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Optimization of osmotic dehydration of orange pieces (*valencia late*) in sugar solution using response surface methodology

A. Khatir [1*], H. Acheheb [1], A. Malek [2†] and A. Ferradji [1]

[1] Département de Technologie Alimentaire, Ecole Nationale Supérieure d'Agronomie
Avenue Hassen Badi, El Harrach, Algiers, Algeria
[2] Division Energie Solaire Photovoltaïque, Centre de Développement
des Energies Renouvelables, CDER, B.P. 62, Route de l'Observatoire, 16340, Algiers, Algeria

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Abstract - *Response surface methodology was used to determine the optimum processing conditions that yield maximum water loss and weight reduction and minimum solid gain during osmotic dehydration orange pieces (valencia late) in sugar solution using response surface. The experiments were conducted according to a Central Composite Design 'CCD'. The independent process variables for osmotic dehydration process were temperature (40, 50, 60 °C), processing time (60, 120 et 240 min) and sugar concentrations (45, 55, 65 % w/w). The optimal conditions for maximum water loss, weight reduction and solid gain (candying) correspond to temperature of 50 °C, processing time of 240 min, sugar concentration of 65 % in order to obtain water loss 60.83 % (g/100 g fresh sample), solid gain of 46,48 % (g/100 g fresh sample) and weight reduction of 57.42 (g/100 g fresh sample). However the optimal conditions for maximum water loss and weight reduction and minimum solid gain (respectively 53.28 %, 57.88 % and 27.18 %) correspond to temperature of 40 °C, processing time of 240 min, sugar concentration of 65 %.*

Résumé - *La méthodologie de réponse de surface a été utilisée pour la détermination des conditions optimales permettant le maximum de perte en eau et de réduction du poids et un minimum de gain en solide au cours de la déshydratation osmotique des tranches d'oranges, dans une solution concentrée de saccharose. Les expériences ont été réalisées en utilisant le Central Composite Design 'CCD'. Les variables indépendantes de la déshydratation osmotique sont la température (40, 50 et 60 °C), la durée du traitement (60, 120 et 240 mn), la concentration de la solution de saccharose (45, 55 et 65 % w/w). Le procédé de déshydratation osmotique a été optimisé pour la perte d'eau, la réduction de poids et le gain en soluté. Les conditions optimales de la déshydratation osmotique des oranges (confisage) sont obtenues à: une température de 50 °C, une concentration de 65 % et une durée de 240 mn. Dans ces conditions, le pourcentage de la perte en eau et en poids et le pourcentage du gain en solide sont respectivement de 60.83 (g/100 g poids initial), 57.42 (g/100 g poids initial) et 46.48 (g/100 g poids initial). Cependant, les conditions optimales pour une perte en eau et une réduction en poids maximales, avec un gain en solide minimal, respectivement de 53,28 %, 57,88 % et 27,18 %, sont: une température de 40 °C, une durée de 240 mn et une concentration de sucre de 65 %.*

Mots clés: Optimisation - Déshydratation osmotique – Orange - Solution de sucre - Méthodologie de surface de réponse.

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[*] khatiramel@gmail.com , acheheb.hakima@hotmail.fr
[†] a.malek@cder.dz , a_ferradji@yahoo.fr

1. INTRODUCTION

In Algeria the annual production of oranges is estimated at 450,350 tons. Approximately 95% of this amount is consumed in fresh state and only 3% are used by food industries for the production of juices and jams. The pieces and peel of oranges are traditionally preserved using the candying and the osmotic dehydration by dipping in solution of sugar concentrated.

The candying facilitates the penetration of solids into fruits and limit the loss in water, whereas the osmotic dehydration facilitates the loss in water and limits the gain of solids.

Saurel *et al.* (1994) are proposed to refer to these two processes as ‘dewatering and impregnation soaking processes. The osmotic dehydration method is widely used for the elimination of the water of the fruit, to obtain a product of intermediate humidity, or as a pretreatment before an additional process such as freezing, freeze-drying, vacuum drying or air drying {Maltini *et al.* (1993); Beristain *et al.* (1990); Parjoko *et al.* (1996); Islam *et al.* (1982); Torreggiani *et al.* (2004)}.

Mass transfer during osmosis depends on operating variables such as concentration and solute type of the dehydration solution, temperature and period of process.

