

Research Article

The Dyeing Procedures Evaluation of Wool Fibers with *Prangos ferulacea* and Fastness Characteristics

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In this study, the dyeing procedure of wool fibers with *Prangos ferulacea* was evaluated and optimized by response surface methodology (RSM). Using this method, the quantitative relationship between dye concentration of *Prangos ferulacea*, mordant concentration, dyeing temperature, and dyeing time on the dyeing procedure was investigated. The effect of these variables as well as plasma pretreatment was examined on the color strength of dyed samples. Finally, the fastness characteristic of dye sampled at proposed optimized condition was reported. The obtained results indicate that the presence of mordant improved the fastness properties and dyes uptake of wool fibers.

1. Introduction

In recent decade, there is a renewed interest to use ecofriendly products in daily life. Natural dyes due to energy saving, environmental protection, and health and safety aspects have been receiving increasing attention from researchers and manufacturers. Moreover, the use of natural dyes in textile dyeing has increased their deodorizing, antimicrobial, and UV protective properties [1]. Natural dye can be obtained from many plants in nature to make a variety of shades on several textile substrates such as cotton, silk, wool, polyester, polyamide, and acrylic fibers [2–5]. In addition, natural dyes are gaining popularity again with concern for the medicinal properties.

The genus *Prangos*, which belongs to the Apiaceae family, consists of about 30 species. *Prangos ferulacea* is one of its species which is called Jashir in Persian [6]. It is a perennial herb up to 2 m tall and a fragrant plant which belongs to the Apiaceae family and is found in the Mediterranean and Middle-East regions including Iran [7]. Its leaves are multipinnate with thread-like to linear segments [8]. Traditional ranchers collect green *Prangos* and then dry the plant and use it as animal fodder especially in winter season [9]. Its fruits and roots possess biological traits that provide it with

the potential to be used for medicinal purposes. In addition, in Iran, it is used in the preparation of cheese [10].

Due to the several applications of *Prangos* and its broad distribution, a number of useful studies on the plant have been carried out. The natural dye contains variable chemical compositions, due to the different conditions and the place of growing, harvesting period, extraction methods and conditions, and application method [11]. The major chemical structures of natural dyes are anthraquinone (madder), alpha naphthoquinones, flavones, indigoids, and carotenoids [11, 12]. According to the chemical investigations on the extracted aerial parts of dried plant, its main chemical components are essential oils (2.3%), α -pinene (24.2%), β -pinene (8.6%), and delta-3-carene (7.7%) [9]. Also the presence of coumarin derivatives was reported in this plant which cause an antibacterial effect [13].

In this study, the dyeing conditions of wool fiber were optimized by response surface methodology (RSM). Response surface methodology is a collection of mathematical and statistical techniques for empirical model building. The four independent numerical variables were selected at two levels. The numerical variables are dye concentration of *Prangos ferulacea*, mordant concentration, dyeing temperature, and dyeing time. The effect of these variables was examined

TABLE 1: Proposed experimental ranges of dyeing procedure and coded levels of independent variable factors.

Process variables	Code	Real values of the coded levels		
		-1	0	1
Dye concentration (% owf)	X1	20	40	60
Aluminum sulphate concentration (% owf)	X2	0	5	10
Dyeing temperature (°C)	X3	50	70	90
Dyeing time (minute)	X4	30	75	120

on the color strength of dyed samples as a dependent factor (response). Response surface methodology comprises a group of statistical techniques for empirical model building and model exploitation. Using an experimental design causes a relationship between a response variable and a number of predictors by analysis of experiments [14]. Central composite design (CCD) and response surface methodology (RSM) have been applied to design experiments to evaluate the interactive effects of the operating variables.

2. Materials and Experimental

2.1. Materials. Wool yarn with linear density of 200/2 tex was used at dyeing procedure. The samples were scoured by 1% Triton X100 at 50°C for 30 min, before dyeing procedure. The Triton X100 and acetic acid were purchased from Merck Company, Germany. The commercial aluminum sulphate was used for mordant of wool samples. The Jashir, *Prangos ferulacea*, as a natural dye was prepared from Fars province, Iran.

