CHARACTERIZATION OF THE FUJI APPLE DRYING IN THIN LAYER: DETERMINATION OF GLUCOSE CONTENT, COLOR AND PRODUCTION

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ABSTRACT
The aim of this work was to study the convective drying of Fuji apple (*Mallus percicae*) in thin layer and to evaluate the characteristics of the dried product. The air flow was perpendicular through the samples with air velocity of 1.5 m s^{-1}. The study factors were the air temperature (60, 70 and 80ºC), sample thickness (3, 4 and 5 mm) and citric acid solution concentration (0, 0.5 and 1 %). The glucose content and the samples color were found through spectrophotometer method. Five empirical models were used to evaluate the experimental data of the drying process. The fit of the drying data for the estimate of drying constant (K) showed high values of correlation for all models (R^2 > 0.99). The Henderson-Pabis model was chosen for determination of the moisture average effective diffusivity (D_{eff}). Through the surface response methodology, it was found that the samples with higher production and better physical-chemical characteristics was in conditions of air temperature at 70ºC, sample thickness of 3 mm and citric acid solution concentration of 1%.

Key-Words: Citric acid. Food drying. Glucose content. Surface response.

I. INTRODUCTION

Apple is an important raw material for many food products, and apple plantations are cultivated all over the world in many countries [1]. The Fuji apple (*Mallus percicae*) is original from Japan and was brought to Brazil in the end of 60´s, it is more adapted in cold climate that others cultivations, as for example the Gala and the Golden Delicious, having better production in the coldest regions in the south of the country. Due to its high moisture content, approximately 85%, the apple is considered a highly perishable food, being very important to define its conditions of preservation and the parameters for its storage and reutilization [2]. The drying is a method widely used in the preservation of fruits and vegetables, therefore it reduces

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the activity of water, thus it decreases the risk of microbial development. Moreover, the dry fruits can be stored and be carried with a relatively low cost [3,4].

The thin layer drying is the process where the material to be dry is entirely exposed to the air that moves through it. Currently, three types of models exist to describe the thin layer drying phenomenon of agricultural products, being, the theoretical model, that considers the internal resistance of moisture content transfer, the half-theoretical and empirical models, who are based on the external conditions, as moisture content, temperature and air velocity of drying [5].

The drying operation must be improvement to lead at products with high quality, for this, the controlled conditions that characterize the operation must be well established in order to preserve the original characteristics of apples [2]. The color is a property that lead the initial acceptability of all the foods, as well as the flavor of the dry product [6]. The main groups of reactions that lead to the darkness are the enzymatic oxidation of phenols and the not enzymatic darkness. The last one is favored by the heat treatments and includes an variety of reactions, such as Maillard reaction, caramelization and chemical oxidation of phenols [7].

The aim of this work was to characterize the drying of Fuji apple (*Mallus percicae*) in thin layer through empirical models of literature. The best conditions of the operation was evaluated by the response surface methodology, being found the characteristics of the final product, the glucose content and color.

II. MATERIAL AND METHODS

The raw material was Fuji apple (*Mallus percicae*). In the drying experiments a tray drier was used, with perpendicular air flow across the material bed, as it shown in FIGURE 1.

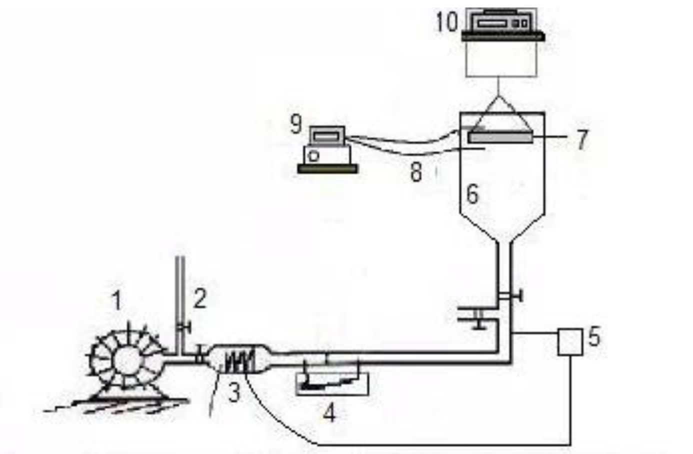

Figure 1 - Scheme of the tray drier with perpendicular air flow.

