The use of D-optimal design in optimization of wool dyeing with *Juglansregia* bark

DOI: 10.35530/IT.069.02.1509

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REZUMAT – ABSTRACT

Utilizarea modelului D-optimal în optimizarea vopsirii lânii cu coaja de *Juglansregia*

În acest studiu, fibrele de lână au fost vopsite folosind coaja de Juglansregia ca o nouă sursă de colorant natural. Alaunul a fost utilizat ca mordant. Metodologia de suprafață a răspunsului şi modelul D-optimal au fost utilizate pentru studierea şi optimizarea procedeului de vopsire, cu scopul de a obține intensitatea maximă a culorii după vopsirea cu extractul apos de coajă de Juglansregia. Rezultatele au arătat că intensitatea culorii fibrelor vopsite a crescut prin creşterea timpului de vopsire şi a temperaturii şi a scăzut prin creşterea valorii pH-ului băii de vopsire. A existat o valoare optimă de aproximativ 6% owf pentru concentrația de mordant. Condiția optimă pentru obținerea intensității maxime a culorii a fost următoarea: pH-ul băii de vopsire: 6, concentrația de alaun: 6,24% owf, temperatura de vopsire: 90 °C şi timpul de vopsire: 90 min.

Cuvinte-cheie: mordant, colorant natural, lână, optimizare, RSM

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In this study, wool fibers were dyed using the Juglansregiabark as a new source of natural dye. Alum was used as mordant. Response surface methodology and D-optimal design were employed to study and optimize the dyeing procedure with the aim of obtaining the maximum color value after dyeing with aqueous extract of Juglansregiabark. The results showed that the color value of the dyed fibers was increased by increasing the dyeing time and temperature and decreased by increasing the dyebath pH value. There was an optimum value of around 6 % owf for mordant concentration. The optimal condition for obtaining the highest color value was as follows: dyebath pH: 6, alum concentration: 6.24 % owf, dyeing temperature: 90°C, and dyeing time: 90 min.

Keywords: mordant, natural dye, wool, optimization, RSM

INTRODUCTION

Natural dyes are known as sustainable and environmentally friendly materials for dyeing and functional finishing of textiles [1]. They can be obtained from vegetable, animal or mineral origin [2]. Several studies have been reported on application of different natural dyes on textile fibers. Barberry tree root, cumin seeds, grape leaves and pomace, red cabbage, milkweed leave, *Achilleapachycephala flowers*, almond shell, pomegranate rinds and wastewater of olive oil production are examples of new sources of natural dyes which have been studied in recent years [3–17]. Despite several advantages associated with the use of natural dyes in dyeing textile goods, there is a great need for optimization of natural dyeing processes to fulfill the equipments of today's industry.

Metal mordants are commonly used in order to increase the uptake and fastness of natural dyes on textile fibers and obtain different shades using a single dye [18]. However, most of metal mordants cause environmental problems as well as health concerns for the consumers [19]. Natural dyeing plants usually posses low color yield and require prolonged time to dye textiles satisfactorily. Several pretreatments like cationization, plasma treatment, enzyme treatment, gamma treatment, and microwave treatment are examples of techniques which have been studied to overcome this drawback [7, 10, 20–24]. To minimize the consumption of energy, dye, mordant, and auxiliaries besides decreasing the required time, while gaining the highest dyebath exhaustion, optimization of the dyeing process is really important [25].

In the traditional method for optimization of processes, experiments are first performed and the measured data is analyzed afterwards. This approach examines one variable at a time and is time and work demanding and the effect of interactions between different factors is not taken into account [26]. In contrast to this, in statistical methods, the experimental design is planned and sets of well selected experiments are performed to get the most informative combination out of the assumed factors with the minimum number of experiments. Response surface methodology (RSM) offers design of experiment (DOE) tools that lead to refined optimization approaches and process performance at minimal cost [27]. D-optimal designs create the optimal set of experiments on the basisof a computer-aided exchange procedure. This method selects the best combination of experimental trials within the limitations provided and provides maximum accuracy in estimating regression coefficients. The optimality criterion results in minimizing the generalized variance of the parameter estimates for a pre-specified model [28–29].

