azo dyes [2]. The use of non-toxic, antimicrobial and eco-friendly natural dyes on textiles, and preferably natural fiber products, has become a matter of increasing importance due to the increased awareness of environmental impact caused by synthetic ones [7].

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Natural dyes are known as sustainable and environmentally friendly materials for dyeing and functional finishing of textiles [3]. They can be obtained from vegetable, animal or mineral origin [1-8]. They are non-carcinogenic, biodegradable [4] and biocompatible with the environment [5,6,8-12]. Due to this fact they are in high demand nowadays.

Saffron is a perennial plant which belongs to the genius *Crocus Sativus.* It is cultivated in warm climate countries like Italy, Iran, Spain, Greece, Turkey, Morocco, etc. [13-17]. Recently, it is cultivated also in Albania, in Elbasan region. After harvesting stigma, the petals produced in huge amount remain as a waste and are reused as a source of natural colorant.

Wool is the fibre from the fleece of the sheep. It is a natural protein with multiple cellular structure. The wool fibre is a crimped, fine to thick, regular fibre [11]. Wool primarily consists of keratin, which consists of long chains of amino acids linked together by peptide bonds. Within the keratin structure, various types of chemical bonds and intermolecular forces play a role in determining the properties of wool. These include disulfide bonds, ionic bonds, hydrogen bonds, and van der Waals forces. The combination of these bonding forces and interactions results in a three-dimensional network of intertwined keratin chains, forming the complex and unique structure of wool. This advanced structure provides wool with its desirable properties, such as elasticity, resilience, moisture absorption, insulation, and durability. Wool is recognized as a high-quality and mid-to-high-end fabric.

Natural dyes exhibit a high affinity for natural fibers, particularly for protein-based fibers [12]. However, there exist some disadvantages such as low color yield, prolonged time to dye, consumption of energy, dye, mordant and auxiliaries. Because of these disadvantages there is a need for optimization of natural dyeing [3]. Optimization of dyeing is definitely the substantial method to determine the optimum condition in order to get highest dyeing uptake exhaustion [14]. In the traditional method, one variable is examined at a time while the other is maintained constant. This method is time consuming and requires too much work [3]. Furthermore, this method may provide inaccurate outcomes, such as the belief that interaction effects cannot be observed, making it difficult to ascertain process parameters performance [13]. Response surface methodology is one of a perfect method to simulate the variables simultaneously. It has been widely used in process and product improvement because it can optimize the complex process and minimize the experimental number of trials [14]. The central composite design is widely used experimental design method due to the selection of appropriate pivot points and the retention of the rotatability and sequentiality of the experimental data, which greatly improves its prediction accuracy [15].

In this study, the dyeing conditions of wool fiber were optimized by response surface methodology. Extract of saffron petals is used as a natural dye source for coloration of wool fibers. Five independent factors including dye concentration, mordant amount, temperature, dyeing time and pH were selected as the most influencing factors according to preliminary experiments. The effect of these factors was examined on the dye uptake of the wool fibers. The optimum conditions were determined to achieve the maximum dye uptake percentage during the dyeing process.

2. MATERIALS AND METHODS

2.1 Wool fabrics:

The wool fabrics were sized in 4x10cm and prepared to be dyed with the pre-mordanting method using alum mordant. The method of mordanting and the alum mordant were determined by our previous experiments during the project.

2.2 Dye extraction:

Saffron petals were obtained from Victus Fed company. They were dried and prepared for extraction. The dye was extracted with traditional boiling method. The mixture was allowed to cool down and filtered to remove the petals. The extracted solution was kept in the dark box and further used in the dyeing process.

2.3 Dyeing process:

The dyeing experiments were performed according to the runs determined by RSM method. The proteinic fiber was dyed with saffron extracts with pre-mordanting method with liquor ratio of 1:45, while the other parameters were ranged as is showed in table 1. At the end, the absorbance of the extract remained in the dyeing bath was measured. The initial absorbance before the dyeing process was measured and the percentage of the dye uptake was calculated as the following equation:

$$E = \left(\frac{A_0 - A_f}{A_0}\right) x\ 100 \qquad (1)$$

Where: A_0 is the absorbance of the dyeing solution before the dyeing process

A_f is the absorbance of the dyeing solution after the dyeing process

2.4 Absorbance measurements:

The absorbance measurements were recorded by using UV-Vis spectrophotometer by using quartz cells at a maximum wavelength of 265nm, which was previously determined in our experiments.

