OPTIMIZATION OF ANNATTO (*Bixa orellana* L.) DRYING IN FIXED BED

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Abstract - The drying of annatto seeds (*Bixa orellana* L.), red piave cultivate, was studied in a fixed bed dryer. The best conditions were estimated to minimize the loss of coloring and to obtain final moisture of the seeds in appropriate levels to its conservation and maintenance of quality. The quantification of the influence of entrance variables in the final contents of bixin and moisture seeds and the identification of the optimal point was performed through the techniques of factorial design, response surfaces methodology, canonical analysis and desirability function. It was verified that the final moisture of the seeds may be estimated by a second-order polynomial model and that the final content of bixin is only significantly influenced by the time of drying being described properly by a linear model, for the seeds used in this study.
Keywords: drying, response surfaces methodology, annatto.

INTRODUCTION

The tendency nowadays demonstrated by the international market to restrict the use of artificial coloring in food, allied to the demand of consumers for natural products also in the pharmacy cosmetics industry (Nothenberg, 1997), has risen the interest of researchers and of the industry for natural raw material (vegetal particularly), which are used as coloring. On this aspect, seeds of annatto (*Bixa orellana* L.) are brought out as an important raw material found in the country for the obtention of coloring due, principally, to its characteristics of natural product, non-toxic, high coloring strenght and wide range of colors.

The trading way of seed, according to quality, mainly the coloring content and moisture proper for conservation, stimulates the producer to obtain seeds of better quality, making the country more competitive in the international market. Brazil is nowadays considered the third most important world exporter of annatto seeds just after Peru and Kenya. However, the qualitaty reputation of the Brazilian product in the international market is considered poor (Ohashi *et al*., 1992)

The improvements in basic researches in the area of genetics and citogenetics, which is important for productivity of the annatto tree, do not reflect directly in a more competitive product in the international market. The agronomic field efforts won't be enough if the treatments that the product receive in the post-harvest or pre-processing operations are not proper and contribute to the reduction of the quality of the seeds.

Among the operations of pre-processing, which include the improvement, drying and storage, drying represents a very important step which should be conducted carefully in order to maintain the wished level of quality (minimum bixin content of 2.5%wb and moisture lower than 10%db) in the final products. Due to the complexity expected when working with natural products, the development of the drying process and the quantification of relationships of the operational entrance variables should be carried out using proper statistics methods.

Bixin is the carotenoid found at the highest concentration in the annatto seeds. This pigment presents instability, being able to degrade when exposed to certain conditions of temperature, light and time of exposition during the process of drying.

According to Simpson (1985), the carotenoids are altered or partially destroyed in acids fragmented by the action of some enzymes and sensible to light exposure, generally stable to operations of thermal treatment, but degrade quickly in usual processes of drying due to oxidation and to reactions of isomerization.

In the separation and analysis of bixin and of other pigments found in annatto seeds as well as the content of bixin and other total pigments found in annatto coloring, the information of literature has shown that bixin is very sensible to light. When the proper care is not taken, rapid photochemical decomposition and oxidation will occur (McKeown, 1961; McKeown and Mark, 1962; Reith and Gielen, 1971; Rouseff, 1988).

Hence, the drying process has a decisive influence in the quality of the obtained seeds, not being deeply studied and nowadays performed in rudimentary and not proper way by the great majority of producers.

The pigment (bixin) is located in the outer layer which covers the seeds and corresponds to approximately 6% of the total seed weight. Bixin can be extracted by attrition of the seeds.

Therefore, it is not advisable that the drying be conducted in moving beds, since friction among the seed can induce loss of coloring material. Therefore, the controlled drying in a fixed bed dryer can be viewed as a feasible alternative to reduce the moisture of the seeds to a value proper to its conservation and trading, maintaining intact the concentration and the quantity of bixin furnished originally by the plant. Hence, the general aim of this paper is to analyze the drying of annatto in fixed bed, and based on response surfaces methodology, to propose the statistic optimization for the seeds final content of bixin and moisture.

MATERIALS AND METHODS

Materials

Annatto seeds, cultivate type Red Piave, were furnished by Agroindustrial Biotropical of the Danish group Chr Hansen, owner of a farm of rational cultivation of annatto trees, located in the Pará State, Brazil. The seeds used in this work were harvested in the crops, in the second semesters of 1996 and 1997. The initial and final concentrations of bixin found in the seeds were analyzed by the KOH methodology (Takahashi, 1987).

Drying

The drying experiments were performed in a prototype of a fixed bed dryer developed by Faria (1998), having at the base an adapted unit able to move and heat the air. This basic equipment consists of a duct of 25.4 cm of square section, with a centrifugal blower with rotor speed control, able to transport the air to the bed of solids with controlled flow rate. Also, this system contains two pairs of electrical resistances, distributed along the unit, which enable the heating of the air used as the drying agent.

The fixed bed dryer is connected to the air unit through a gradual reduction, which changes the square section to a circular one, with 7.5 cm of inner diameter and 1.35 m of length. The tubes are made of stainless steel and thermally insulated with fiber glass.

The body of the drying bed is made of Pyrex glass, having sets to measure the temperature of entrance and exit air and also for sampling the air to evaluate its psychometric properties. The equipment has also a cylindrical net basket built in stainless steel, in which the annatto seeds are deposited. In this system, the seeds are exposed to the drying air at the base and through the lateral area of the basket.