2.2. Experiment. The scoured wool sample was treated with different aluminum sulphate concentration (0, 5, 10% w/w). The sample was immersed into the prepared mordant solution (fibers:liquor ratio; 1:40) at 40°C. The temperature of the mordant solution was gradually increased with of 3°C min⁻¹ up to the boiling point (95°C at the laboratory condition) and continued for 30 min. Then, the wool fibers were rinsed with distilled water and dried at the room condition.

To extract the dye from the Jashir, *Prangos ferulacea*, the well-milled parts of leaves and stems were boiled in water for 60 minutes and stirred simultaneously. After that, the solution was cooled to the room temperature and the cooled solution was passed through the filter, and finally the liquid dye solution was extracted and adjusted to 10% w/w. The dye bath solution was prepared according to the proposed recipe of experimental design (Table 1). The independent numerical variables ranges were determined according to the results of preliminary experiments.

The temperature of the dye bath solution was increased gradually up to 50°C. Then, the wool sample yarn was inserted in the dye bath solution with increasing temperature at the rate of 3°C min⁻¹ until the proposed temperature, according to Table 1. The dyeing procedure was continued at the proposed time and then the sample was rinsed thoroughly with distilled water and dried at room condition. Finally, the natural dyeing condition of wool fibers was optimized by

RSM using the trial version of Design Expert 8.0.1.0 from Stat-Ease, Inc. (USA).

The spectral reflectance of all wool yarn samples was measured using a Color-Eye spectrophotometer from Gretag Macbeth in the visible region. In order to evaluate the color characteristic of dyed wool samples, the CIE color coordinates, namely, L^* , a^* , b^* , and C^* , were measured under illuminant D₆₅ and 10° standard observer. Color strength (K/S) values of the dyed wool samples were calculated using Kubelka-Munk equation (1) as follows:

$$\frac{K}{S} = \frac{(1 - R)^2}{2R}, \quad (1)$$

where R is the observed reflectance at the wavelength of maximum absorption ($\lambda_{\max} = 450$ nm), K is the absorption coefficient, and S is the light scattering coefficient. The color fastness to washing and light of dyed wool samples was tested according to ISO standard test method. Color fastness to washing was according to ISO105-CO2:1989, and color fastness to light was assessed according to ISO105-B02:1994.

Finally, effect of plasma treatment on wool fibers was investigated on dyeing properties of wool fiber. Plasma pre-treated on wool fibers results in an increase of dye diffusion into the wool fiber which can reduce the dyeing temperature [14]. So, the wool yarn samples were modified using radio frequency low pressure plasma equipment (model: Junior plasma, Europlasma, Belgium) with oxygen gas. The sample chamber was evacuated to 100 mTor and maintained during the process. The flow rate of oxygen was 20 sccm (standard cubic centimeters per minute) and plasma was generated at 100 W. The plasma treatment duration was 1, 2, 4, and 6 min. After that, air was introduced into the chamber and the plasma-treated sample was removed. The morphological changes on the surface of plasma-treated wool fibers were analyzed using a scanning electron microscope (Philips XL30 SEM). The wool fiber samples were sputtered with a thin layer of gold for 5 minutes prior to SEM analysis.

3. Results and Discussion

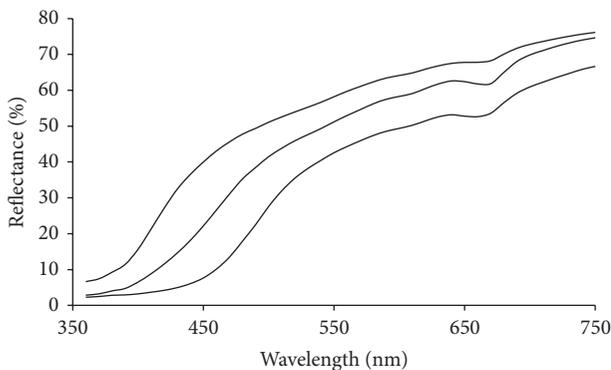
3.1. Color Measurement. The spectral reflectance of low, middle, and high color strength of wool dyed samples is presented in Figure 1. It is indicated that these samples absorb the blue light and therefore have a lower reflectance in the 400–480 nm region of the spectrum. In addition, the color coordinates of sample with high depth color ($L^* = 72.17$, $a^* = -1.83$, and $b^* = 52.69$) indicate that dyeing wool fibers

TABLE 2: ANOVA results of the experimental results fitting to different models.