2.5 Experimental design

In this study the response surface methodology was applied to optimize the process variables. Prior designing the experimental runs, preliminary studies were done to determine the range for each parameter. Minitab18 statistical software was used for the design of experiments and statistical analysis of the process. The experimental ranges of factors are shown in table 1. A total number of 32 experiments were proposed by the software. Analysis of variance (ANOVA) was employed to select or to reject the model terms with a P-value with 95% confidence level. Dye uptake percentage was used as response. The experimental data were analyzed and fitted to the second-order polynomial model, including linear, quadratic and interaction. The following equation of the non-linear multiple regression quadratic model was used:

$$Y = b_0 + \sum_{j=1}^{5} b_j x_j + \sum_{i<j} b_{ij} x_i x_j + \sum_{j=1}^{5} b_{jj} x_j^2 \qquad (2)$$

Where: y is the response factor, b_0 is the constant term, b_j is the linear term, b_{ij} is the coefficient of interaction, b_{jj} is the quadratic coefficient.

Table 1. Independent variables and their levels used for response surface study

Factors	Unit	Levels				
		-2	-1	0	+1	+2
(x1) dyeing time	Minute	40	50	60	70	80
(x2) dye concentration	g/L	0.2	1.9	3.6	5.3	7
(x3) mordant amount	g	0	0.4	0.8	1.2	1.6
(x4) dyeing temperature	^{o}C	90	95	100	105	110
(x5) pH	pH	1.5	1.8	2.1	1.8	2.7

2.6 Fastness properties assessment on the dyed wool fabric

Color fastness is used to determine the degree to which dye holds fast to the fiber. A good or high fastness means that they do not bleed in washings and perspiration processes. The color fastness to washing test and acid and alkaline perspiration test were performed and evaluated according to ISO standards respectively ISO 105-C10:2006, ISO 105-E04, ISO 105-A02, ISO 105-A03.

Color fastness to washing: A specimen of the textile in contact with two specified adjacent fabrics is mechanically agitated under specified conditions of time and temperature in a soap solution, then rinsed and dried. The change in color of the dyed cotton specimen and the staining of the adjacent fabrics are assessed with reference to the original fabric with grey scale, ISO 105- C10:2006.

Color fastness to perspiration: Specimens of the textile in the contact with adjacent fabrics are treated in two different solutions, acidic and alkaline solution containing histidine, drained and placed between two plates under a specified pressure in a test device. The change in color of each specimen and the staining of the adjacent fabrics are assessed by comparison with grey scale ISO 105-E04.

3. RESULTS AND DISCUSSIONS

3.1 Model fitting and ANOVA results

In order to check the model adequacy, the experimental data were fitted to various models. According to the values of P and adjusted R – squared we can conclude that the dyeing process of wool fibers with saffron petals extract was most suitably described by a quadratic model. The analysis of variance was used for measuring up the significance of the effect of the dyeing process variables and their interactions on the dye uptake as the response. The ANOVA results of the fitted quadratic model are shown in table 2. According to the probability value of the independent variables, the significant or insignificant factors were determined. A probability value less than 0.05 indicates that the effect of this independent variable is significant. In our case, the primary impact towards the rate of dyeing uptake are dyeing time (x1), dyeing temperature (x4) and mordant amount (x3) were emerged to be the most substantial factors. Followed by the second order effect of dyeing time (x1*x1), dye concentration (x2*x2), mordant amount (x3*x3), dyeing temperature (x4*x4) and pH value of the dyebath (x5*x5) and the two-way interactions between dyeing time and dyeing temperature (x1*x4), between dye concentration and pH value (x2*x5) and between mordant amount and dyeing temperature (x3*x4). Furthermore, the primary influence of dye concentration (x2) and pH of the dyebath (x5), the 2-way interaction between dyeing time and mordant amount (x1*x3) and between dye concentration and mordant amount (x2*x3) were found to be corresponding to the secondary effect on the dyeing uptake percentage. The obtained second-order polynomial equation from the regression analysis of the experimental data is shown below:

$$Y = -597 + 0.72\, x1 + 6.69\, x2 - 54.3\, x3 + 6.65 x4 + 238.9\, x5 - 0.04102\, x1{*}x1 - 2.333\, x2{*}x2 - 32.38\, x3{*}x3 - 0.0479\, x4{*}x4 - 61.57\, x5{*}x5 + 0.114\, x1{*}x3 + 0.0446\, x1{*}x4 + 0.535\, x2{*}x3 + 4.786\, x2{*}x5 + 1.000\, x3{*}x4 \quad (3)$$

The obtained empirical model was used in the optimization and prediction of the dyeing uptake percentage within the range of variable factors in this experimental work. The coefficient of determination was 0.982 and adjusted R2 was 0.97. These values imply that the fitted model is highly significant and can explain the relationship among variables and response.

