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Natural dyeing of wool with Arnebia euchroma optimized by plasma treatment and response surface methodology

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Abstract

The aim of the study was to investigate the possibility of dyeing of wool fibers with the aqueous extract of *Arnebia euchroma* and improve the dyeability of wool fiber using a simple environmentally friendly pretreatment with oxygen plasma. The process of plasma pretreatment and dyeing was optimized by response surface methodology. Four independent factors including plasma time, Alum mordant concentration, dyeing temperature and pH were selected and the effects of these factors on the color strength of the dyed samples were examined and optimized using D-optimal approach.

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Introduction

Natural dyes are found in different parts of plants including bark, leaf, root, fruits or seeds, and flower that contain coloring materials such as tannin, flavonoids and quinonoids. The natural dyes also come from some types of insects and microorganisms such as fungi, algae and bacteria (Kasiri and Safapour 2014). They have been used since ancient times. With the invention of synthetic dyes in 1856, the use of natural dyes decreased significantly because of the advantages of synthetic compared to natural dyes including: color fastness, good reproducibility of shades, brilliance of color and ease of use (Tayade and Adivarekar 2013; Haji 2013).

Recently, a resurgence of interest in natural dyes has occurred mainly due to their environmentally friendly characteristics. They are clinically safer than their synthetic analogs in handling and use and have several advantages to several applications such as non toxic functions, specific medical actions and environmentally friendly finishes. Furthermore, the strict regulations and laws enacted by governments is another driving force for interest in natural 'green' dyeing. The use of natural dyes can significantly minimize the volume of toxic effluents resulting from the conventional dyeing processes (Savvidis *et al.* 2014; Haji 2012; Tayade and Adivarekar 2013).

Arnebia euchroma (Abukhalsa) is one of the plants representing a source of natural colorant. It belongs to the family of Boraginaceae and its root contains a copious purple *dye*. Eight active naphthazarins including alkannin, its enantiomer shikonin (Fig. 1), and their analogues has been isolated from the extract of *Arnebia euchroma* (Shen *et al.* 2002).

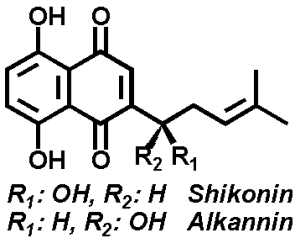

Fig. 1. Chemical structure of Aklannin and Shikonin.

According to previous studies, plasma treatment can improve the dyeability of wool with natural and synthetic dyes (Haji and Shoushtari 2011). In this study, the aqueous extract of *Arnebia euchroma* was used to dye wool fibers. Plasma treatment prior to dyeing was employed to improve the dye absorption. The aim of the study was to investigate the possibility of replacement of toxic mordants with plasma pretreatment as an environmentally friendly treatment. The effects of plasma time, Alum mordant concentration, dyeing temperature and pH on the color strength of the dyed samples were studied.

Materials and methods

Materials

Wool yarn with linear density of 200 Tex was scoured with 1% non-ionic detergent at 50 °C for 30 min, then dried at ambient temperature *Arnebia euchroma* was obtained from a local store and powdered after washing and drying. All chemicals used in this study were of analytical grade and purchased from Merck. Distilled water was used throughout the work.

Methods

Experimental Design

Design Expert software (version 7.0) was used for the design of experiments and statistical analysis of responses. In this study, the response surface methodology (RSM) and D-optimal design were applied to optimize the four important operating variables of the dyeing process. The corresponding codes besides lower and higher values for each variable are listed in Table 1.

Table 1. Experimental ranges of factors.

Factor	Name	Unit	Low Level	High Level
A	Dyeing pH	-	5	9
B	Alum Concentration	% owf	0	10
C	Dyeing Temperature	°C	50	100
D	Plasma Time	min	0	5

Plasma Treatment

The woolen yarns were pretreated using radio frequency (13.56 MHz) low pressure plasma

equipment (model: Junior plasma, Europlasma, Belgium) with oxygen gas. The sample chamber was evacuated to 150 mTor and maintained at this pressure during process. Then, oxygen was introduced with a flow rate of 30 sccm (Standard Cubic Centimeters per Minute). Plasma was generated at 150 W for predefined time. After that, air was introduced into the chamber and the plasma treated sample was removed.

Mordanting

The scoured wool yarns were mordanted using the predefined amount of Alum (Potassium Aluminum Sulfate) at 80 °c and L:G = 30:1, for 45 minutes. After mordanting the samples were rinsed with distilled water (Haji 2012).