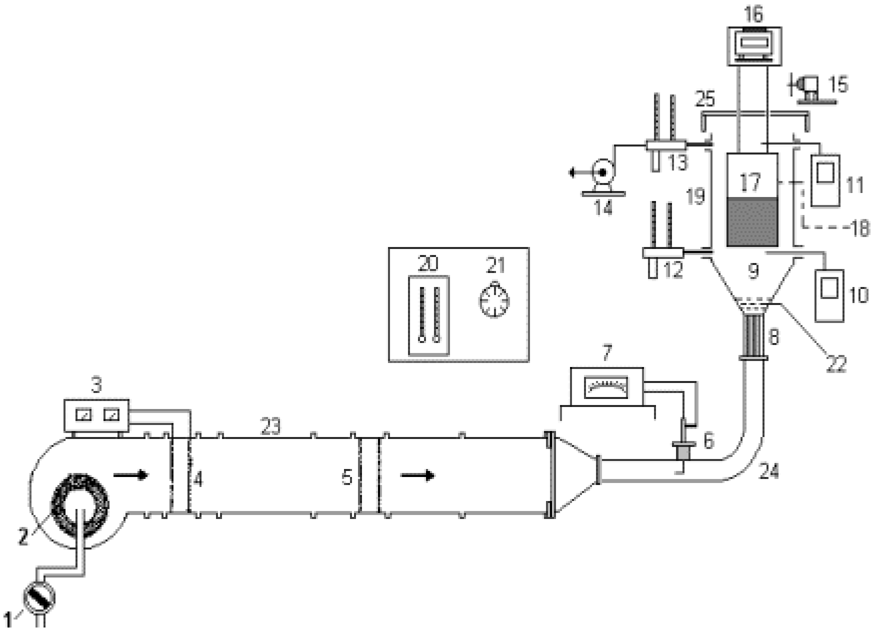
This basket, which constitutes the bed itself, has an inner diameter of 12.5 cm and height of 25.5 cm, making a total volume capacity of 3.15L. During the experimental runs, the basket is suspended inside the cylindrical body of glass and connected to an electronic balance (sensitivity of 0.1g), which indicates the mass decrease during drying.

The experimental arrangement is completed with an exhaustor, placed next to the balance with the objective of cooling the drying air in its exit.

The instrumentation of the prototype consists in a Pitot tube connected to a micromanometer used for indication of the inlet air mass flow rate and two thermocouples, type K, to indicate the temperatures of entrance and exit of the drying air.

The use of special designed psychrometers for the determination of psychrometric properties of the air in drying processes was recommended by Rocha and Faria (1992). Hence, two psychrometers built according to the recommendations of ASHRAE (1996) and Rocha and Faria (1992) were connected to the entrance and exit of the bed. The psychrometer at the exit of the bed used a vacuum pump, with negative pressure of 700 mm Hg, sucking the air at the outlet to maintain a proper speed (>5m/s) when it passes through the wet cloth, so that the thermometer indicates its wet bulb temperature with a minimum error (Wexler and Brombacher, 1951).

The environmental conditions before, during and after the process were monitored through thermometers of maximum and minimum and through a thermohygrometer, with a working range of 0 to 40^0C and 0 to 100% of relative humidity. The complete experimental assembly is showed in Figure 1.



1. control of speed of the blower
2. centrifugal blower
3. power controller
4. pair of resistances power of 2kW
5. pair of resistances power of 1kW
6. Pitot tube
7. micromanometer
8. air distributor
9. plenum chamber
10. temperature measurer of the air at the entrance
11. temperature measurer of the air at the exit
12. psychrometer (Ts and Tu at entrance)
13. psychrometer (Ts and Tu at exit)
14. vacuum pump
15. exhauster
16. precise electronic balance
17. seed layer
18. basket (bed)
19. body of the dryer bed
20. thermometer of maximum and minimum
21. thermohigrometer
22. air diffusion net
23. basic unit of drying
24. section of connection of the bed
25. sheet

→ air flow

Figure 1: Scheme of the fixed bed dryer prototype.

The experiments were performed in batch, samples of the seeds were collected at the end of the specified drying time, in order to determine the variation of bixin concentration. The seeds moisture content was determined using the oven method until constant weight. The material was left in the oven for 24 hours at 105° C. The initial and final solids temperature were measured in a calorimeter by a thermocouple.

Experimental Design

In the convective drying in fixed bed performed in this work, the air humidity was a non-controllable parameter, measured by the psychrometers installed in the equipment or through a thermal hygrometer located in the space next to the equipment.

The controllable variables, which originally may have a relevance in the behavior of the drying of the material are the ones which are related to the air (temperature and mass flow rate), to the solid (initial moisture content , load or height of the bed) and to the process (drying time).

The dried seeds of annatto should present final contents of moisture and bixin compatible with the patterns and quality of the desired product. These dried seeds are usually stored in the industrial plant for further processing to obtain the colorants. Therefore, the final contents of moisture and bixin were taken as response variables of interest when applying the statistical method.

Response Surfaces Methodology

The technique of response surfaces is employed in the optimization of processes which present a considerable number of entrance variables able to influence the responses. This technique permits the identification, among these factors, of the ones which will affect the process under analysis.

The strategy of the methodology consists on selecting models through which the responses may be expressed by the independent variables involved in the process. A good estimation of the experimental errors and of the surface curvature is obtained performing replications of experimental runs in the central point, which correspond to average values, between the lower and upper levels of the inlet variables considered. The statistical analysis of the data performed in this work was based on the fit of a second-order polynomial model, as described by Khuri and Cornell (1987).

According to these authors, the total number of runs, N_t, may be given by Equation 1:

$$N_t = n_f + n_\alpha + n_c \tag{1}$$

where n_c represents the number of repeated observations in the central design point and the number of experiments referring to the factorial

portions of design, n_f and axial, n_α , are obtained as:

$$n_f = 2k \tag{2}$$

$$n_\alpha = 2k \tag{3}$$

with k being the number of independent variables or factors, taken as 3 in this work.