During the osmotic dehydration the water and little amounts of natural solutes (vitamin C) are transferred from fruit to the solution and the solute is transferred from the osmotic solution to the fruit in a countercurrent manner {Park *et al.* (2002)}.

Solute transference to the fruit causes an increase of soluble solids content of the fruit which decreases the phenolase activity and hence reducing the susceptibility of the fruit to the enzymatic browning during air dehydration and limits the decrease of vitamin C {McBean *et al.* (1971); Ponting *et al.* (1966); Vial *et al.* (1990)}. There are a very few studies on osmotic dehydration of oranges {Chafer *et al.* (2008)}.

The present study aims at modeling the influence of the temperature, processing time, sugar concentration, changes in mass of the oranges and to determining the optimal conditions of temperature, processing time, for the water loss and solute gain in oranges during osmotic dehydration using response surface methodology.

2. MATERIALS AND METHODS

2.1 Materials

Fresh oranges (*valencia late*) are purchased locally, which thoroughly washed with water. Sugar, the osmotic agent, was purchased from a local supermarket. The osmotic solution is prepared by mixing the sugar with proper amount of pure water.

2.2 Experimental procedure

Experiments were carried out with a product/solution ratio (1:5) such that the concentration of the solution remained approximately constant during the experiments. The oranges were peeled and the pieces were separated. One piece of orange was used to determine the initial water content.

The samples were individually weighed and immersed in a thermostatic bath at a selected temperature and osmotic solution concentration. The agitation was necessary to improve mass transfer, maintain uniform concentration, temperature profile and prevent the formation of a dilute solution film around the samples.

For each experiment an agitation speed of 100 rpm was used and maintained constant (Fig. 1).

At pre-selected sampling times samples were removed from the bath, carefully blotted with absorbent paper, individually weighed again and analyzed for moisture content using a infra rouge humidimeter (Fig. 2).

Fig. 1: Thermostatic bath and agitator

Fig. 2: Infrared moisture –meter

In order to follow adequately the osmotic dehydration kinetics, an individual analysis for each sample was carried out and weight reduction (WR), water loss (WL) and solid gain (SG) data were obtained, according to the expressions proposed by Azuara *et al.,* (1998).

$$WR = \frac{M_0 - M}{M_0} \times 100 \tag{1}$$

$$WL = \frac{(M_0 \times X_{W0}) - (M \times X_W)}{M_0} \times 100 \tag{2}$$

$$SG = \frac{(M_0 \times X_S) - (M \times X_S)}{M_0} \times 100 \tag{3}$$

M_0 : Initial mass of sample, (g) ; M : Mass of sample after dehydration, (g) ; X_{W0} : Initial mass of water ; X_W : Mass of water after osmotic dehydration at time (t) ; X_{S0} : Initial mass of solids ; X_S : Mass of solids after osmotic dehydration at time (t).

2.3 Experimental design and statistical analysis

Response surface methodology (RSM) was used to estimate the main effects of osmotic dehydration process on water loss (WL) and solid gain (SG) in orange slices. A miscellaneous design was used with independent proves variables viz. temperature (40 – 50 - 60°C), processing time (60 -120 – 240 minutes), sugar concentrations (45 – 55 – 65 % w/w) **(Table 1)**. For the generated 32 experiments, RSM was applied to the experimental data using Design Expert ® v.8.0.7.1.

3. RESULTS AND DISCUSSION

Table 2 shows that in order to obtain high levels of water loss, osmotic dehydration should be conducted at elevated temperatures and long times, but the increase in solid gain is inevitable in this case.

The increase of solids gain with increasing dehydration temperature favored obviously the process of candying.

Table 1: The levels of different process variables in coded and un-coded form for the osmotic dehydration of oranges independent variable range and levels

	Range and levels		
Independent variable	-1	0	1
Temperature (A , °C)	40	50	60
Sugar concentration (B %, w/w)	45	55	65
Processing time (C , min)	60	120	240

The increase in solid gain decreases the water activity of the product considerably .If the aim is to minimize the solid gain, lower temperatures and concentrations should be recommended. However, in this case, very long processing times are required to reach the desired amount of water removal.

At short processing times, increasing temperature raises water loss more than solid gain which causes an increase in weight reduction. This phenomenon is attributed to the diffusional differences between water and solutes as related to their molar masses {Lazarides *et al.* (1995), Raoult-Wack *et al.* (1991) and Torregiani, (1993)}.