The fitted empirical model was examined by the normal plot of residuals. Residuals are the difference between actual and predicted values for each point and show how well the model satisfies the assumptions of the analysis of the variance. The normal probability plot of the residuals for the data tests the hypothesis that the residuals have a normal distribution. From figure 1 it is clear that the points were almost distributed in a line, which indicated that the model was satisfactory and accurate.

Table 2. ANOVA results of fitted quadratic model to the experimental data of wool dyeing process

Source	DF	Adj SS	Adj MS	F-value	P-value
Model	15	3608.14	240.54	58.11	0.000
Linear	5	519.77	103.95	25.11	0.000
x1	1	286.75	286.75	69.27	0.000
x2	1	9.57	9.57	2.31	0.148
x3	1	27.57	27.57	6.66	0.020
x4	1	183.15	183.15	44.24	0.000
x5	1	12.65	12.65	3.06	0.100
Square	5	2844.22	568.84	137.41	0.000
x1*x1	1	493.66	493.66	119.25	0.000
x2*x2	1	1333.33	1333.33	322.09	0.000
x3*x3	1	787.54	787.54	190.24	0.000
x4*x4	1	42.00	42.00	10.15	0.006
x5*x5	1	900.60	900.60	217.56	0.000
2-way interaction	5	244.44	48.89	11.81	0.000
x1*x3	1	3.31	3.31	0.80	0.384
x1*x4	1	79.64	79.64	19.24	0.000
x2*x3	1	2.12	2.12	0.51	0.485
x2*x5	1	95.31	95.31	23.02	0.000
x3*x4	1	64.06	64.06	15.48	0.001

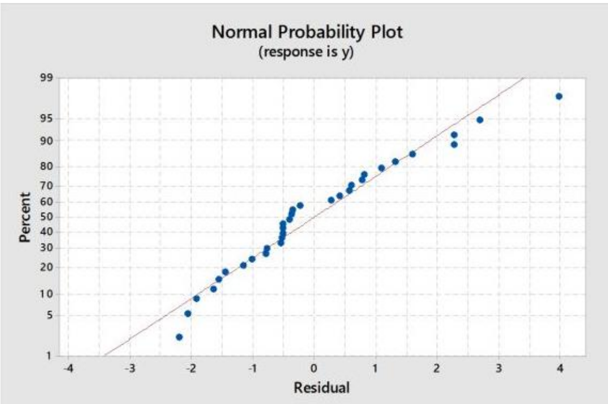


Figure 1. Normal plot of residuals

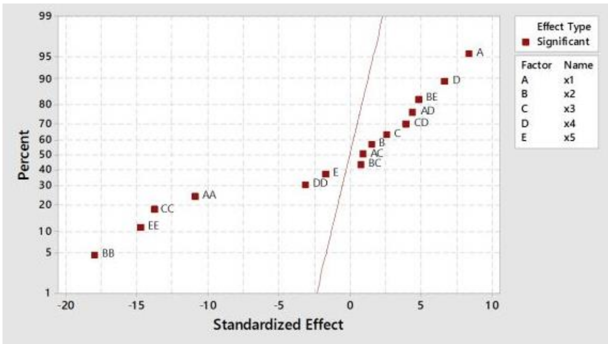


Figure 2. Normal probability plot of standardized effects

The normal probability plot of the standardized effects is shown in figure 2. This graph was used to verify the statistically main and interaction effects that were included in the model. According to the normal probability plot, the points located closer to the fitted line are not considered as significant, whereas points located far away from the line tend to be more significant.

The main factors A, B, C and D on the right side have a positive effect on the adsorption efficiency, while the factor E lies in the left and has a negative effect. The time of dyeing (point A) has the largest effect since its point lies farthest from the line. The second important factor is the dyeing temperature (point D), which is more significant than amount of mordant, dye concentration and pH. The interaction and quadratic terms have significant effect on adsorption efficiency because they stay away from the line. Quadratic term of dye concentration has the most significant effect.