For the experimental design to present uniform precision, the number of repetitions in the central point can be estimated by Equation 4:

$$n_c = 0{,}8385\left(\sqrt{n_f} + 2\right)^2 - n_f - n_\alpha \tag{4}$$

The central composite design may have the property of rotation and it is simple to calculate the value of the axial space, α , through the following equation:

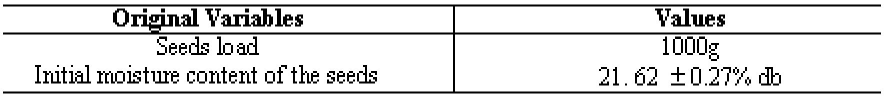
$$\alpha = n_f^{1/4} \tag{5}$$

According to equations (2) to (5), the central composite design with three factors (k=3) used in this paper for pursuing the properties of uniform precision and rotation resulted in $n_f = 8$; $n_\alpha = 6$; $n_c = 6$ and $\alpha = \pm 1{,}68$; and a total of 20 experimental runs ($N_t = 20$).

To obtain statistical models able to properly predict the final contents of moisture and bixin in the annatto seeds, it was developed a statistic design based on the Response Surfaces Methodology, using as inlet variables the temperature and mass flow rate of the air and the drying time.

The value of the average initial moisture content of the annatto seeds, shown in Table 1, was obtained as the mean value of the initial moisture contents of the 20 experimental runs. The samples used in the experiments remained at constant conditions of temperature and relative humidity for a convenient time, which allowed an homogeneity in their initial moisture contents.

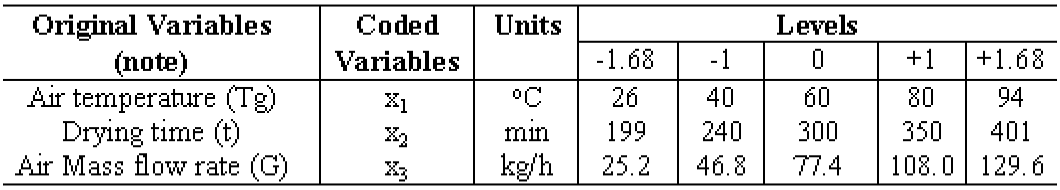
Table 1: Variables fixed in the experimental planning.



Original Variables	Values
Seeds load	1000g
Initial moisture content of the seeds	21.62 ±0.27% db

In Table 2 are represented the variables and the respective levels used in the optimization of the fixed bed drying of annatto seeds.

Table 2: Coded and original values for variables used in optimization by the response surfaces methodology.



Original Variables (note)	Coded Variables	Units	Levels				
			-1.68	-1	0	+1	+1.68
Air temperature (Tg)	x_1	°C	26	40	60	80	94
Drying time (t)	x_2	min	199	240	300	350	401
Air Mass flow rate (G)	x_3	kg/h	25.2	46.8	77.4	108.0	129.6

The statistical analysis of the experiments, aiming to find a representative model for the final contents of bixin and moisture, was performed using existent computer programs (Statistica®).

Analysis of Second-Order Models

The characterizations of the stationary point and of the response surfaces, described by quadratic polynomial models are performed in the following way: when the stationary point is in the vicinity of the optimal point of the experiment, a model of second order for a response $\hat{Y}$is proper.to a precise estimation of the initial conditions of process operation.

The levels of the points to optimize the predicted response $\hat{Y}$may be obtained from its partial derivative in relation to the independent variables x_1, x_2, ... x_k, as shown in the following equation:

$$\frac{\partial \hat{Y}}{\partial x_1} = \frac{\partial \hat{Y}}{\partial x_2} = ... = \frac{\partial \hat{Y}}{\partial x_k} = 0 \qquad (6)$$

The solution of the resultant K equations furnishes a point of coordinates x_{1s}, x_{2s}, ... x_{ks} named stationary point, which may represent a point of minimum or maximum response, and also a minimax or a saddle point (Montgomery, 1991). The methodology for the general solution and the location of the stationary point and of the correspondent value of response in this point, from a second-order polynomial model, is described by Khuri and Cornell (1987) and by Box and Draper (1987).

Once the coordinates of the stationary point are found, it is necessary to characterize the response surface in the immediate vicinity of this point, with the aim to certify if this region corresponds to a minimum, maximum or minimax (saddle point), and verify the relative sensitivity of the responses to the entrance variables. This may be performed through the transformation of the proposed model into a new system of coordinates with the origin in the stationary point, performing a rotation of axis of this system until they become coincident with the main axis of the response surface proposed, based on experimental data, resulting on an expression of the kind:

$$\hat{Y} = \hat{Y}s + \lambda_1 W_1^2 + \lambda_2 W_2^2 + \ldots + \lambda_k W_k^2 \qquad (7)$$

in which W_i (i = 1,2,...k) corresponds to transformed independent variables and λ_i (i = 1,2, ...k) are constants, known as eigenvalues or characteristic roots of the symmetric matrix (k x k) of the main quadratic coefficients and other elements by half of the values of other coefficients of the proposed model, according to Montgomery (1991).

Equation 7 corresponds to a canonical form of the model and represents the same function of the second-order polynomial model. The nature of the response surface may be determined from the stationary point and from the signal and magnitude of the eigenvalues λ_i. If all of them are negative, the surface corresponds to a maximum; on the other hand, if the eigenvalues in the stationary point are all positive, the surface is a minimum. If not every λ_i presents the same signal, the stationary point characterizes a point of saddle in the surface of response fitted to the model.

