STUDY ABOUT THE POSSIBILITY OF DYEING NATURAL PROTEAN FIBRES (WOOL), WITH NATURAL PIGMENTS EXTRACTED FROM SAFFRON FLOWERS (*Carthamus tinctorius* L.)

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Abstract

This paper shows the results obtained for dyeing protean fibers (wool) with carthamin, which is a natural pigment extracted from saffron flowers (*Carthamus tinctorius* L.). We aimed mainly for the optimisation of the dyeing process in terms of investigation of the quantity of natural pigment ingrained on wool fiber. The used method for this study was the multiple regression method which through data processing on computer has lead to the getting of a curve in space and in plan which allowed the establishment of optimal parametres for dyeing: pigment concentration, temperature of dyeing bath and duration of dyeing process.

Key words: : dyeing natural, carthamin, multiple regression method, protean fibres

Saffron, Carthamus tinctorius, L., is an annual plant, which is cultivated mainly for oil which is extracted from akenes (fruits), used in food industry because of high concentration of saturated fatty acids and especially of linoleic acid (vitamin F).

Flowers of this plant are used to extract carthamin (a very durable red pigment, also called “Safflower Red”) and a yellow pigment (“Safflower Yellow”), which is supposed to be 2 geometrical isomeres, quite unstable (“Safflomin A” and “Safflomin B”), of wide use in food, pharmaceutical, cosmetic and textile industries.

In terms of chemistry these pigments belong to flavones class, and in terms of technology they have a structure close to that of dispersion pigments. In Colour Index we find the carthamin in position C.I. Natural Red 26-75140 (Colour Index, 1982) (figure1).

In terms of structure, the carthamin holds in its molecule 5 carbonyl groups (>C=O), which form the chromophores (they are groups of atoms which colour the substance, groups “which bring colour”) and 13 hydroxyl groups (-OH) which play the role of auxochromes (they are groups of atoms which intensify the colour).

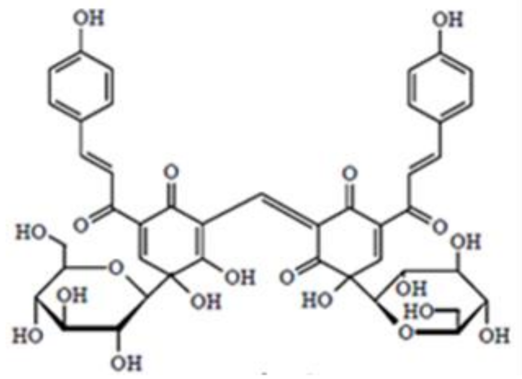

Figure 1 **Structure carthamin**

The first informations about use of vegetal pigments date around 2650 B.C. In China. Since then and until now, man has been always interested in using these natural resources, close to him, for dyeing and decorating textile materials both for utilitarian interest and aesthetic reasons and also for killing boredom of daily life. (Jolin&Margaret Cannon, 1994,).

Of all the protean natural fibers, wool – because of its complex physical-chemical composition- presents an exceptional elastic and tinctorial behaviour.

Therefore through the presence of polypeptide bonds (-NHCHRCO-) and isopeptide bonds and disulfide bonds -bonds which are

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created during the growth of fibre through the so called process of keratinisation -and also of ionic bonds (type salt bonds) between lateral acyclic bonds (carboxylic) and basic (amic) which give an amphoteric character to wool fibers, make wool fibers a textile support with a superlative behaviour.

MATERIAL AND METHOD

For this study there have been used samples of sheep fur, of endemic production, with a length of wool yarn of 15 mm.

We aimed mainly the optimisation of dyeing process in terms of investigating the quantity of natural pigment ingrained on wool fibre. For that we used as experimental instrument the mathematical modelling, using factorial programming in a central composite rotatable 2k program, considering the use of a mathematical model with three independent variables to be expressive and efficient because these have the quality of taking in account a reasonable number of technological parametres, with a higher degree of covering and conclusions can be applied with a higher degree of probability (Mihail, R.,1976; Gluck, A., 1971; Ciocoiu Mihai, et al., 2002).

The general form of mathematical model is:

$$y = b_0 + \sum_{i=1}^{k} b_i x_i + \sum_{i=1}^{k} b_{ii} x_i^2 + \sum_{\substack{i=1\\ j=1}}^{k} b_{ij} x_i x_j$$

The general form of mathematical model with three independent variables is:

$$y = b_0x_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2$$

As independent variables we chose dyeing duration, pigment concentration and temperature of dyeing bath, and as dependent variable we chose a quantity which reflects the two major aspects in the process development, respectively tinctorial characteristics and those regarding textile material integrity, namely the pigment quantity ingrained on wool fibre, Y.