Experimental Methodology

The apples were selected, peeled, and had its core removed. After they had been sliced in different thicknesses, immersed in citric acid solution (when necessary), and disposed in stainless steel tray perforated. The sample load in tray was of 6 kg m^{-2}. The drying temperature was measured with constantan-copper thermocouples, (Contemp, model CSC99, Brazil), connected to a millivoltimeter (Technology, model N1400 Novus, Brazil) with 1ºC precision. The air velocity was measured with an anemometer with 0.1 m s^{-1} precision (Smart Sensor, model AR816, TFA, Germany). The wet samples were put in the drier and its weight measures were found in a digital scale (Marte, model AS2000C, Brazil), with 0.01 g precision, in each 5 min, until the product achieves the commercial moisture content of 8 to 10% (wet basis, wet basis). The relative humidities of the ambient and drying air were determined with a thermohigrometer (Cole Parmer, model 3310-00, USA) with 0.1 % precision. The samples thickness of the were measured with a digital caliper (Guo gen, Digital Caliper, China). All the experiments were carried out with two repetitions, with air velocity of 1.5 m s^{-1} and absolute humidity of 0.010 ± 0.001 $kg_w\,kg_{ds}^{-1}$.

Drying Characterization

The drying experiments was studied by the curves of drying rate (N) versus moisture content (X) and dimensionless free moisture content $[(X-X_E)/(X_0-X_E)]$ versus drying time. Through the curves of dimensionless free moisture content were found the drying constant (K) values from fit of experimental data by the empirical models: Lewis (Equation 1), Henderson and Pabis (Equation 2), Henderson and Henderson Equation 3), Overhults (Equation 4) and Page (Equation 5).

$$Y = \exp(-Kt) \tag{1}$$

$$Y = A\exp(-Kt) \tag{2}$$

$$Y = A.\ \exp(-Kt) + \frac{1}{9}\exp(-9Kt) \tag{3}$$

$$Y = \exp[(-Kt)]^n \tag{4}$$

$$Y = \exp[(-Kt^n)] \tag{5}$$

where Y is the dimensionless of free moisture content of the material, K is the drying constant (min^{-1}), t is the drying time (min), A and n are the adjustment parameters (dimensionless).

The estimate of the moisture effective diffusivity (D_{eff}) values were found by analogy of the empirical solution of Henderson and Pabis model (Equation 2) with the solution of the Fick diffusion equation, that consider the material in the form of infinite slab without internal resistance of mass transfer ($v_{air} > 1.5$ m s^{-1}) and drying for both sides, according to Equation 6 [8].

$$D_{eff} = \frac{K L_m^2}{\pi^2} \tag{6}$$

where L_m is the thickness of the sample.

Statistic methodology

The effects of the slice thickness (3, 4 and 5 mm), the drying air temperature (60, 70 and 80°C) and the citric acid concentration (0, 0.5 and 1%) in drying process were analyzed. A complete factorial experimental design was used (2^3), with central points, to define the significance of the study factors, through the variance analysis [9]. After, the response surface methodology was used in order to found the best drying conditions, for the response considered in the statistic analysis (glucose content, color and production) [10]. TABLE 1 presents the matrix of the experimental design, with the study factors in the codified form and real values.

Table 1: Matrix of the experimental design on the apple drying.

Experiment	Study factors					
(Nº)	Temperature (°C)	X_1*	Acid (%)	X_2*	Thickness (mm)	X_3*
1	60	-1	0	-1	3	-1
2	80	+1	0	-1	3	-1
3	60	-1	1.0	+1	3	-1
4	80	+1	1.0	+1	3	-1
5	60	-1	0	-1	5	+1
6	80	+1	0	-1	5	+1
7	60	-1	1.0	+1	5	+1
8	80	+1	1.0	+1	5	+1
9	70	0	0.5	0	4	0
10	70	0	0.5	0	4	0
11	70	0	0.5	0	4	0