The necessary calculation to locate the stationary point, the prediction of the response in the stationary point and the determination of eigenvalues, λ_i, and its correspondent eigenvectors, may be done analytically or numerically, with the help of computer programs, such as Mathematica®.

The nature of the response surface described according to the methodology above is valid when the stationary point is located inside the experimental region, otherwise, it is risky to try to conclude about the nature of the response surface. The distance from the stationary point to the center of the experiment (centre point) can be estimated by Equation 8:

$$Ds = \left(\sum_{i=1}^{k} x_{is}^2 \right)^{1/2} \qquad (8)$$

If the coordinates of the stationary point are near the centre point, which results in values of Ds close to one, a canonical form of the type indicated by Equation 7 is proper for the comprehension of the response surface.

The determination of an optimal point, taking into account restrictions on the entrance variables, may be gotten through the concept of overall desirability Dj, according to what was described by Akhnazarova and Kafarov (1982).

The concept of overall desirability is useful and it is desired to optimize processes where more than one response variable is involved. The responses obtained are transformed and interpreted in terms of acceptability, in a non-dimensional scale which varies from 0 (undesirable) to 1 (completely acceptable), according to Akhnazarova and Kafarov (1982). In this criteria dj consists of the individual desirability for each analyzed response and Dj is calculated as the geometric mean of many values of dj. The numerical optimization may be obtained through available computer programs.

RESULTS AND DISCUSSION

In Table 3 are represented the original and coded variables, and their respective levels. It is also shown the values of the responses experimentally obtained for drying of annatto seeds. The experimental runs were planned according to a factorial design (runs 1 through 8), central composite design (runs 9 through 14) and replication of the center point (runs 15 through 20).

Table 3: Experimental conditions and results of responses X_f and B_f.

Runs	Variables in coded units			Variables in original units			Response	
	x_1	x_2	x_3	Tg (°C)	t (min)	G(kg/h)	X_f (%db)	B_f (%db)
01	-	-	-	40	240	46.8	15.84	3.00
02	+	-	-	80	240	46.8	11.28	2.79
03	-	+	-	40	360	46.8	17.83	2.32
04	+	+	-	80	360	46.0	7.70	2.56
05	-	-	+	40	240	108.0	13.10	2.56
06	+	-	+	80	240	108.0	9.22	3.00
07	-	+	+	40	360	108.0	12.78	2.50
08	+	+	+	80	360	108.0	6.51	2.76
09	-1.68	0	0	26	300	77.4	16.16	2.69
10	+1.68	0	0	94	300	77.4	6.75	2.67
11	0	-1.68	0	60	199	77.4	11.10	2.87
12	0	+1.68	0	60	401	77.4	10.07	2.54
13	0	0	-1.68	60	300	25.2	15.15	2.59
14	0	0	+1.68	60	300	129.6	10.76	2.50
15	0	0	0	60	300	77.4	11.35	2.30
16	0	0	0	60	300	77.4	11.27	2.90
17	0	0	0	60	300	77.4	11.36	2.74
18	0	0	0	60	300	77.4	10.65	2.75
19	0	0	0	60	300	77.4	11.90	2.65
20	0	0	0	60	300	77.4	11.29	2.84

For the determination of the statistical significance of the effects of the entrance variables in the final moisture content of the annatto seeds, X_f, a variance analysis was performed and the results are presented in Table 4. The effects of the variables may be evaluated based on the F-test and on the value of probability (Pr).

Table 4: Analysis of Variance for X_f.

Effects	Sum of Squares	Degrees of Freedom	Mean Square	Test F	Probability (Pr)
x_1: Tg	121.0891	1	121.0891	766.84	0.0000
x_2: t	2.95463	1	2.95463	18.71	0.0075
x_3: G	24.85268	1	24.85268	157.39	0.0001
x_1 x_2	7.92020	1	7.92020	50.16	0.0009
x_1 x_3	2.57645	1	2.57645	16.32	0.0099
x_2 x_3	0.25920	1	0.25920	1.64	0.2563
x_1 x_1	0.07847	1	0.07847	0.50	0.5195
x_2 x_2	0.78777	1	0.78777	4.99	0.0758
x_3 x_3	5.25951	1	5.25951	33.31	0.0022
Lack of fit	1.86878	5	0.37376	2.37	0.1831
Pure Error	0.78953	5	0.15791	-	-
Total (Corr.)	168.8503	19	-	-	-

$R^2 = 0.9843$

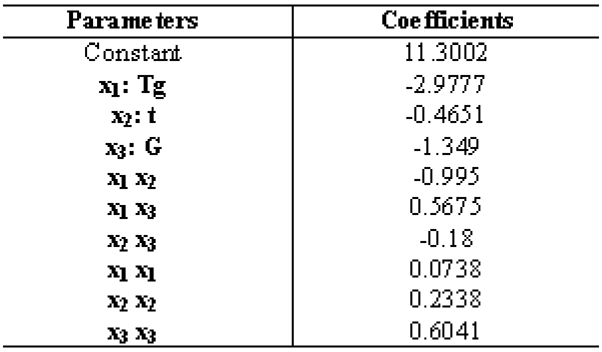
By the examination of Table 4, it can be verified that the inlet variables x_1 (Tg), x_3 (G) and the combination x_1 x_2 (Tgt) and x_3x_3 (G^2) are statistically significant, with a significance level inferior to 0.05; x_2 (t) and x_1x_3 (TgG) at a significance level inferior to 0.01 and the combination x_2x_2 (t^2) may affect the response at a significance level inferior to 0.10. The other combinations are not statistically significant to the response X_f.