Juglansregia is a tree native to central Asia and can be found in several countries all over the world. Many parts of this tree including green walnuts, shells, seed, bark, and leaves are used in the pharmaceutical and cosmetic industry. The bark of this tree is used as a toothbrush and a dye for coloring the lips for makeup purpose is some parts of south of Iran. It contains several phenolic compounds namely, β-sitosterol, juglone, folic acid, gallic acid, regiolone, and quercetin-3-α-L-arabinoside [30–31].

In this study, the bark of *Juglansregia*tree was chosen as a new source of natural dye for coloration of wool fibers. Four independent factors including mordant concentration, dyebath pH, and temperature besides the dyeing time were selected as the most influencing factors according to preliminary experiments. To find out the optimum conditions for dyeing procedure, D-optimal design was used and the effect of dyeing process factors on the color value of the dyed samples was determined.

EXPERIMENTAL WORK

Materials and methods

Pure wool fabric (plain weave, 250 g/m^2) was purchased from Iran Merinos Textile Company, Iran, and used for the experiments after scouring and drying (1% non-ionic detergent (Triton X-100, Sigma-Aldrich, USA), 50 °C, for 30 min). All other chemicals used in this study were analytical grade reagents obtained from Merck, Germany.

Juglansregia bark was washed with tap water, dried and then powdered. 100 g of powder was used for preparation of 1 liter of the original dye solution. Distilled water was used for this purpose and boiling was continued for 2 h and then the solution was filtered. The concentration of the prepared solution is 10 % W/V.

Experimental Design: The formulation of experiments and statistical analysis of responses were performed using Design Expert software (version 7.0). In this study, the most influencing operating factors of the natural dyeing process were optimized using response surface methodology (RSM) and D-optimal design. The practically feasible ranges for each factor were determined by preliminary studies before designing the experiments. Table 1 presents the corresponding codes besides lower and higher values for each variable.

Table 1

EXPERIMENTAL RANGES OF FACTORS

Factor	Name	Unit	Low level	High level
A	Dyeing pH	-	4	8
B	Mordant concentration	% owf	0	10
C	Dyeing temperature	ºC	50	90
D	Dyeing time	min	30	90

A total number of 25 experiments were proposed by the software. P-value with 95% confidence level was considered for the selection or rejection of the model terms. To analyze the results, ANOVA was employed. Response surfaces were drawn to determine the individual and interactive effects of the process variables on the color value of dyed samples.

Mordanting: The mordanting bath was prepared using the required amount of alum (aluminum potassium sulfate) according to the experimental design and acetic acid was used for adjustment of pH at 5. The liquor to goods ratio (L:G) was 50:1 and the mordanting was done at boil for 1 h.

Dyeing: Dyeing of the samples was performed using 50% owf of the natural dye(L:G= 40:1, pH=4–8). The dyeing was started at 40 °C and the temperature was raised to the final temperature at the rate of 2 °C per minute. Then the samples remained in that condition for the predefined time according to the experimental design, and then rinsed and air dried.

Color value measurements: the reflectance of dyed samples were measured on a Color-eye 7000A spectrophotometer using illuminant D65 and 10° standard observer. Color strength (K/S) of each dyed sample was calculated using kubelka-munk equation for each wavelength ranging between 360–740 nm:

$$K/S = (1 - R)^2 / 2R \quad (1)$$

Where R is the observed reflectance, K – the absorption coefficient and S – the light scattering coefficient. For better comparison of the samples in the full range of the visible spectrum, the sum of color strengths measured at all wavelengths (color value sum or CV_{sum}) was calculated and considered for further analysis.

$$CV_{sum} = \Sigma_{360}^{740} (K/S) \quad (2)$$

RESULTS AND DISCUSSION

Model fitting and statistical analysis

The experimental conditions and color values (CV_{sum}) of the woolen fabric samples dyed with 50 % owf of natural dye are shown in table 2. The data obtained from the colorimetric analysis of the dyed samples were fitted to various models. ANOVA results of fitting different models to the obtained data are shown in table 3. The quadratic model was the most suitable model for describing this process. The analysis of variance was used for measuring up the significance of the effect of the dyeing process variables and their interactions on the CV_{sum} as the response. A P-value less than 0.05 was considered as a sign which confirms that the model and the terms are statistically significant. In case that many insignificant model terms are found, model reduction which means the elimination of the insignificant factors from the model can improve the final model. In this study, model reduction was performed by the software and some insignificant interactions of the variables having P-values higher than 0.05 were eliminated.