Through this method we aimed to obtain statistical mathematical models which can correlate the variation module of investigated coefficients with development parametres of tinctorial process, method which holds two consecutive directions of work: the programming of experiments and the analysis of experimental data.

For this study we have taken the parametres presented in Table 1. which show the values of these in coded and real units.

Table 1

Coded and real values of independent variables

Parameter	U.M.	Coded Value				
		-1.682	-1	0	+1	+1.682
		Real Value				
Dyeing duration, X_1	min	40	50	65	80	90
Pigment concentration, X_2	g/l	0.184	0.360	0.600	0.840	1.016
Dyeing temperature, X_3	°C	39.4	46.0	55.0	64.0	70.6

For the establishment of independent variables limits we chose a temperature interval between 39,4 °C and 70,6°C because in the scientific literature it is stated that at temperatures higher than 65-68 °C, the red pigment (carthamine) becomes unstable, the colour going for yellow.

The determination of pigment quantity ingrained on wool fiber (from fur) has been realised with the help of a spectrophotometer SPECTROFLASH SF-300- Data Color through the measurement of K/S proportion (function Kubelka-Munk which represents a relation between the optical properties of dyed textile materials and the concentration of pigment in material, respectively the concentration of pigment used for dyeing, using the optical properties of dyeing – light scattering and absorption – and luminance at the wave length where the absorption of light energy is maximised, λ_{max}) (Pustianu, M., et al., 2002), at wave length of 520 nm. For the difference of colour we used the relation DE*CIELAB.

Data obtained from measurements is presented in column $Y_{măs}$ of experimental matrix and have been processed with the help of library program MATHLAB (found in the facilities of lab), which carries out the following:

- Calculus of the values of coefficients of regression equation;
- Check-out of the signification of numeric coefficients, b, by Student test;
- Check-out of the adequacy of obtained mathematical model, through Fisher test.
- Graphic representation in 2D and 3D.

RESULTS AND DISCUSSION

The experimental plan and results for the pigment quantity ingrained on wool fiber, for each method are presented in *table 2*:

Table 2

Experimental matrix and results

Nr. crt.	X_{1cod}	X_{2cod}	X_{3cod}	X_{1real}	X_{2real}	X_{3real}	$Y_{measuriment}$
1	-1	-1	-1	50	0.360	46.0	47.11
2	+1	-1	-1	80	0.360	46.0	44.23
3	-1	+1	-1	50	0.840	46.0	41.13
4	+1	+1	-1	80	0.840	46.0	35.67
5	-1	-1	+1	50	0.360	64.0	46.22
6	+1	-1	+1	80	0.360	64.0	43.09
7	-1	+1	+1	50	0.840	64.0	43.18
8	+1	+1	+1	80	0.840	64.0	33.84
9	-1.682	0	0	40	0.600	55.0	39.09
10	+1.682	0	0	90	0.600	55.0	39.97
11	0	-1.682	0	65	0.184	55.0	34.12
12	0	+1.682	0	65	1.016	55.0	57.77
13	0	0	-1.682	65	0.600	39.4	40.24
14	0	0	+1.682	65	0.600	70.6	48.49
15	0	0	0	65	0.600	55.0	49.76
16	0	0	0	65	0.600	55.0	41.65
17	0	0	0	65	0.600	55.0	40.42
18	0	0	0	65	0.600	55.0	52.67
19	0	0	0	65	0.600	55.0	45.33
20	0	0	0	65	0.600	55.0	46.21

After verifying the signification of multiple correlation coefficients, by applying the Fisher test, whose calculated value is F=30,742 and by elimination of inconsistent terms, by the smallest squares method, we obtained the following regression equation:

$$Y = 41,1856+3,7574x_1+3,3548x_2+7,8359x_3 +1,1738x_1x_2 +0,7938x_1x_3 +4,1113x_2x_3+0,7610x_1^2 - 3,8733x_3^2$$

To follow the influence of independent variables exerted on dependent variable Y, on the base of presented data in table 2 we realised 3D representations of response surfaces (figures 2 ÷ 4), diagrams which contain the representation of dependencies $Y=f(X_1,X_2)$, $Y=f(X_1,X_3)$ și $Y=f(X_2,X_3)$, when the 3rd independent variable is constant and equal with the adequate value of experimental field center.