Source	Sum of squares	df	Mean square	F value	P value (Prob > F)
Linear versus mean	19.41	4	4.85	7.628684	0.0002
2FI versus linear	5.97	6	1.00	1.790179	0.1389
Quadratic versus 2FI	2.47	4	0.62	1.133695	0.3654
Cubic versus quadratic	5.45	8	0.68	1.441997	0.2576

TABLE 3: ANOVA results obtained from fitting the linear model to experimental design space of dyed samples with *Prangos ferulacea*.

Source	Sum of squares	df	Mean square	F value	P value (Prob > F)
(A) Dye concentration	2.403661	1	2.403661	3.206681	0.1469
(B) Mordant concentration	8.104051	1	8.104051	12.7403	0.0011
(C) Dyeing temperature	7.46555	1	7.46555	11.73652	0.0017
(D) Dyeing time	2.437037	1	2.437037	3.831241	0.0588
Residual	20.99117	33	0.636096		
Lack of fit	14.637	20	0.73185	1.497294	0.2298

FIGURE 1: The three spectral reflectance of dyed wool samples with *Prangos ferulacea*.

with *Prangos ferulacea* as a natural dye cause a yellow shade to appear.

In order to find out the dyeing condition of *Prangos ferulacea* effect on the color strength of dyed wool fiber yarn, the RSM design called a central composite design (CCD) was used to determine the experimental runs. It is composed of a core factorial that forms a cube with sides that are two-coded units in length. The cube samples are experiments which cross lower and upper levels of the design variables. The four independent numerical variables were selected which led to determine 38 runs. These runs include 6 center point samples that replicate the experiment which cross the mid-levels of all design variables and usually used to determine the experimental error of the design.

The 38 wool yarn samples were dyed according to the proposed recipes dyeing which is designed by the central composite design method of RSM. The dyeing condition (dye concentration of *Prangos ferulacea*, mordant concentration, dyeing temperature, and dyeing time) effects were examined on the color strength of dyed samples as a dependent factor (response). The experimental data of dyeing samples were fitted to various models, and finally according to the ANOVA

results (Table 2), the linear model was chosen for the dyeing procedure of wool fibers. This table shows how terms of increasing complexity contribute to the total model. The probability value of linear model is 0.0002, which is much lower than 0.05. The ANOVA results in this case confirm the adequacy of the linear model ($F = 7.63, P < 0.05$).

The ANOVA results of applied linear model to the experimental design space of wool fibers dyeing with *Prangos ferulacea* are shown in Table 3. The probability value of the independent variables (dye concentration of *Prangos ferulacea*, mordant concentration, dyeing temperature, and dyeing time) is an indicator to determine the significant or insignificant factors on the dyeing condition of wool fibers with *Prangos ferulacea*. Therefore, a probability value less than 0.05 indicates that the effect of this independent variable on the response (color strength) is significant. From Table 3, it can conclude that the mordant concentration, dyeing temperature, and dyeing time are the significant independent factors on the color strength of dyed wool fibers with *Prangos ferulacea*, due to their low probability values and their high F -values.

The effect of dyeing temperature at two different mordant concentrations on the dyeability of wool fiber with *Prangos ferulacea* is presented in Figure 2. The effect of temperature on color strength (K/S) values was evaluated by dyeing different samples of wool fibers with *Prangos ferulacea* leaf extract solution and aluminum sulfate as mordant. The K/S values obtained are shown in Figures 2(a) and 2(b). It is clear that the color strength (K/S) values increase with an increase in the dyeing temperature of wool fibers. Increasing the dyeing temperature from 50°C to 90°C causes the K/S values of dyed samples to improve. This is due to the higher molecular energy of dye and fiber at the higher temperature that results in higher exhaustion (Figure 2(a)). In addition, the use of higher temperatures leads to increase in the rate of diffusion of dye molecules into the fibres. However, this effect is increased significantly in the presence of aluminum sulfate as a mordant (Figure 2(b)). The effect of dyeing time and mordant concentration on the dyeability of wool fiber