*****Codified form

Analytic methodology

The moisture content of the apple samples *in natura* carried out by gravimetrical analysis in vacuum stove, 500mm Hg, 70°C [11]. The sugar content of *in natura* and dehydrated apple samples were determined by sulfuric phenol method in spectrophotometer (Quimis, model Q-108 DRM, Brazil) [11]. The color determinations of dehydrated and *in natura* apples samples were through spectrophotometer method (Quimis, model Q-108 DRM, Brazil), in which the samples were dissolved in water, getting itself an apple extract. A concentration curve of caramel food dye was plotted in function of the absorbance read in spectrophotometer at 620 nm. From a linear regression, a standard curve was found, and the color in the samples will be determined.

III. RESULTS AND DISCUSSION

Drying characterization

FIGURE 2 shows the curves of the drying rate versus moisture content.

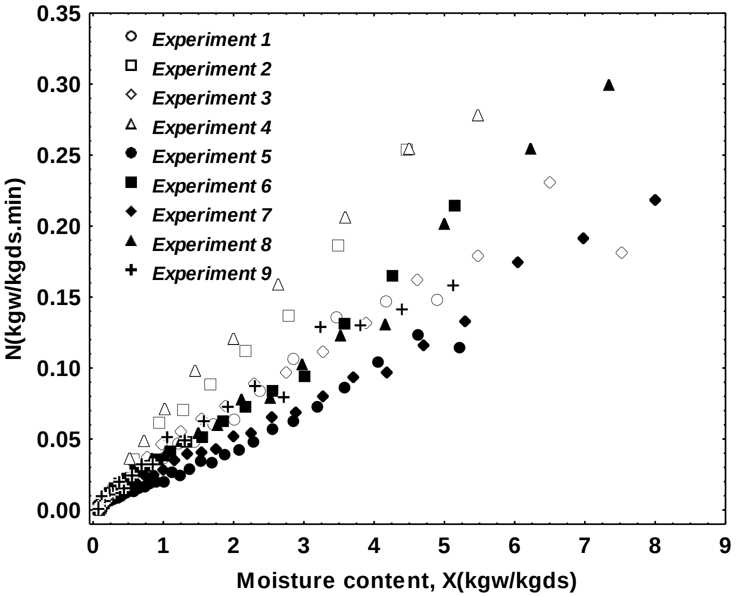

Figure 2 - Curves of the drying rate in function of the moisture content for the apple drying of the experiments from 1 to 9 of the matrix of the experimental design,.

In FIGURE 2 was not observed the period of the drying constant rate. Chirife [8] reported the critical moisture content value (X_c) for apple of 6.0 $kg_w\ kg_{ds}^{-1}$ (dry basis) to similar drying conditions. In addition, the drying of material occurred only in the falling rate period, this suggest that the mass transfer is found through the intrinsic properties of the product and by the resistance to water diffusion [12].

FIGURE 2 shows that the use of a higher air temperature resulted in higher drying rate, due to the increase of the thermal transfer between the air and the apple slices [13]. The drying rates decrease with the increase of the time occur because the free water, in higher volume at the beginning of the operation, lead to higher values of the diffusion coefficient in relation to diffusion of the enclosed water in the cells tissues [14]. The not existence of the constant rate period and the temperature influence on the drying rate also were observed by Mandala et al.[4], Togrul[1] and Kaya et al [15]. in the apple drying and Simal et al.[16] in the kiwi drying.

The equilibrium moisture content (X_E) of the samples apple, for temperatures at 60 and 70ºC was calculated through equilibrium isotherms found by Moraes et al.[17]. For the temperature of 80ºC it was carried out through drying experiments until reaching the constant mass of the sample. The values of the equilibrium moisture content at temperatures of 60, 70 and 80°C were 0.008, 0.006 and 0.003 $kg_w\ kg_{wb}^{-1}$ (in dry basis), respectively. FIGURE 3 shows curves of the dimensionless free moisture content versus drying time.