The test of lack of fit, indicated in Table 4, consists of an estimation of the fail that the model may present to predict the response and the coefficient R^2 represents the proportion of variability around the average, which is explained or described by the equation of regression. Therefore, it is verified that for the second-order polynomial model proposed, there is no evidence of lack of fit, since the value of F calculated ($F_{5,5}$ = 2.37) is lower than the value of F tabled ($F_{5,5}$ = 5.05) at the significance level of 0.05.

The value of the coefficient of determination R^2 indicates that 98.43% of the variance is explained by the regression, in a maximum of 99.53% of explainable variation (Barros Neto, Scarminio and Bruns, 1995). Due to the high proportion of explained variability for the response X_f, the second-order model proposed is proper for the process description. The model may predict, with safety, the variations in the final moisture contents of the annatto seeds, undergoing a convective drying in fixed bed, as functions of the statistically significant variables.

The results of the analysis of multiple regression with the indication of the respective coefficients, for each entrance variable and their combinations are indicated in Table 5.

Table 5: Coefficients of regression for X_f.



Parameters	Coefficients
Constant	11.3002
x_1: Tg	-2.9777
x_2: t	-0.4651
x_3: G	-1.349
$x_1\ x_2$	-0.995
$x_1\ x_3$	0.5675
$x_2\ x_3$	-0.18
$x_1\ x_1$	0.0738
$x_2\ x_2$	0.2338
$x_3\ x_3$	0.6041

Based on the values obtained, a statistical second-order model is proposed as:

$$\hat{Y} = 11.3002 - 2.9777x_1 - 0.4651x_2 - 1.349x_3 - \quad (9)$$

$$- 0.995x_1x_2 + 0.5675x_1x_3 - 0.2338x_2^2 + 0.6041x_3^2$$

Equation 13 describes a model to estimate the final moisture content of annatto seeds, Red Piave cultivate, as a function of original variables Tg (°C), t (min) and G (kg/h), through the relationships given by the Equations 10, 11 and 12.

$$x_1 = \frac{Tg - 60}{20} \quad (10)$$

$$x_2 = \frac{t - 300}{60} \quad (11)$$

$$x_3 = \frac{G - 77.4}{30.6} \quad (12)$$

Taking into account these relations in Equation (9),we obtain:

$$X_f = 13.372 + 0.028\mathbf{Tg} + 0.081\mathbf{t} - 0.2\mathbf{G} -$$

$$-0.00083\mathbf{Tgt} + 0.00093\mathbf{TgG} - 0.000065\mathbf{t^2} + \quad (13)$$

$$+ 0.00065\mathbf{G^2}$$

The model shows that the air temperature, air flow rate and drying time exert a considerable influence on the reduction of the moisture content of the annatto seeds. The results and the statistical analysis indicate that a raise in the values of **Tg, t** and **G**, causes a sensible decrease in the value of X_f, improving drying.

For the quadratic equation of the model given by Equation 9, we can write:

$$\frac{\partial \hat{Y}}{\partial x_1} = -2.97767 - 0.995x_2 + 0.5675x_3 \tag{14}$$

$$\frac{\partial \hat{Y}}{\partial x_2} = 0.465131 - 0.995x_1 - 0.467602x_2 \tag{15}$$

$$\frac{\partial \hat{Y}}{\partial x_3} = -1.349 + 0.5675x_1 + 1.208228x_3 \tag{16}$$

Solving this system of equations, one can find that the coordinates of the stationary point x_{1s}, x_{2s} and x_{3s} and the correspondent value of $\hat{Y}_s$ through the substitution of these point in Equation 9. The coordinates found are:

$x_{1s} = 0.7317$; $x_{2s} = -2.5518$; $x_{3s} = 0.7728$; and

$\hat{Y}_s$ = 10. 2829.

Now, it is possible to determine the nature of the response surface in the immediate vicinity of this point through the canonical analysis previously described. Hence, the distance from the stationary point (x_{1s}, x_{2s}, x_{3s}) to the centre point is: Ds = 2.765.

The value obtained for Ds permits to transform the proposed model to a canonical form of the type presented in Equation 7, which gives a better comprehension of the nature of the surfaces under analysis.

Calculating the eigenvalues and eigenvectors correspondent to this system of equations we can write the canonical equation as:

$$\hat{Y} = 10.2829 + 0.7613W_1^2 - 0.6536W_2^2 + 0.2627W_3^2 \tag{17}$$

with the linear relationships of initial coordinates represented by the following Equations:

$$W_1 = 0.4708x_1 - 0.2354x_2 + 0.8502x_3 - 0.7356 \tag{18}$$

$$W_2 = 0.6382x_1 + 0.7563x_2 - 0.1440x_3 + 0.0463 \tag{19}$$

$$W_3 = 0.6091x_1 - 0.6104x_2 - 0.5063x_3 - 0.7297 \tag{20}$$

where W_1, W_2 and W_3 represent the independent variables in the canonical form.

It is verified by the signal and the magnitude of the eigenvalues (λ_1 and λ_3 positives and λ_2 negatives) that the stationary point corresponds to a minimax or saddle point. The canonical Equation (Equation 17) indicates that the value of the response predicted in the center of the experiment is 10.2829, that is, the moisture of seeds is 10.28%db.

The magnitudes (modes) of the eigenvalues λ_1, λ_2 and λ_3, which are the respective coefficients of W_1, W_2 and W_3 in the canonical equation, indicate that the height of the response surfaces predicted changes more rapidly along the axis W_1 than along the axis W_2, and that the response changes less in the direction of axis W_3, as it gets away from the stationary point. It demonstrates that the order of sensitivity related to the output variable due to modifications in the entrance variables associated to directions W_1, W_2, W_3 is $x_1 > x_2 > x_3$, or in original variables: Tg >t > G.