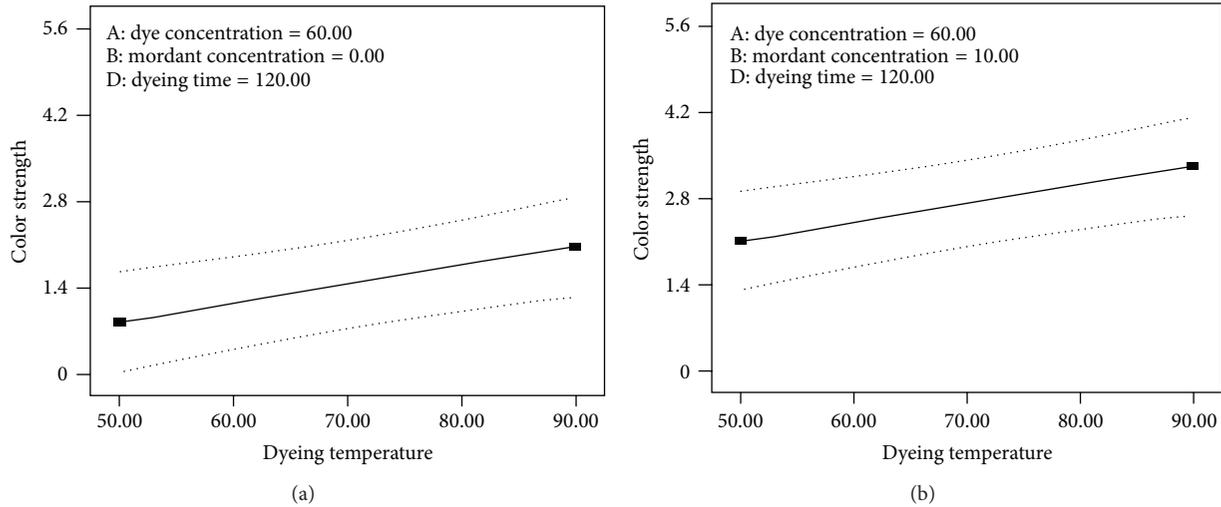


FIGURE 2: The effect of dyeing temperature on the color strength of dyed wool fibers with *Prangos ferulacea* at different mordant concentrations (a) 0% and (b) 10% (the dotted lines are the confidence bands).

TABLE 4: The proposed optimized condition for dyeing of wool fibers with *Prangos ferulacea* which is obtained from RSM results.

Dye concentration (%, owf)	Proposed optimum condition			K/S	
	Mordant concentration (%, owf)	Dyeing temperature (°C)	Dyeing time (min)	Actual value	Predicted value
60	10	90	120	3.45	3.33

dyed with *Prangos ferulacea* is presented in Figure 3. It is clear that the color strength (K/S) values are increased with an increase in the dyeing time of wool fibers, because diffusion rate of dye molecules into the fiber structure depends on the time (Figure 3(a)).

In addition, transition metal ions which have been used as a mordant in the textile substrate usually have strong coordinating power. Mordant can act as bridging material to create substantivity of natural dyes into a textile material being impregnated with such metallic salt. However, natural dyes usually have some mordantable groups to facilitate fixation of such dye [15]. It can be seen in Figure 3(b) that the K/S values increase with an increase on the mordant concentration and the dye exhaustion is greater at the higher mordant concentrations.

3.2. Optimization of Dyeing Condition. The dyeing condition of wool fibers was optimized using the optimal function of the Design Expert software. The dyeing condition was chosen in the proposed range that was adjusted in the highest amount of color strength. According to this, the proposed optimized condition of wool fiber dyeing is presented in Table 4.

The predicted value of color strength of the sample at the proposed optimum condition with *Prangos ferulacea* is 3.33, whereas the actual value of dyeing wool sample is 3.45 at the proposed condition. Comparison of experimental and predicted values (Table 4) revealed that there is a good correlation between them and thus confirms that the empirical model derived from RSM can be used to adequately describe

the relationship between the factors and the response in *Prangos ferulacea* dyeing of wool fibers.