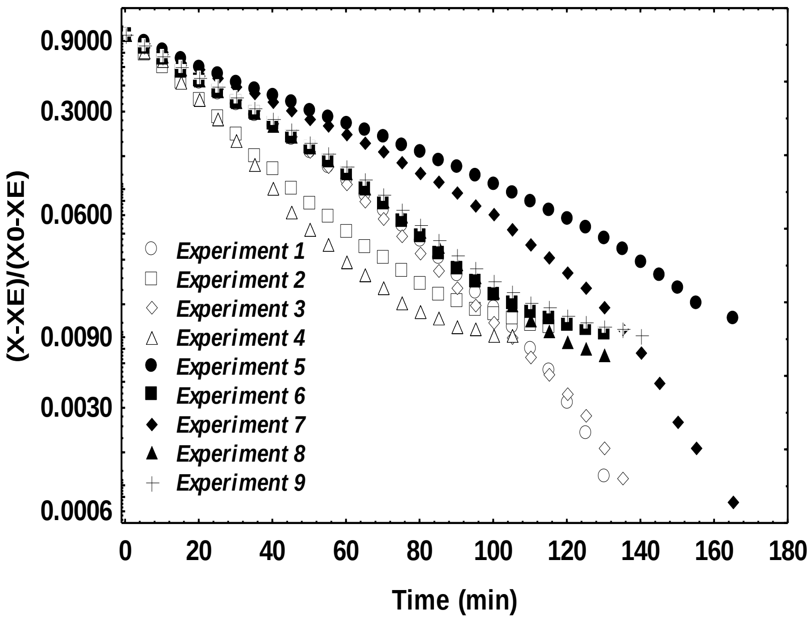

Figure 3 - Curves of the dimensionless free moisture content, in log-linear scale, function of the apple drying time, for the experiments from 1 to 9 of the matrix of the experimental design.

In FIGURE 3 can be observed two distinct regions. The first period of falling rate, where occurs an higher variation of the sample moisture content in function of the time. The second period of falling rate begin after the transition moisture content (X_{trans}), and the variation of moisture content becomes lower. The values found for the transition moisture content were on the range from 0.0092 to 0.0137 $kg_w\ kg_{wb}^{-1}$, and they are next to the values of commercial moisture content of apple (0.008 to 0.010 $kg_w\ kg_{wb}^{-1}$) showing that drying operation occurred practically in the first period of falling rate. Velic et al. [2] also verified the highest importance of the first period of falling rate in the apple drying, due to the short time of the second period of falling rate. This figure can also show, that with the increase of the air temperature of drying, for same samples thickness and concentrations of citric acid, the drying time decreased, according to literature for apple drying [1,6,13,15,18], kiwi drying [16] and apricots drying [19].

The drying constant (K) values were found by non linear regression of the experimental data in FIGURE 3, for first period of falling rate, by models (Equations 1-5). TABLE 2 shows the coefficient of determination (R^2) and the drying constant values for all the used models.

In all cases in TABLE 2, R^2 values were higher than 0.99 which indicates that all models showed good fit to the experimental data. However, it was chosen Henderson and Pabis model (Equation 2) by physical significance in its interpretation, because, it can be seen as a simplification of the analytical solution for the diffusive model on larger time of drying operation, where only the first term of the series is taken into consideration [20].

TABLE 3 shows the total times of drying and moisture average effective diffusivity (D_{eff}) calculated by the drying constant (K) estimated by the Henderson and Pabis model. The values of average thickness of apple slices were used, due the shrinkage of the material during the operation.

Table 2: Values of drying constant (K) and coefficient of determination (R^2) for the empirical models on the apple drying.