Dyeing

Dyeing of the samples was performed using 50% owf of the dye powder (L:G= 40:1, pH= 5-9, Temperature= 50-100 °C). The dyeing was started at 40°C and the temperature was raised to final temperature at the rate of 2°C per minute. Then the samples remained in that condition for 1 hour, and then rinsed and air dried. All dyeing and mordanting processes were carried out using a laboratory dyeing machine made by Rissanj co.-Iran.

Color measurements

The reflectance of dyed samples was measured on a Color-eye 7000A spectrophotometer using illuminant D65 and 10°standard observer. For each sample, three measurements on three different places were made and the average values were reported. Color strengths (K/S) of dyed samples were calculated using kubelka-munk equation:

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

Where R is the observed reflectance, K is the absorption coefficient and S is the light scattering coefficient.

Results and discussion

25 samples were prepared according to the experimental design. The color strengths of the dyed samples were calculated and fed as the response to the Design Expert software for statistical analysis. The data were fitted to various models and the described process was most suitably described with quadratic model.

Table 2. ANOVA results of the established model for responses.

Factor	F-Value	P-Value
Model	14.02	<0.0001
A: Dyeing pH	5.29	0.0344
B: Alum Concentration	6.82	0.0182
C: Dyeing Temperature	59.80	<0.0001
D: Plasma Time	15.80	0.0010
AB	12.29	0.0027
BD	3.47	0.0798
D^2	8.41	0.0099
Lack of Fit	2.40	0.1718

Table 2 shows the analysis of variance (ANOVA) results of the established model for responses. The model F-value of 14.02 implies that the model is significant and there is only a 0.01% chance that a "Model F-Value" of this large value could occur due to noise. Values of Prob>F less than 0.05 imply that the model terms are significant at the 95% confidence level, whereas the values greater than 0.1 are usually considered as insignificant. In this case A, B, C, D, AB, BD and D^2 are significant model terms. The insignificant interactions of parameters were removed using the backward function of the software. The "Lack of Fit F-value" of 2.40 implies the Lack of Fit is not significant relative to the pure error. A high R^2 coefficient advocated a satisfactory adjustment of the proposed model to the experimental.

"Adeq Precision" measures the signal to noise ratio. It compares the range of predicted values at design points to the average prediction error. A ratio greater than 4 is desirable and indicates adequate model discrimination (Rahbar and Haji 2013). In this case, the ratio of 11.865 indicates an adequate signal. This model can be used to navigate the design space.

Regression analysis of experimental data was performed and the model equation in terms of coded factors is as follows:

$$K/S = 1.43 + 0.16A - 0.20B + 0.54C + 0.29D - 0.30AB + 0.16BD + 0.41D^2 \quad (2)$$

The effects of parameters on color strength

Fig.s 2-5 illustrate the effect of each parameter on the color strength of dyed samples. It is evident from fig. 2 that this natural dye has been slightly higher color strength on wool fiber in alkaline dyebath. This may be due to effect of alkali on the chemical structure of the dye molecules and shift in the adsorption of dye molecules in alkaline media.

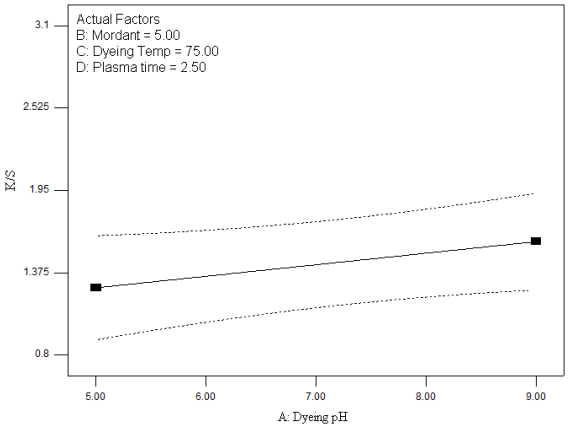

Fig. 2. The effect of the dyebath pH on color strength.

Fig. 3 shows the effect of mordant concentration on the absorption of the natural dye by wool fibers. Unlike most natural dyes, using alum mordant decreased the affinity of the fiber. The non-mordanted and plasma treated samples, showed acceptable color strengths and it shows that plasma treatment can be used to eliminate or at least reduce the amount of toxic mordants usually used with natural dyes.