Table 2

EXPERIMENTAL DESIGN OF DYEING PROCEDURES AND RESPONSES					
	Factor 1	Factor 2	Factor 3	Factor 4	Response
Run	A: pH	B: Mordant Concentration (% owf)	C: Temperature (ºC)	D: Dyeing time (min)	CV_{sum}
1	4	10	50	30	71.4
2	8	0	50	90	72.2
3	8	0	50	90	61.5
4	4	0	50	30	141.2
5	6	5	90	60	215.5
6	6	5	50	60	146.4
7	8	10	50	90	127.5
8	4	5	50	90	220.4
9	6	10	70	60	85.2
10	4	5	90	30	192.7
11	4	0	90	90	62.2
12	4	10	90	90	172.4
13	88	10	50	90	95.8
14	6	10	90	30	59.6
15	8	0	90	30	133.2
16	8	0	70	30	66.1
17	8	5	90	90	231.6
18	4	0	90	90	142.2
19	6	2.5	70	60	56.8
20	4	0	50	30	92.4
21	8	10	90	30	100.2
22	8	5	50	30	62.9
23	4	5	70	60	179.7
24	8	0	90	60	99.4
25	8	5	70	60	123.8

Table 3

ANOVA RESULTS OF THE FITTING THE EXPERIMENTAL DATA TO VARIOUS MODELS				
Source model	F value	P value Prob > F	R-Squared	
Linear	2.78	0.1320	0.2685	
2FI	3.05	0.1163	0.4920	
Quadratic	0.95	0.5196	0.8469	Suggested
Cubic			0.9217	aliased

Table 4

ANOVA RESULTS OF THE ESTABLISHED MODEL FOR RESPONSES		
Factor	F-Value	P-Value
Model	7.83	0.0003
A: Dyeing pH	5.24	0.0360
B: Mordant concentration	0.65	0.4331
C: Dyeing temperature	7.90	0.0126
D: Dyeing time	9.44	0.0073
AC	4.25	0.0559
BD	10.84	0.0046
B^2	26.32	0.0001
C^2	5.96	0.0267
Lack of Fit	0.73	0.6947

Table 4 shows the analysis of variance (ANOVA) results of the established model for responses. The model *F*-value of 7.83 implies on the significance of the model. When the calculated Value for Prob>*F* related to a certain variable is less than 0.05, itmeans that the corresponding model term is significant at a confidence level of 95%. In this case *A, C, D, BD*, B^2 and C^2 are significant model terms. A high R^2 coefficient confirmed a sensible concurrence between the proposed model and the experimental data.

The "Pred R-Squared" of 0.5245 was in reasonable agreement with the "Adj R-Squared" of 0.6947. "Adeq Precision" shows the extent of divergence in predicted response regarding its associated error or signal to noise ratio and compares the range of predicted values at design points to the average prediction error. A desirable "Adeq Precision" should be higher than 4 and indicates that the mode has been selected suitably [26]. In this case, the ratio of 9.652 impliesthat this model was well selected and can be used forhandling the design space.

Regression analysis was performed on experimental data and the following model equation in terms of coded factors was fitted:

$$CV_{sum} = 136.75 - 16.07A + 6.23B + 19.67C + 25.42D + 17.35AC + 28.00BD - 69.09B^2 + 39.06C^2 \quad (3)$$

The effects of parameters on color value

To compare the effect of four factors on color value of dyed samples, perturbation plot (figure 1) was drawn. This plot shows the effect of changing each factor on CV_{sum} while holding three other factors constant. The reference amounts of the factors to draw the plot are shown on it. A steep slope or curvature in the resulting trace indicates sensitivity of the response to that factor. From the curvature of the plot *B* and *C*, it can be concluded that the response is more sensitive to mordant concentration and dyeing temperature compared with other factors. The lower steep of the pH line shows less sensitivity of the color value to change in this factor at the range investigated in this study.