	Lewis		Henderson and Pabis		Henderson and Henderson		Overhults		Page	
Experiment (n°)	K (min^{-1})	(R^2)	K (min^{-1})	(R^2)	K (min^{-1})	(R^2)	K (min^{-1})	(R^2)	K (min^{-1})	(R^2)
1	0.0364	0.999	0.0372	0.999	0.0350	0.997	0.0286	0.999	0.0359	0.999
2	0.0510	0.999	0.0508	0.999	0.0475	0.998	0.0510	0.999	0.0510	0.999
3	0.0350	0.997	0.0364	0.998	0.0343	0.995	0.0198	0.999	0.0339	0.999
4	0.0522	0.996	0.0537	0.996	0.0502	0.993	0.0306	0.998	0.0506	0.998
5	0.0236	0.999	0.0234	0.999	0.0222	0.999	0.0253	0.999	0.0237	0.999
6	0.0361	0.999	0.0357	0.999	0.0337	0.998	0.0362	0.999	0.0362	0.999
7	0.0268	0.999	0.0268	0.999	0.0253	0.998	0.0254	0.999	0.0267	0.999
8	0.0353	0.998	0.0363	0.999	0.0341	0.997	0.0280	0.999	0.0349	0.999
9	0.0344	0.999	0.0348	0.999	0.0328	0.997	0.0279	0.999	0.0279	0.999
10	0.0382	0.996	0.0363	0.997	0.0343	0.998	0.0539	0.998	0.0394	0.998
11	0.0394	0.996	0.0377	0.997	0.0356	0.998	0.0511	0.997	0.0403	0.997

Table 3: Values of total time of drying and moisture average effective diffusivity on the apple drying.

Experiment (n°)	Drying time* (min)	D_{eff}* ($m^2\ s^{-1}$)×10^{10}
1	130 ± 5	2.50 ± 0.13
2	115 ± 5	5.38 ± 0.27
3	105 ± 5	2.45 ± 0.12
4	135 ± 5	5.67 ± 0.28
5	185 ± 5	3.32 ± 0.18
6	130 ± 5	6.56 ±0.33
7	165 ± 5	3.79 ± 0.19
8	130 ± 5	6.68 ± 0.34
9	145 ± 5	4.27 ± 0.21
10	145 ± 5	4.47 ± 0.22
11	145 ± 5	4.63 ± 0.24

* mean values ± standard error (in replicate)

Statistic analysis

Equations 7 and 8 are shown the fits of the standard curves of glucose (R^2 > 0.99) and caramel food dye (R^2 > 0.99), respectively.

$$y = 0.02142C_A - 0.159 \quad (7)$$

$$y = 0.03864C_C - 0.00186 \quad (8)$$

where C_A is the sugar concentration, in $mg_{glucose}\ mL^{-1}$ and C_C is the caramel dye concentration, in $mg\ g^{-1}_{apple}$.

The sugar content and the color determination of the dehydrated samples were determined through Equations 7 and 8, respectively. The response production capacity was obtained by apple solids loads in the tray for each analysis of drying, for the total times of drying showed in TABLE 3.

TABLE 4 shows the results of the matrix of experimental design to the responses glucose content (Y_1), color (Y_2) and production (Y_3) to the apple drying.

Table 4: Results of experimental design matrix to the responses glucose content, color and production on the apple drying.

Experiment (n°)	C_A* ($mg_{glucose}\ mL^{-1}$)	C_C* ($mg_{caramel.}\ g^{-1}_{apple}$)	Production* ($kg\ m^{-2}\ h^{-1}$)
1	25.75 ± 0.06	16.32 ± 0.02	2.77 ± 0.11
2	24.43 ± 0.04	15.07 ± 0.02	3.13 ± 0.13
3	27.92 ± 0.07	4.40 ± 0.09	2.67 ± 0.10
4	22.64 ± 0.04	4.26 ± 0.05	3.43 ± 0.14
5	23.07 ± 0.05	13.94 ± 0.06	1.94 ± 0.08
6	24.37 ± 0.09	9.06 ± 0.08	2.77 ± 0.11
7	23.80 ± 0.04	4.11 ± 0.04	2.18 ± 0.09
8	19.58 ± 0.07	8.36 ± 0.09	2.77 ± 0.11
9	24.56 ± 0.08	12.87 ± 0.05	2.48 ± 0.10
10	24.64 ± 0.07	12.79 ± 0.04	2.48 ± 0.10
11	24.69 ± 0.07	12.84 ± 0.04	2.48 ± 0.10

* mean values ± standard error (in replicate)

The results of the statistic analysis, applied to the experimental data, showed in TABLE 4, were determined through the pure error for significance level of 95% ($p < 0.05$). The estimated effect of the drying air temperature (X_1), sample thickness (X_3) and citric acid concentration (X_2) are presented in TABLE 5, for the responses glucose content (Y_1), color (Y_2) and production (Y_3).