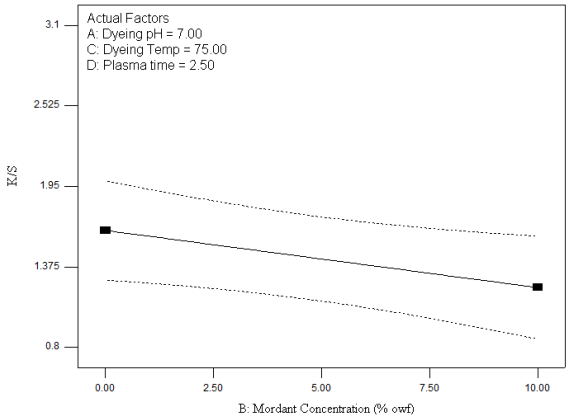

Fig. 3. The effect of mordant concentration on color strength.

Fig. 4 shows that by increasing the dyebath temperature the color strength has been increased. This is due to the breakdown of the dye aggregates, increase in the dye kinetic energy and swelling of the fiber at elevated temperatures.

Fig. 5 shows the effect of plasma treatment on the dyeability of wool fibers with the natural dye used in this study. The surface of raw wool fiber is covered with compact and intact scales, which makes the wool fiber surface difficult to get wet and diffused by dye molecules. However, after plasma treatment for more than 2.5 min, the surface scales of the wool fibers are destroyed due to the etching effect caused plasma leading to better dyeing of the fibers and increasing the color strength of dyed samples. This has been confirmed by SEM observations in our previous studies (Haji and Shoushtari 2011).

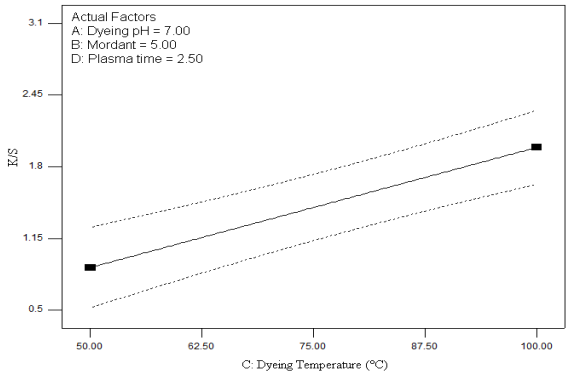

Fig. 4. The effect of dyeing temperature on color strength.

Optimization of dyeing process

The maximum color strength was taken as the desired response and the optimal conditions for obtaining the highest dye uptake 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 3 (desirability = 1).

Table 3. The optimal conditions for obtaining the highest color strength.

Factor	Dyeing pH	Alum Concentration	Dyeing Temperature	Plasma Time	K/S
Amount	9	0.14	95	5	3.07

It can be seen that with 5 minute plasma treatment, only a very little amount of mordant is needed to obtain the highest color strength compared to when high amounts of mordant are used. This finding is important regarding the environmental problems caused by the use of high amounts of mordants in natural dyeing processes.

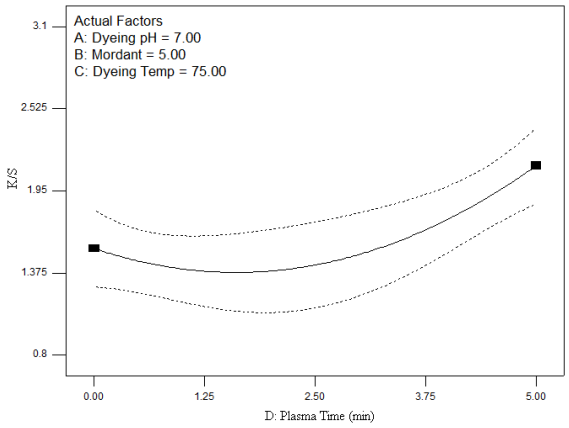

Fig. 5. The effect of plasma treatment time on color strength.

Conclusion

Low pressure oxygen plasma was applied on wool fibers prior to dyeing with *Arnebia euchroma* as a natural dye. The process of dyeing and plasma pretreatment was optimized using RSM. The plasma treated samples showed better dyeability. The samples dyed at higher pH and temperatures showed higher color strengths. The use of plasma as a pretreatment instead of mordanting increased the absorption of the dye to wool fibers. This dye can be used on wool fibers successfully and the dyeability can be improved by pretreatment with oxygen plasma.

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