Figure 2 shows a comparison among the experimental and calculated values of X_f. It is verified a good fit of the calculated values to the experimental ones. The random distribution of residuals verified in Figure 3, indicates that the proposed model is satisfactory.

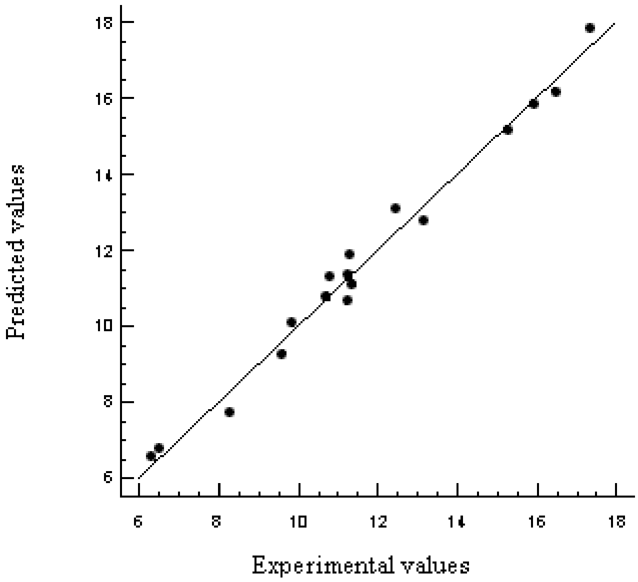


Figure 2: Comparison of the proposed model with the experimental results for X_f
(— model; •experimental)

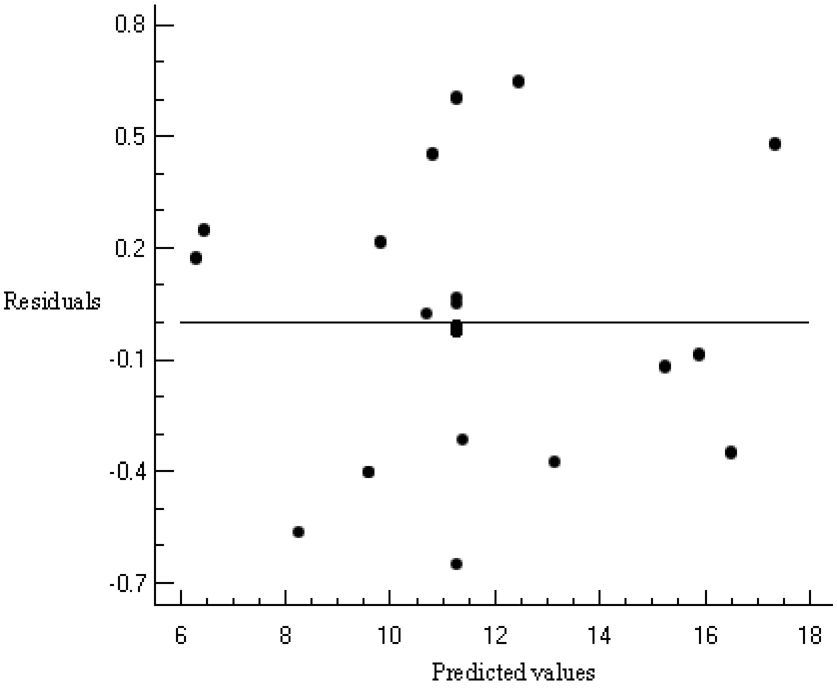


Figure 3: Distribution of residuals for X_f.

The same statistical analysis was performed for the final value of bixin content, B_f. The analysis of variance, presented in Table 6, shows that only the drying time presented a statistical significance for B_f, at the significance level of 0.10.

Table 6: Analysis of variance for B_f.

Effects	Sum of Squares	Degrees of Freedom	Mean Square	Test F	Probability (Pr)
x_1: Tg	0.0355075	1	0.0355075	0.79	0.4252
x_2: t	0.2281047	1	0.2281047	5.04	0.0747
x_3: G	0.0000001	1	0.0000001	0.00	0.9987
x_1 x_2	0.0091125	1	0.0091125	0.20	0.6769
x_1 x_3	0.0561125	1	0.0561125	1.24	0.3160
x_2 x_3	0.0465125	1	0.0465125	1.03	0.3571
x_1 x_1	0.0002132	1	0.0002132	0.00	0.9486
x_2 x_2	0.0023190	1	0.0023190	0.05	0.8321
x_3 x_3	0.0277523	1	0.0277523	0.61	0.4769
Lack of fit	0.1039402	5	0.0207880	0.46	0.7932
Pure error	0.2261333	5	0.0452267	-	-
Total (Corr.)	0.73805500	19	-	-	-

$R^2 = 0.5528$

It can also be verified in Table 6, that there is no evidence of lack of fit, as the value calculated for F is equal to 0.46, much smaller than the value tabled at the significance of 0.10, $F_{5,5} = 3.45$. For the response B_f, the coefficient of determination R^2 indicates that the model is able to explain 55.28% of the variability in a maximum explainable of 69.37%.

Using only the constant and the coefficient for the only significant variable, x_2 (t), given in Table 7, it is proposed a linear model to express the response B_f, given by Equation 22, in function of the coded variable:

Table 7: Coefficients of Regression for B_f.