TABLE 5 shows that all the study factors were significant for the response glucose content and color ($p \leq 0.05$). It is verified that all the main parameters had showed negative effect, so an increase in these factors occur a reduction the glucose content as in the color of the samples. The drying temperature (X_1) and citric acid (X_2) presented the higher effects on the glucose content and color, respectively, these are the factors that influence more this responses on the apple drying in thin layer. Sacilik et al. [13] also found that adding lemon solution in the apple slices presented a lower darkness, when compared to the samples without pre-treatment. Simal et al. [16] were found an important influence of the drying air temperature on the final color of dehydrated kiwi.

The response production (TABLE 5), were significant the factors drying temperature (X_1) and thickness (X_3), the temperature presented negative effect and the thickness presented positive effect on the production.

In TABLE 6 it is observed that the models for the responses glucose content and production were predictive, for presenting values of $F_{calculate}$ four times higher than the value of the F_{table} [22]. The model for the response color was not predictive, not being possible to generate the response surface.

The Equations 9 and 10 represents the theoretical-statistic models found by the regression analyses for the glucose content (Y_1) and production (Y_3) responses, with the main effect and its interactions in the codified form. The values of the coefficients of determination indicate that the models had explained 0.96 and 0.89, respectively, of variation of the experimental data.

$$Y_1 = 24.16 - (1.27\,X_1) - (0.98\,X_3) - (0.51\,X_2) + (0.48\,X_1X_3) - \\ - (1.22\,X_1X_2) - (0.67\,X_3X_2) \qquad (9)$$

$$Y_3 = 2.54 + (0.31\,X_1) - (0.28\,X_3) \qquad (10)$$

Table 5: Variance analysis (ANOVA) of the study factors to the glucose content, color and production responses on the drying apple

Effect	Value	Pure error	t- Student	Significance (p)
		Glucose content (R^2 = 0.96)		
Average	24.16	0.04	514.76	≤0.0001
X_1	-2.53	0.11	-23.01	0.0019
X_2	-1.02	0.11	-9.24	0.0115
X_3	-1.96	0.11	-17.78	0.0031
$(X_1)(X_2)$	-2.43	0.11	-22.09	0.0020
$(X_1)(X_3)$	0.97	0.11	8.79	0.0127
$(X_3)(X_2)$	-1.35	0.11	-12.24	0.0066
		Color (R^2 = 0.84)		
Average	10.36	0.01	850.64	≤0.0001
X_1	-0.50	0.03	-17.67	0.0032
X_2	-8.31	0.03	-290.96	≤0.0001
X_3	-1.14	0.03	-40.06	0.0006
$(X_1)(X_2)$	2.56	0.03	89.58	0.0002
$(X_1)(X_3)$	0.19	0.03	6.65	0.0219
$(X_3)(X_2)$	3.05	0.03	106.73	≤0.0001
		Production (R^2 = 0.91)		
Average	2.66	0.03	80.50	0.0002
X_1	0.63	0.08	8.19	0.014
X_2	0.11	0.08	1.42	0.2919
X_3	-0.58	0.08	-7.54	0.017
$(X_1)(X_3)$	0.07	0.08	0.97	0.4356
$(X_1)(X_2)$	0.04	0.08	0.51	0.6574
$(X_1)(X_3)$	0.07	0.08	0.97	0.4356
$(X_3)(X_2)$	0.01	0.08	0.13	0.9092

Table 6: Analysis of variance (ANOVA) of the theoretical-statistic models to the glucose content, color and production responses on the drying apple.