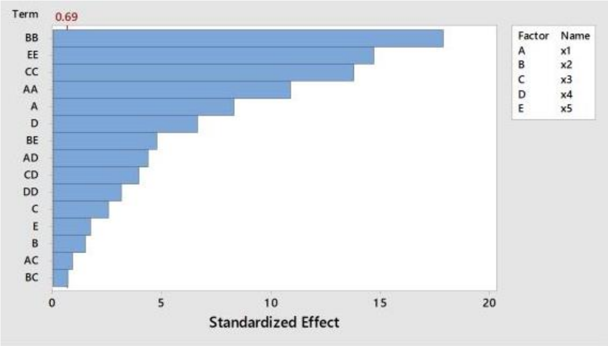
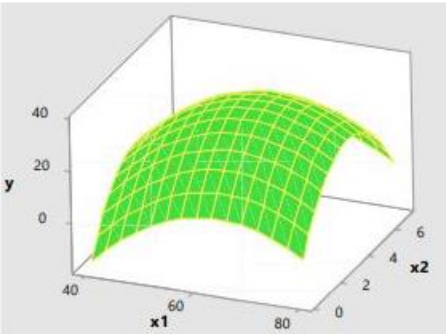


Figure 3. Pareto chart of the standardized effects of model variables

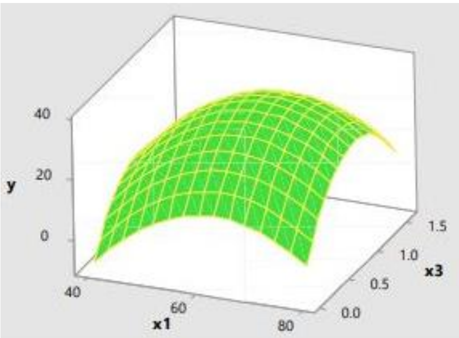
The Pareto chart of this experimental design is shown in figure 3. According to this chart the quadratic effect of dye concentration has the most significant effect on adsorption efficiency. The other quadratic effects EE, CC, AA, main terms A and D and 2-way interactions BE, AD, CD have greater effects on adsorption efficiency. Other factors C, E, B, AC and BC have significant effect and are statistically significant at 95% confidence level.

3.2 Three dimensional surface plots

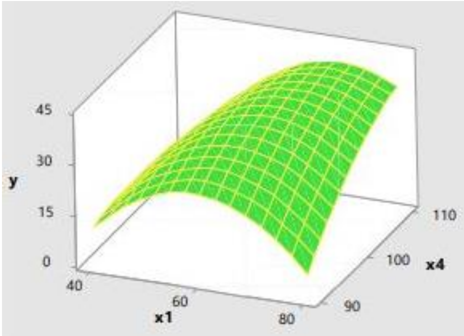
The effect of process parameters on the dye uptake efficiency was further analyzed with the assistance of three dimensional plots. The plots were obtained by using Minitab18 software. In all response surface plots, three other variables which are not mentioned are in their midpoints. These plots are represented in figures 4, 5 and 6.



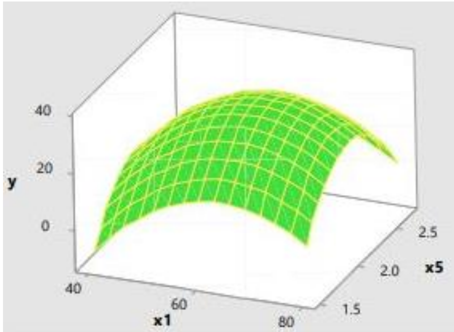
(a)



(b)

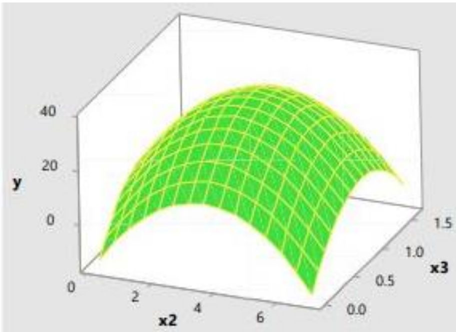


(c)