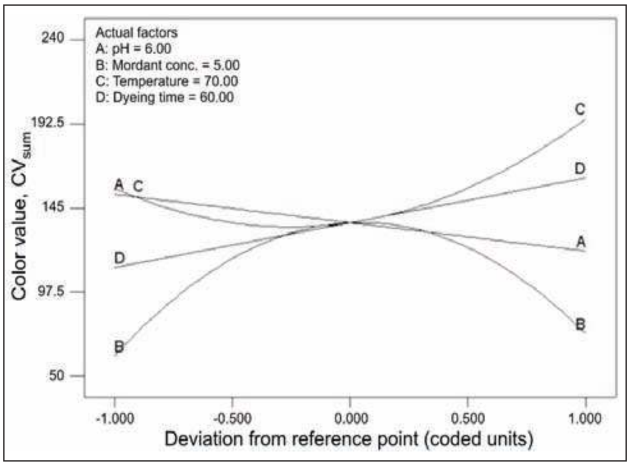

Fig. 1. Perturbation plot for CV_{sum}

Figure 2 shows the individual and simultaneous effects of the dyeing procedure factors on color value of the dyed samples. It can be seen that the addition of alum mordant and increasing its concentration up to 6% owf has increased the color value of the dyed samples. It means that the dye uptake of the mordanted samples has been higher than the non-mordanted sample. Mordanting increases the interaction between the amine groups of wool fibers and hydroxyl and carbonyl groups of juglone as the main colorant present in the extract used for dyeing [32]. When using more than 6% owf of alum, the color strength has been decreased probably due to increasing the physical damage to the wool fibers. The 3D graphs show the simultaneous effects of factors on the response in which the red area indicates the amounts of the factors resulting in the maximum color value. These graphs are useful for establishing response values and operating conditions that are needed.

Figure 3 shows the mechanism of complex formation between the wool protein, aluminum ion, and dye molecule.

Increasing the dyeing time increased the color value due to the higher amount of dye molecules absorbed by the fibers at prolonged time. Increasing the dyebath pH from 4 to 8 has decreased the color value of the dyed samples. Wool fiber gains more positive charges at acidic pH values and the juglone molecules can be better absorbed by positively charged wool fibers at this condition [20, 33]. Increasing the dyeing temperature has increased the color value of the dyed samples due to increasing the exhaustion especially at temperatures higher than 70 °C. This increase in dye-uptake is due to the fibre swelling and breaking the aggregations of dye molecules at higher temperatures which improved the dye diffusion into the wool fiber [20, 34].