3.3. Surface Morphology of Plasma Pretreatment of Wool Fibers. Scanning electron microscopy (SEM) micrographs of untreated and plasma pretreatment of wool fibers are presented in Figure 4. Structurally, a wool fiber is an assembly of cuticle and cortical cells [16], and the overlapped cuticle cells cover the surface of wool fiber (Figure 4(a)). It clearly shows that the plasma pretreatment has an etching effect on the surface of wool fibers compared to the untreated wool fibers and reduces the scale on the surface of wool fibers. Plasma pretreatment results in physical alteration on the epicuticle layer of wool fiber surface [17], which is being a hydrophobic barrier hindering dye diffusion inside the fiber. An increase in the plasma treatment duration causes smoother wool fibers.

3.4. Effect of Plasma Pretreatment on the K/S . The effect of plasma pretreatment on the color strength values of dyed samples compared to the one dyed at optimum condition was investigated. Plasma pretreatment on wool fibers results in an increase of dye diffusion into the wool fiber which can reduce the dyeing temperature and time. According to this, the plasma pretreated samples were dyed at a shorter dyeing time (30 min) than the proposed optimum condition. The obtained results of the K/S values of dyed wool samples are presented in Figure 5.

It can be seen that pretreatment procedure less than one minute has a little effect on the color strength of dyed samples,

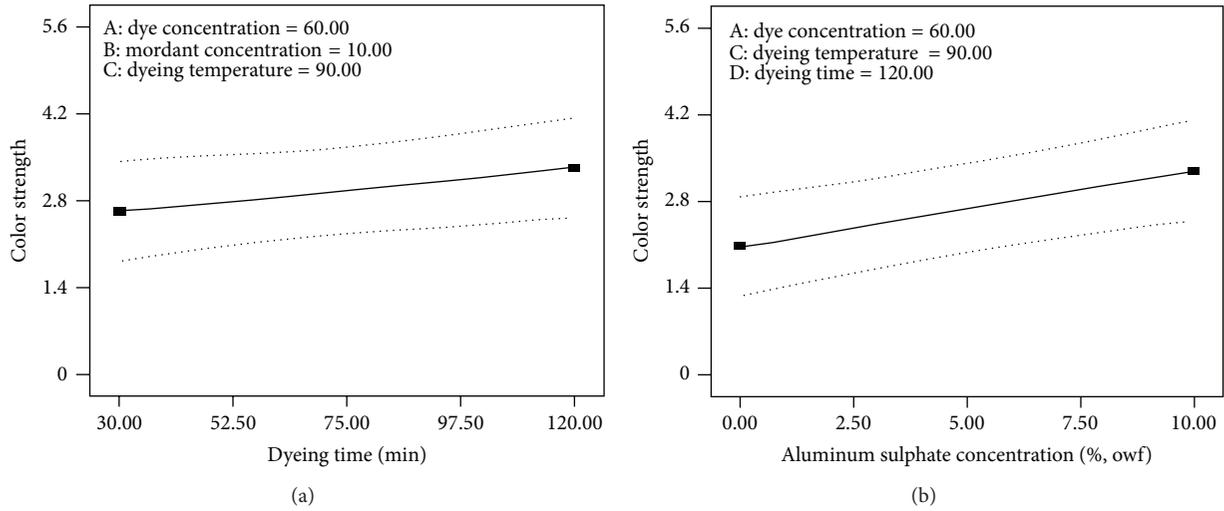


FIGURE 3: The effect of independent variable dyeing conditions on the color strength of dyed wool fibers with *Prangos ferulacea*. (a) Dyeing time, and (b) aluminum sulphate concentration (the dotted lines are the confidence bands).