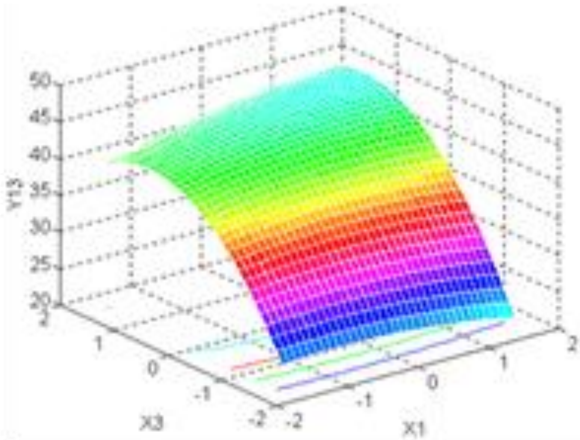

Figure 3. **Influence of parametres X_1 and X_3 on variable Y**

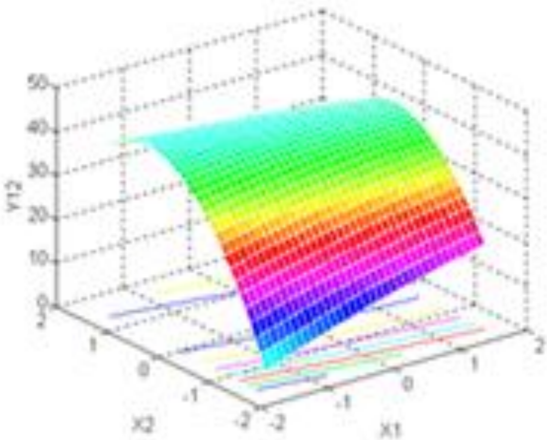

Figure 2. **Influence of parametres X_1 and X_2 on variable Y**

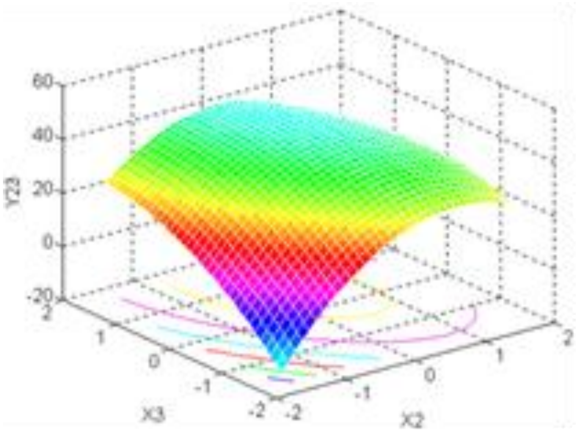

Figure 4. **Influence of parametres X_2 and X_3 on variable Y**

Both in resulted mathematical model equation and in graphical representations we can see that all three independent variables exert a positive influence on variable Y, but from all the temperature (X_3) is the most important for increasing the quantity of pigment ingrained on wool fibre. This can be observed most clearly when we analyse the variation of ingrained pigment quantity for a single parametre, the other two being constant (figure 5).

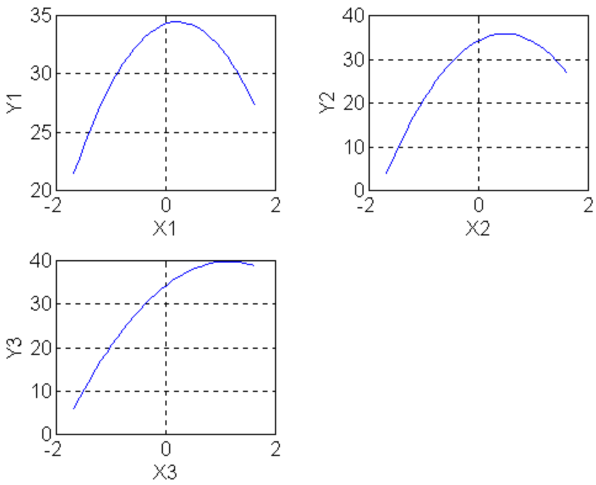

Figure 5. **Influence of the three variables on purpose function Y**

We can see that purpose function Y has the following values:

x_1 cod = 0 x_1 real = 65 minutes
x_2 cod = +0,5 x_2 real = 0,720 g/l
x_3 cod = +1 x_3 real = 64 °C

So, we can say that the maximum effective power for carthamin quantity absorbed by wool fiber has been obtained in the above conditions. The overhaul of 64 °C (x_3 cod = +1) temperature, leads to a decrease of tinctorial effective power, which can be explained by the fact that at temperatures higher than 65 °C takes place a turning of colour from red to yellow.

CONCLUSION

Both from the analysis of obtained mathematical model equation, and from diagrams we can can see that the influence of parametre x_3 (temperature) is higher on the effective power of reaction of dyeing wool fibres with carthamine.

We established the optimal values of investigated parametres to obtain a maximum effective power of dyeing:

x_1 = 65 minutes
x_2 = 0.720 g/l
x_3 = 64 °C

The mathematical modelling of dyeing process of natural protean fibres (wool) with natural pigments, has shown the fact that the quality index doesn't increase constantly with the increase of the values of the three investigated parametres, dependencies presenting both minimum and maximum points.de bază

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