Sources of variation	SQ	GL	MQ	$F_{calculate}$	F_{table}
			Glucose content		
Regression	39.86	6	6.64	18.34	4.53
Residue	1.44	4	0.36		
Lack of adjustment	1.39	2	0.69		
Pure error	0.05	2	0.02		
Total	41.3	10			$R^2 = 0.96$
			Color		
Regression	173.20	6	28.87	3.48	4.53
Residue	33.16	4	8.29		
Lack of adjustment	33.16	2	16.58		
Pure error	0.003	2	0.001		
Total	206.36	10			$R^2 = 0.84$
			Production		
Regression	1.37	2	0.69	34.5	4.35
Residue	0.19	8	0.02		
Lack of adjustment	0.18	6	0.03		
Pure error	0.01	2	0.005		
Total	1.56	10			$R^2 = 0.89$

FIGURES 4 and 5 present the responses surfaces for the glucose content and the production, respectively, for apple drying by Equations 9 and 10.

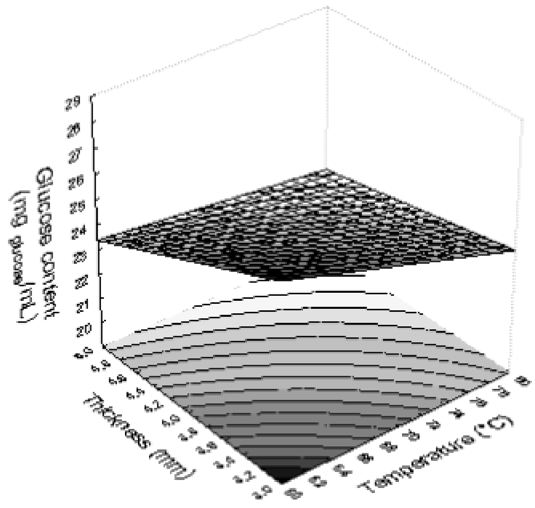

Figure 4 – Response surface for the glucose content of the dried apple.

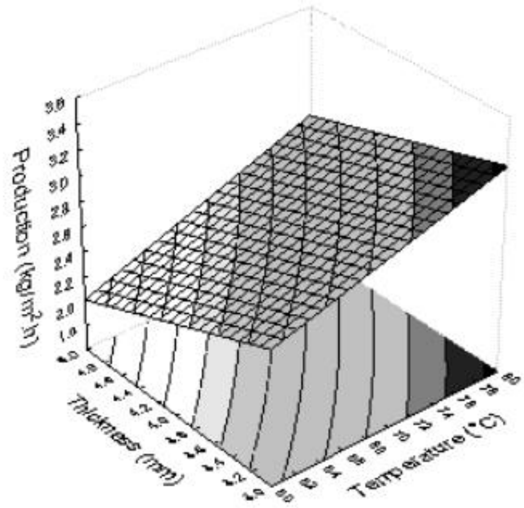

Figure 5 –Response surface for dried apple production.

FIGURE 4 shows that the glucose content of the sample decrease with the increase of the drying temperature and with the increase of the sample thickness, due the caramelization of the glucose that transforms this sugar into the products of the Maillard reaction. FIGURE 5 shows that for to obtain a higher production , it must be used higher drying temperatures and lower thicknesses. It occurs due to the production to be a relation of the mass for area and time, thus, in higher temperatures and lower thicknesses, low will be the drying time, being higher the production.

The factors that involve the conservation of the raw material characteristics during the drying process are associated the production. These factors have great importance in the drying, because they are related with the cost-benefit of the operation. Therefore, the best condition for the obtained for the dehydrated apple will be in a condition of high values of glucose content and production, and low values of color, being this in the region of air temperature at 70°C, apple slice thickness of 3 mm and concentration citric acid solution of 1%.

IV. CONCLUSION

The apple drying with perpendicular air flow occurred only in the falling rate period. All the empirical models presented high determination of coefficients ($R^2 > 0.99$), being the correlation of Henderson and Pabis the chosen one to the determination of the constant of drying. The effective diffusivity (D_{eff}) values were on the range from 2.45×10^{-10} to 6.68×10^{-10} $m^2\,s^{-1}$.

All the study factors in apple drying were significant ($p \leq 0.05$) in the responses glucose content and color by statistic analysis. To the response production only the factor citric acid concentration was not significant. The best conditions, through surface response methodology, for the obtain dried product with good characteristic, with higher value of glucose content, lower value of color and higher production were air temperature at 70°C, apple slice thickness of 3mm and citric acid solution concentration of 1%.

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