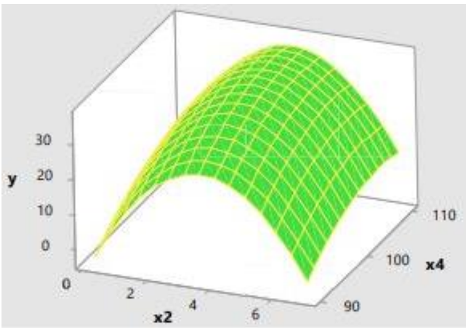


(d)

Figure 4. The 3D surface plots of the dye uptake for (a) dyeing time and dye concentration, (b) dyeing time and mordant amount, (c) dyeing time and dyeing temperature, (d) dyeing time and pH



(a)



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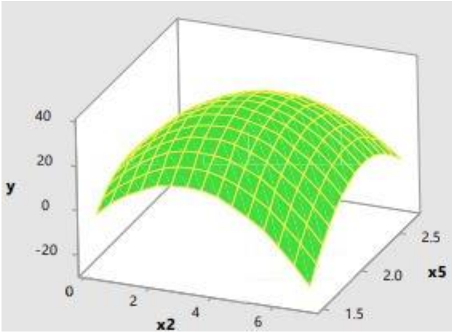
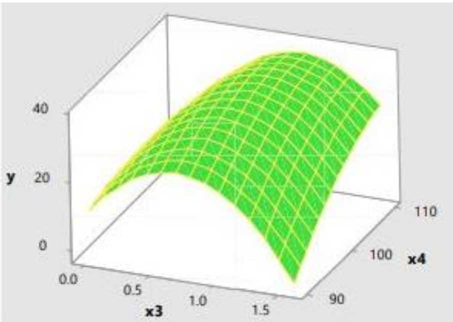
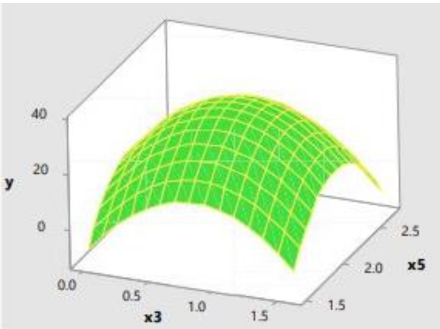


(c)

Figure 5. The 3D surface plots of the dye uptake for (a) dye concentration and mordant amount, (b) dye concentration and dyeing temperature, (c) dye concentration and pH



(a)



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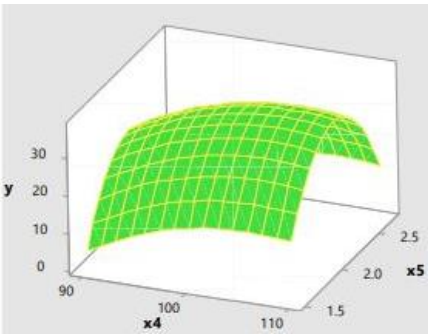

(c)

Figure 6. The 3D surface plots of the dye uptake for (a) mordant amount and dyeing temperature, (b) mordant amount and pH, (c) dyeing temperature and pH

The effect of dyeing time, dye concentration, mordant amount, dyeing temperature and pH, illustrated in figures 4, 5 and 6, showed that the dye uptake percentage decreased when dyeing time, dye concentration, mordant amount and pH values increased and increases with increasing the dyeing temperature. The increase of the dyeing time from low values improves the dye uptake due to long contact time between the dye and the wool fiber, but prolonged dyeing time can cause to desorption or degradation of the natural colorant, resulting in lower dye uptake values. Increasing the initial dye concentration leads to an increase in mass gradient between the dye solution and the wool fiber. This acts as a driving force leading to transfer of dye molecules from solution to fiber surface, but in high concentration values occurs the association of the dye molecules so they cannot penetrate into the fiber pores. Mordanting increases the interaction between the functional groups of wool fibers (-COOH and –NH2) and the flavonoid and phenolic groups present in the saffron petals extract. Increasing the amount of mordant leads to a higher dye uptake until all the active sites present in the fiber are filled. Higher amounts of mordant can cause the agglomeration of dye molecules around the mordant molecules present in the dye bath. Increasing the pH beyond acidic values causes the neutralization of the functional groups of wool fiber and forming of unstable structures for the dyeing fixation. Thus acidic values are suitable for wool dyeing. Increasing the dyeing temperatures leads to increase in the dye uptake due to the intensification of molecular movement. This intensification leads to more hits with the fiber surface and successful fixation of the dye molecules. The 3 dimensional graphs show the simultaneous effects of parameters on the response in which the highest points of the curved surface plots indicate the values of factors resulting in maximum dye uptake. These graphs are useful for establishing response values and operating conditions that are needed. We can conclude that the dye uptake was higher near the optimum conditions (mentioned below) of the dyeing process.