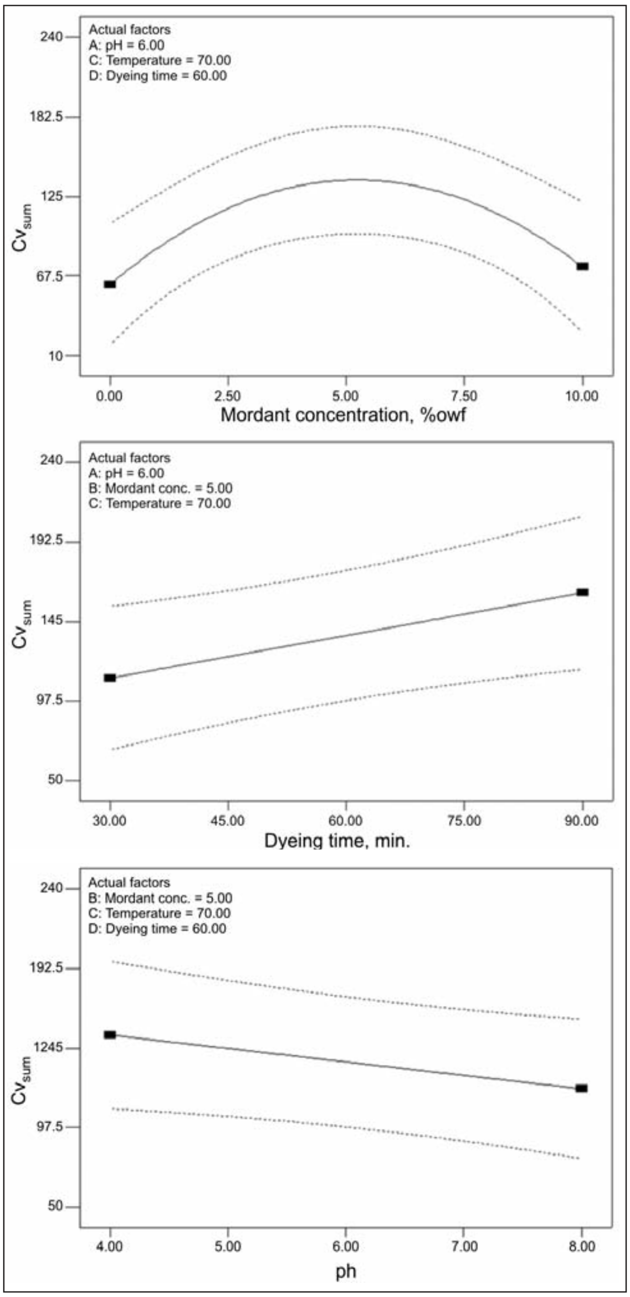

Fig. 2. The individual and simultaneous effects of each factor on color value of dyed samples