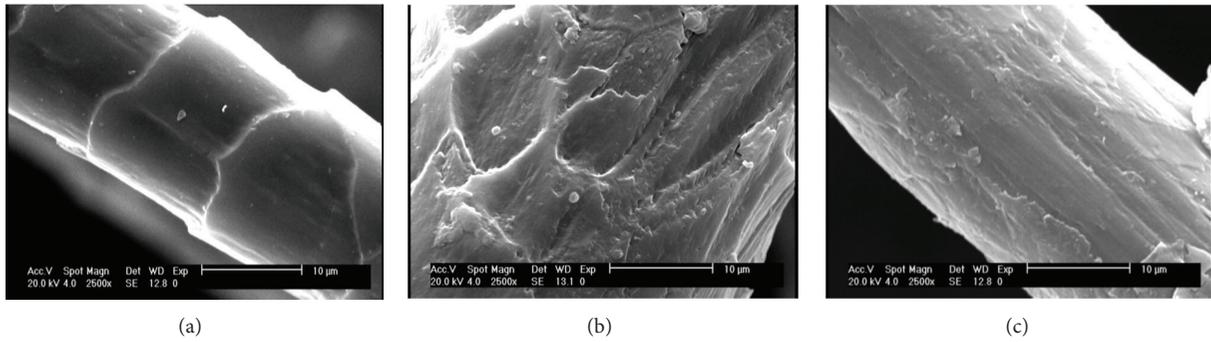


FIGURE 4: SEM image of wool fiber surface morphology: (a) untreated and (b) 2 and (c) 6 minutes of plasma pretreatment before dyeing procedure.

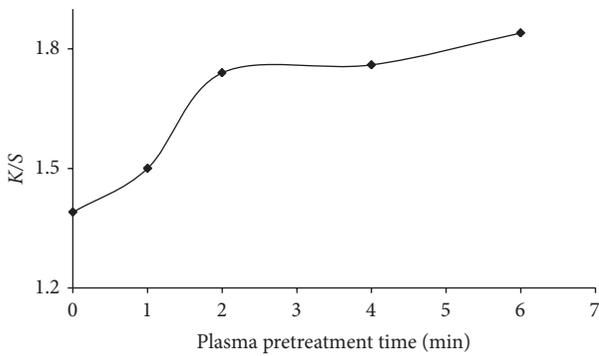


FIGURE 5: The effect of plasma pretreatment time on the color strength of dyed wool fiber with *Prangos ferulacea*.

while increasing the plasma treating time up to 2 minutes leads to higher color strength. This is due to the altering of the surface of the wool fiber by plasma treatment which results in higher uptakes of dyestuff. Plasma pretreatment of wool fibers

TABLE 5: Fastness properties of dyed sample with proposed optimized condition with and without mordant.

Fastness	Sample	
	With mordant	Without mordant
Washing	4-5	3
Staining on wool fibers	5	5
Light	6-7	4-5

results in an increase at the diffusion rate of dye molecules into the wool fiber which can reduce the dyeing time [14].

3.5. Fastness Properties of Dyed Samples. The color fastness to washing and light of the sample dyed with *Prangos ferulacea* is presented in Table 5. The samples were dyed at optimized proposed conditions with and without mordant in order to find out the effect of mordant on its fastness properties. Washing fastness results of these samples were assessed according to grayscale, and the results of light fastness were

assessed according to blue scale. Presence of mordant at the dyeing recipe results in comparable value of washing fastness. The light fastness result of the dyed sample indicates that the use of aluminum sulphate concentration as a mordant was advantageous for wool fibers dyed with *Prangos ferulacea* dye. Aluminum ions as mordant have a strong affinity to wool fibers and easily serve as a bridge between multiple dye molecules and/or between the fiber and dye [18]. The presence of mordant in the wool fiber increases the color strength of dyed sample due to higher dye uptake and as well as enhance the fastness properties of dyed samples.

4. Conclusion

Wool fibers are successfully dyed with *Prangos ferulacea* as a natural dye. The dyeing conditions of wool fibers with this natural dye were optimized by response surface methodology (RSM). The dyeing temperature with and without mordant increased the color strength value of dyed samples. In addition, an increase in the dyeing time and mordant concentrations leads to higher color strength value. Plasma pretreatment of wool fiber results in altering the surface of the wool fiber and enhances *Prangos ferulacea* dye uptake of wool fibers. The presence of mordant in the dyeing wool fiber with *Prangos ferulacea* improves its fastness properties.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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