3.3 Optimization of dyeing conditions

In our case the optimization of process parameters by using CCD method was done to maximize the response with desirability values of d=1.000 in order to obtain a response almost similar to optimum. From software the optimum conditions of dyeing wool with extract of saffron petals were found to be dyeing time 69.89 minutes, dye concentration 3.7%, mordant amount 1.02g, dyeing temperature 110°C and pH 2.08 (figure 7). The good agreement between predicted values and the experimental ones means that the derived model can be used to adequately describe the relationship between factors and response in this study (figure 8 and 9).

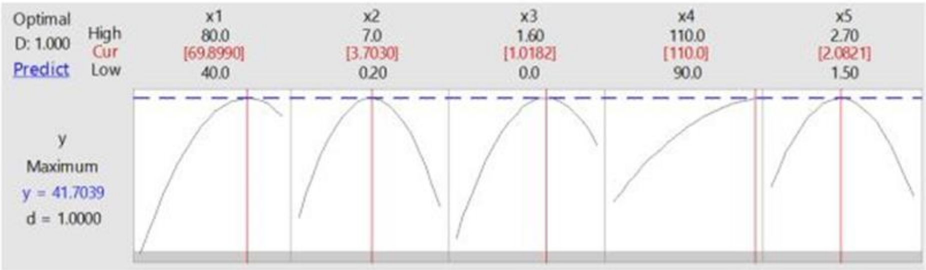

Figure 7. Optimum dyeing conditions for wool fiber

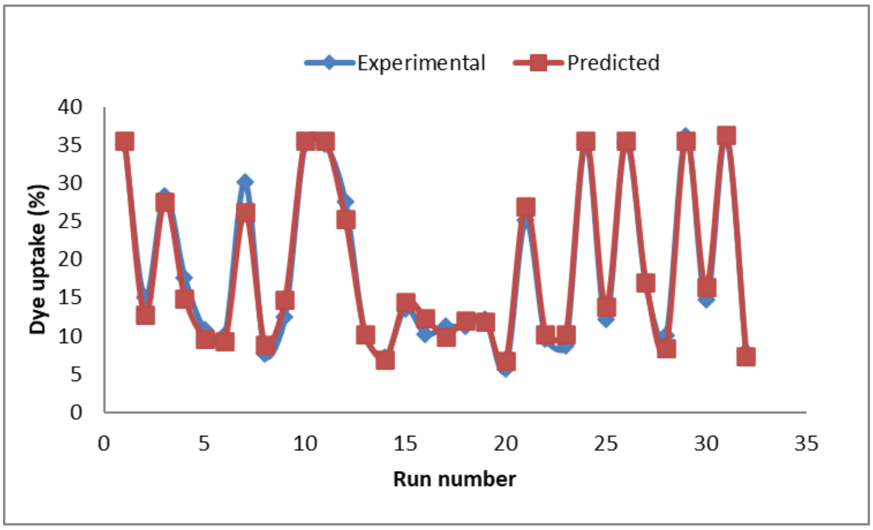

Figure 8. Relative deviation between experimental and predicted values of dye uptake for wool fiber dyed with extract of saffron petals

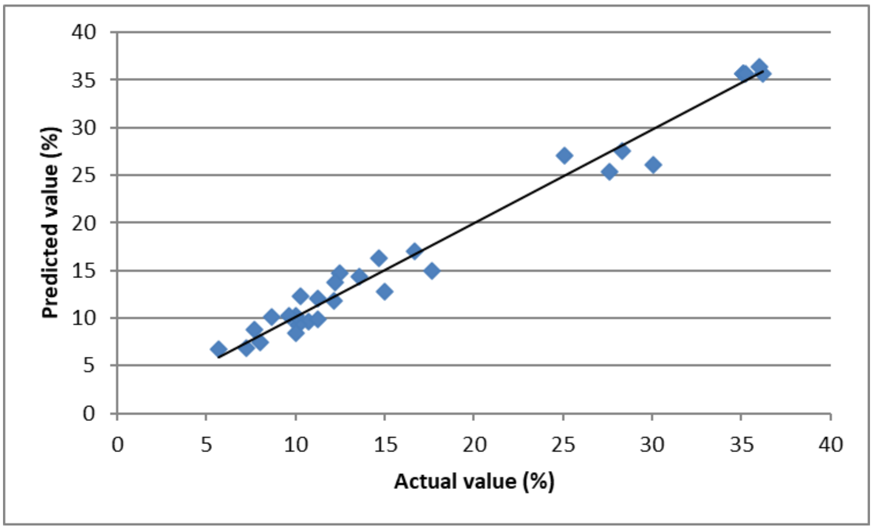

Figure 9. Experimental values versus predicted values for dye uptake for wool fiber dyed with extract of saffron petals