Parameters	Coefficients
Constant	2.69518
x_1: Tg	0.05099
x_2: t	-0.12924
x_3: G	-9.97298×10^{-5}
x_1 x_2	0.03375
x_1 x_3	0.08375
x_2 x_3	0.07625
x_1 x_1	3.84641×10^{-3}
x_2 x_2	0.01269
x_3 x_3	-0.04388

$$\hat{Y} = 2.69518 - 0.12924x_2 \qquad (22)$$

The same model is represented in Equation 23, as a function of the original variable:

$$B_f = 3.35 - 0.0022\mathbf{t} \qquad (23)$$

This result indicates that the bixin content is practically independent of the drying air temperature and strongly dependent on the drying time. This result confirms the statements of the literature about the higher importance of undesirable effects caused by excess of exposition time of bixin to light and oxygen.

The comparison among the experimental and calculated values through the linear model for B_f is illustrated on Figure 4. The comparison shows the existence of a reasonable fit of the predicted values.

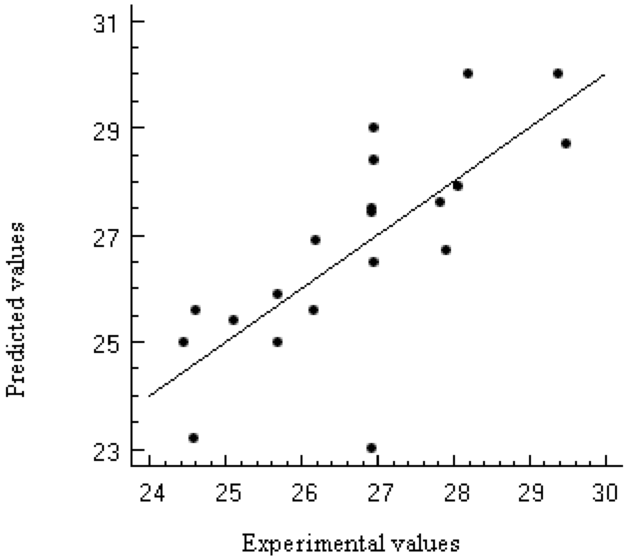


Figure 4: Comparison of the proposed model with the experimental results for B_f (— model; •experimental)

Figure 5 represents a randomized distribution of the residuals, showing the absence of a tendencial behavior, indicating that the mathematical model represents properly the variations of final concentration of bixin in function of the drying time, by a linear relationship.

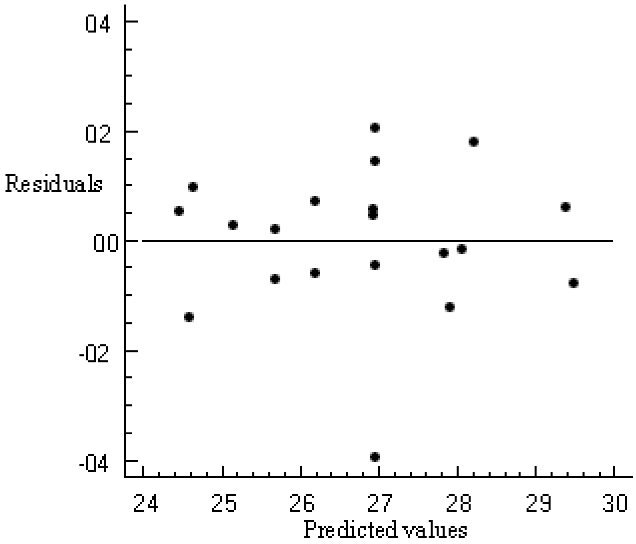


Figure 5: Distribution of the residuals to the response B_f

Optimization of the Drying Process

Using the response surfaces methodology discussed previously, it is possible to determine the operational conditions in function of the variables of interest for the process and the optimum value for the response variable considered. However, when it is desired to optimize more than one response variable, it is necessary to verify if the optimal conditions for a response does not make the process impracticable due to an undesirable response for the other output variable.

Hence, the identification of the optimal region for both X_f and Bf, the responses considered in this work, was performed through the analysis and the visualization of contour curves. These curves allow the identification of the range of the operational conditions of Tg, t and G which may correspond to the optimal for both responses.

Figures 6 and 7 show the contours for X_f and B_f, with the variables x_1 (Tg) and x_2 (t), for x_3= 0 (G = 77.4 kg/h). Keeping the drying air mass flow rate in the central point fixed, the shadow in the contour curves for X_f and B_f denotes the regions where the process may be properly conducted, aiming to obtain final moisture contents between 5 to 10%db and concentration of bixin higher than 2.86%db (degradation less than 10%). A superposition of the contours indicates that the ranges of temperature and drying time should be between 74 to 94 °C and 199 to 336 min, respectively.

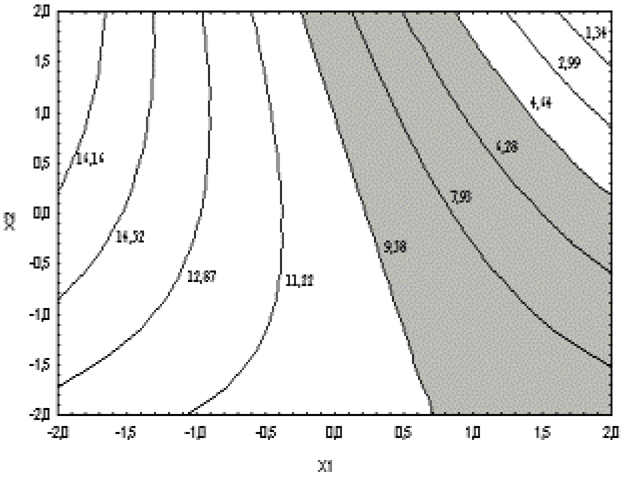


Figure 6: Contour plot for X_f in function of x_1 and x_2, with the shade indicating the optimal region.