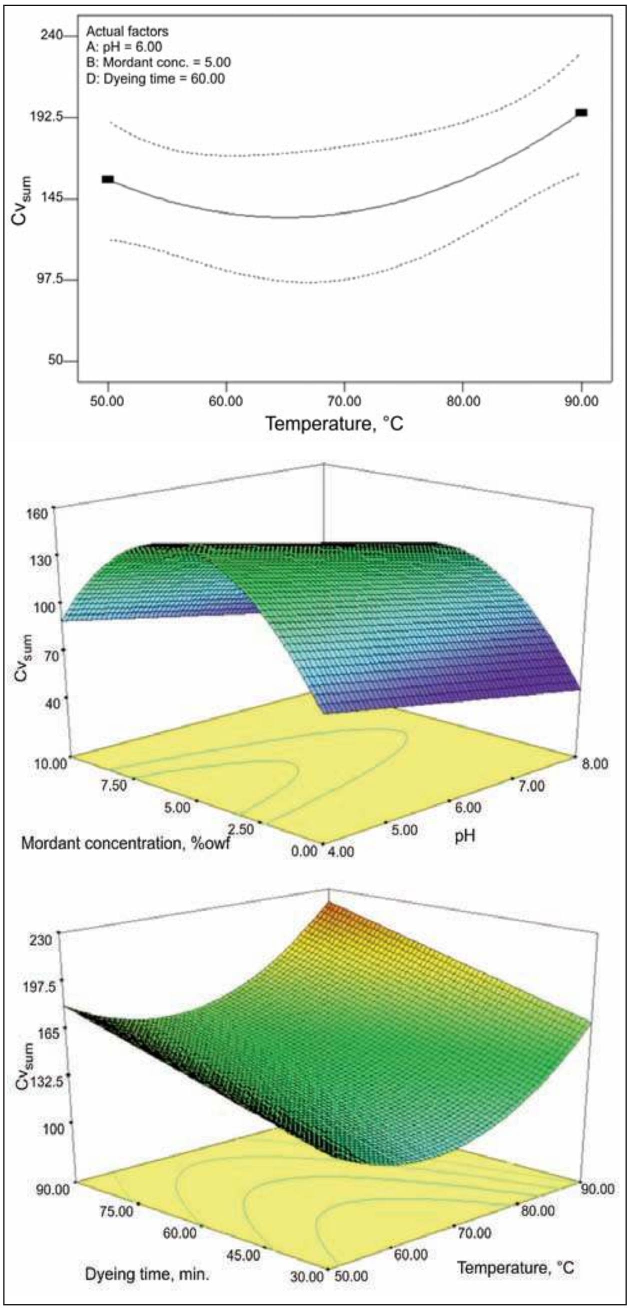

Fig. 2. Continued

Table 5

OPTIMAL CONDITIONS FOR THE DYEINGOF WOOL FIBERS TO OBTAIN MAXIMUM COLOR VALUE						
Dyeing pH	Mordant concen-tration (%owf)	Dyeing temper-ature (ºC)	Dyeing time (min)	Predict-ed CV_{sum}	Experi-mental CV_{sum}	Desir-ability
4	6.24	90	90	223.76	226.93	0.977

Optimization of dyeing process

The maximum color value was taken as the desired response and the optimal conditions for obtaining the maximum CV_{sum} were predicted using the optimization function of Design Expert software. All factors were selected to be "in the range". The optimized conditions are shown in table 5. Good agreement between the predicted CV_{sum} and the experimental value means that the empirical model derived from RSM can be used to adequately describe the relationship between the factors and response in this study.

CONCLUSION

In this study, the aqueous extract of *Juglansregia* bark was used as a natural dye for dyeing of wool. Alum was applied on wool fibers as a mordant using pre-mordanting method. The effects of four independent factors of the dyeing procedure on the color value of the dyed samples were statically studied using response surface methodology. The results showed that the CV_{sum} had the highest sensitivity to mordant concentration and dyeing temperature compared with other factors. Increasing dyeing time and temperature resulted in increasing the CV_{sum}, but the color value was decreased by increasing the dyebath pH, while there was an optimum amount for mordant concentration (around 6% owf) to obtain highest effect on color value. The optimal conditions to obtain the highest color value were derived from statistical data. This natural dyecan be considered as a suitable source of natural dye for coloration of wool fibers.

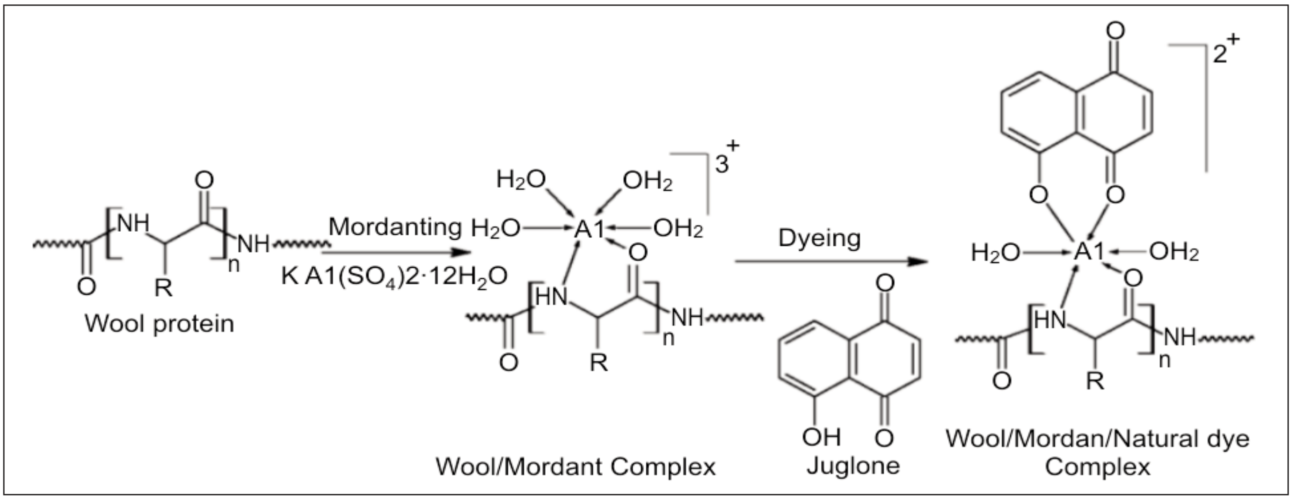

Fig. 3. Mechanism of complex formation between wool, aluminum mordant and juglone dye [32–33]

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