The actual forecast graph shows how well the model fits the data. The diagonal line indicates that the predicted value and the actual value have the same position. If all the points are on or around this diagonal, it proves that the predicted value is more representative. The relationship between the predicted value and the actual value of the response in shown in figure 9. This figure shows that there is sufficient consistency between the actual data and the data obtained from the model.

3.4 Fastness properties of dyed samples

The color fastness to washing and perspiration of wool fabrics dyed with extract of saffron petals is presented in table 3. The wool fabrics were pre-mordanted and dyed according to the proposed optimized conditions in order to find out the fastness properties.

Table 3. Fastness properties of dyed wool fabrics with proposed optimized conditions.

Washing fastness			Perspiration Color fastness					
			Basic			Acidic		
Color fastness	CS cotton	CS wool	Color fastness	CS cotton	CS wool	Color fastness	CS cotton	CS wool
4-5	5	4-5	5	5	5	5	5	5

From the experimental results, it can be concluded that the fastness properties of the dyed wool fibers were improved. The metal ions form a complex with the dye molecule; making it insoluble in water and resulting in a higher color fastness. Aluminium ions as mordant have a strong affinity to wool fabrics and fix several dye molecules onto the fiber, creating a larger complex and helping the fiber to retain the color and consequently increasing the color fastness. Both, the washing and perspiration color fastness tests gave good fastness grades for dyed wool fabric in the optimized dyeing conditions.

4. CONCLUSIONS

Wool fibers were successfully dyed with extract of saffron petals. Response surface methodology was used for the optimization of the dyeing conditions. The quadratic model was most suitably to describe the relationship between variables and the response. The optimum conditions of the process dyeing of wool fiber with extract of saffron petals were: dyeing time 69.89 minutes, dye concentration 3.7%, amount of mordant 1.02g (0.56%), dyeing temperature 110^{o}C and pH 2.08. The R2 value of 0.982 shows the satisfactory explanatory of the fitted model. The experimental values were in good agreement with the model predicted values. The dyed wool fibers at optimal conditions proposed by RSM method have shown improvement in the washing and perspiration fastness properties. They gave good and increased fastness grades.

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IZVOD

OPTIMIZACIJA PROCESA BOJENJA VUNE EKSTRAKTOM LATA ŠAFRANA KORIŠĆENJEM METODOLOGIJE ODGOVARAJUĆIH POVRŠINA

Nedavno je povećana svest o potražnji za ekološki prihvatljivim bojama u tekstilnoj primeni, jer prirodne boje mogu otkriti bolju biorazgradljivost i generalno imaju veću kompatibilnost sa životnom sredinom. Postoje stalni pokušaji da se prevaziđu neki nedostaci u vezi sa prirodnim bojenjem, kao što su produženo vreme bojenja, potrošnja energije i korišćene hemikalije. Sa ove tačke gledišta, optimizacija parametara procesa je dobra alternativa. U ovom radu je prikazano bojenje vunenih vlakana ekstraktom latica šafrana. Metodologija površine odgovora se koristi za optimizaciju parametara procesa i za istraživanje odnosa između različitih faktora koji se uzimaju u obzir, kao što su vreme bojenja, koncentracija boje, količina jedka, temperatura bojenja i pH. Upijanje boje je procenjeno merenjem apsorbancije, korišćenjem UV-Vis spektrofotometra i korišćeno je kao odgovor. Optimalni uslovi bojenja za dobijanje najveće vrednosti apsorpcije boje bili su sledeći: vreme bojenja 69,89 minuta, koncentracija boje 3,7%, količina jedila 1,02g (0,56%), pH kupke za farbanje 2,08, temperatura bojenja 110°C.

Ključne reči: prirodna boja, optimizacija, RSM, ekstrakt šafrana, vuna

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