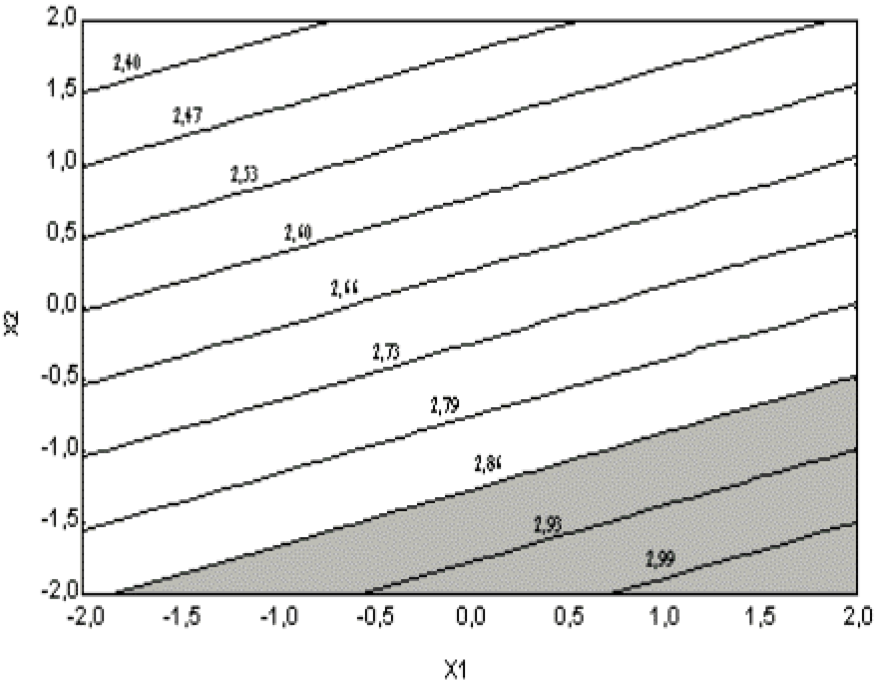


Figure 7: Contour plot for B_f in function of x_1 and x_2, with the shade indicating the optimal region.

The calculation of the function Dj was performed based on the described methodology by Akhnazarova and Kafarov (1982), with the following restrictions on the output variables X_f and B_f: 5%db < X_f < 10%db and B_f > 2.84% db.

The limiting value of 5% db for the final moisture content corresponds to the mean value of moisture content in the monolayer, in the range of 35 to 80°C (Faria, Rocha and Costa, 1998).The maximum value of 10%db was chosen aiming the material good quality and conservation. The minimum limit of 2.86%db for the final content of bixin was chosen, in order to maintain the stability of the coloring related to the initial content, at a maximum of 10% of degradation.

The optimization was performed numerically and the value 0.8732 was obtained for the function Dj. This value is considered acceptable and excellent according to the classification by Akhnazarova and Kafarov (1982). The optimal point obtained for the coded variables was x_1 = 1.68; x_2 = -1.68 and x_3 = -0.8918, which correspond to Tg = 94°C, t = 199 min and G = 50 kg/h, reaching the imposed restrictions shown in Table 8. With these values the optimal point for the input variables was established, obtaining X_f = 10%db and B_f = 2.98%db, which corresponds to a degradation of only 5.7% related to the initial content of bixin.

Table 8: Restrictions of the response for location of the optimal point.

Response Variables (%db)	Restriction	Objective
Final moisture (X_f)	X_f< 10%db	minimize
Final content of bixin (B_f)	B_f > 2.84 %db	maximize

CONCLUSIONS

Through the analysis of the contour curves of the responses X_f and B_f keeping the air mass flow rate constant at 77.4kg/h, it was possible to identify the optimal region of work for the fixed bed drying process of annatto seeds, considering the inlet variables: **Tg** (74 to 94°C) and **t** (199 to 336 min). The final moisture content was obtained in the interval of 5 to 10%db and final concentration of bixin was higher than 2.86%bs, values recommended in order to maintain the quality of the material.

The calculated value for the over-all desirability function was considered satisfactory and excellent, enabling the correct estimation of the optimal operation point for the input variables: Tg=94°C, t =199 min and G = 50 kg/h. The responses obtained at this point were X_f = 10%db and B_f=2.98%db.

The information provided by this work may be used to evaluate the efficiency of similar drying processes under the same range of operational conditions and, if necessary, to promote the changes in the process conditions towards the direction of optimization, aiming to predict the moisture content and the final concentration of bixin in the annatto seeds.

NOMECLATURE

B	bixin content, %db
Dj	overall desirability function, dimensionless
Dj	desirability function, dimensionless
Ds	distance of the estationary point from the design center
F	Fisher statistic (F-test), dimensionless
G	air mass flow rate, kg/s
i	order number

k	number of independent variables or factors
n_α	number of axial points
n_C	number of center points replications
n_f	number of factorial point of the project
N_t	total number of design points
Pr	probability, dimensionless
R	coefficient of correlation, dimensionless
R^2	coefficient of determination, dimensionless
t	time , min
Tg	gas temperature, °C
W_1, W_2,	transformed independent variables in
$W_{3...}W_k$	canonical forms
X	moisture content, %db
x_1, x_2, x_3	coded variables
x_{1S}, x_{2S}, x_{3S}	coordinate of the stationary point
$\hat{Y}$	fitted value of response variable
$\hat{Y}_S$	predicted variable at stationary point
α	axial space value
λ	eigenvalues

Subscripts

db	dry basis
f	final
i	initial
s	dry bulb
u	wet bulb
wb	wet basis
1	inlet
2	exit

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