Optimization of wool dyeing process with woad

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1. INTRODUCTION

The preference of natural dyes compare to the synthetic dyes is due to environmental friendly, lower toxicity, antibacterial properties, biodegradability, and harmonizing natural shades [1]. The major chemical structures of natural dyes are such as anthraquinone (madder), alpha napthoquinones (hena), flavones (weld), indigoids (indigo and woad), and carotenoids (annatto and saffron) [2]. The indigo is the most important and oldest natural blue dyestuff (C.I.NaturalBlue1) [3]. Indigo dyestuffs have been obtained from a variety of plant sources such as Indigofera, Polygonum tinctorium, and Isatis tinctoria (Woad). Woad is a biennial plant which it leaves grows at first year and produces blue dyestuff and at the second year it produces yellow flowers [4]. Indigo it doesn't exist in the leaves of indigo producing plants. Instead there are its precursors, indican in Indigofera species and Polygonum tinctorium [5] and isatans in woad [6].

Indigo is formed after the extraction of indigo precursors in the leaves of woad plants (isatans) [7]. These compounds are extracted by steeping leaves in warm water. With woad, the addition of alkali to the steep water releases free indoxyl, which forms indigo after avigorous aeration.

2. EXPERIMENTAL

Materials

Wool fibers with linear density of 200 tex was used. They were scoured with1% anionic detergent at 60˚C for 30min, and then used at dyeing process. The ammonia, sodium hydroxide, sodium carbonate, hydrogen peroxide, sodium dithionite was purchased from Merck Company. The woad, isatis tinctoria, as a natural dye was prepared from the Yazd province, Iran.

Dyestuff extraction

The extraction of dyestuff was done as follows; the well milled woad leaves were preserved in water solution in dark place according the time which proposed with experimental design. After that, the solution was passed through the filter, and finally the filtered solution was used at preparation of dyeing bath.

Dyeing and oxidation procedure

Wool fibers were dyed with woad as follows; sodium dithionite (a%, owf), alkaline agent, and dye solution which prepared from previous steps are mixed to gather and increased water which reaches to aproporiate liquor ratio. A yellow-green solution was obtained by the reduction of woad blue constituents in their leuco-forms. The temperature of dyeing bath was rose to the proposed temperature according to experimental design. Then, the wool fibers were added in the vat dye bath solution and left it for proposed time. Then it was removed and should oxidize the soluble forms in to insoluble form (indigoids). The three methods was used for oxidation steps which were was aerated for 15 minute, oxidized at fluent tap water for 5 minute, and oxidized at hydrogen peroxide (%5 w/w) for 5 minute. The dyed wool fiber was washed thoroughly with water and left to dry.

Tensile strength

The tensile properties of fibers were measured according to ASTM D 2256. Gage length was 10 cm and crosshead speed was 25 cm/min. The samples were chosen randomly by ten readings for each specimen.

Color Measurement

The reflectance spectra of the samples were measured by Color-Eye spectrophotometer from Gretag Macbeth in the visible region. The CIE terms namely, L*, a*, b* and C* color-coordinates under illuminant D65 and 10º standard observer were measured for evaluating the color of samples. Color strength as (K/S) values calculated from the reflectance factor at the wavelength of maximum adsorption (λmax= 650 nm).

Design of experimental with D-optimal

D-optimal design is very attractive for researchers, as its application provides maximal accuracy in estimating regression coefficients. In this study, the dyeing condition of wool fibers with woad was optimized by RSM using the trial version of Design Expert 8.0.1.0 from Stat-EaseInc (USA). The independent variables were coded with low and high levels in D-optimal design as shown in Table 1, while color strength (K/S) and tensile properties were the response (dependent variable).

The D-optimal designed experiments were augmented with five replications in order to evaluate the pure error and were carried in a randomized order as required in many design procedures.

3. RESULTS

The model adequacy check is an important part of the data analysis procedure as the approximating model would give poor or misleading results if it were an in adequate fits. Data are fitted to various models and their subsequent ANOVA showed (Table 1). The dyeing process of wool fibers with woad were most suitably described with linear model. The probability value is 0.0001 which is much lesser than the 0.05 and the high F value (6.56) indicate that this model is significant.

Table 1. ANOVA results of the fitting of the experimental data to various models

Responce	Source model	Sum of squares	df	Mean Square	F Value	Prob > F
Color strength K/S	Mean vs Total	1798.69	1	1798.69		
	Linear vs Mean	271.98	10	27.20	6.56	< 0.0001
	2FI vs Linear	195.85	43	4.55	1.50	0.1910
	Quadratic vs 2FI	15.51	6	2.58	0.78	0.6034
	Cubic vs Quadratic	20.74	5	4.15	1.68	0.2916
	Residual	12.35	5	2.47		
	Total	2315.11	70	33.07		

ANOVA results of the first order model of woad dyeing of wool fibers are presented in Table 1, indicating that this linear model can be used to navigate the design space. The applied approximating model is examined by the residual plots. The normal probability and studentized residuals plot is shown in Fig. 1. 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 variance (ANOVA).

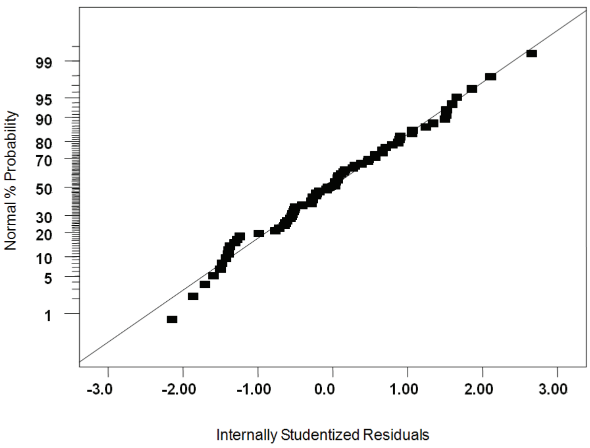

Fig. 1. The normal probability versus studentized residuals plot.

Studentized residuals in Fig. 1 are the residuals divided by the estimated standard deviation of that residual. It measures the number of standard deviations separating the actual and predicted values. The normal probability plot indicates whether the residuals follow a normal distribution, in which case the points will follow a straight line.

4. CONCLUSION

The optimum condition for the dyeing of wool fibers with woad was predicted using the optimization function of the Design Expert Software. The independent Numerical factors such as reducing agent percentage, dyeing temperature, dyeing procedure time, and dye percentage are the significant terms of this model.

5. ACKNOWLEDGMENT

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