However, towards equilibrium end point, water loss was not affected significantly by the temperature, whereas solid gain continued to increase. The effect of temperature was observed at high sucrose concentrations.

Chenlo *et al.*, (2002) and Moreira *et al.*, (2003) explained this effect as increasing temperature gives better water transfer characteristics on the product surface due to lower viscosity of the osmotic medium.

Multiple linear regression analysis of the experimental data yielded second order polynomial models for predicting WL, WR, SG, as assumed at the beginning of the study. **Table 3** shows the analysis of variance for fitting the second order polynomial models to experimental data. The statistical significance of all main effects, linear, quadratic, and interaction of effects calculated for each response can also be shown in **Table 3**.

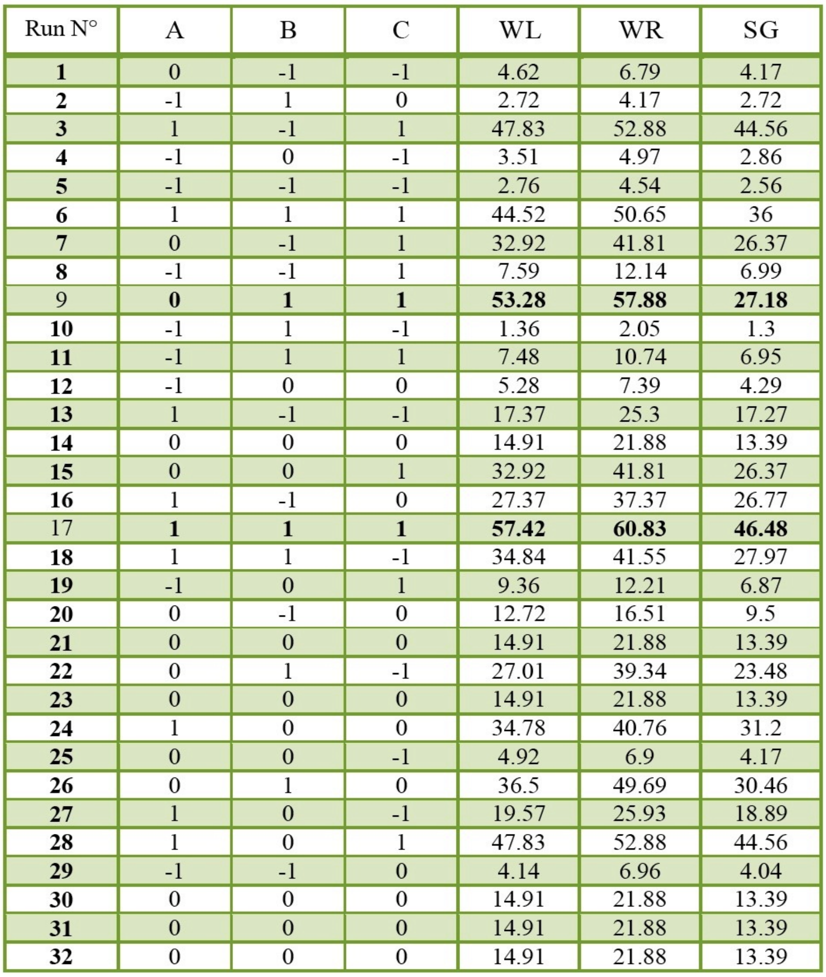
A model F-value of 31.13, 21.59, 44.27 for WL, WR and SG implies respectively that the model is significant 0.01% chance that a ‘Model F-Value’ this large could occur due to noise. The goodness of fit of the model is checked by the determination coefficient (R^2).

The coefficient of determination (R^2) was calculated to be 0.9272, 0.902 and 0.9477 for WL, WR and SG respectively. The Pred R^2 for WL (0.8259), SG (0.8907) and WR (0.7338) are in reasonable agreement with the Adj, respectively 0.8974, 0.768 and 0.9263 for WL, SG and WR. Adeq Precision measures the signal to noise ratio.

The Lack of Fit F-value of 36.68, 17.34 and 58.51 for WL, SG and WR implies the Lack of Fit is significant. There is only a 0.01% chance that a "Lack of Fit F-value" this large could occur due to noise. A ratio greater than 4 is desirable.

In this work the ratio is found to be > 20, which indicates an adequate signal. The experimental results are analyzed through RSM to obtain in empirical model for the best response (**Table 2**). The regression equation coefficients of the proposed models for each response are given in **Table 3**. The mathematical expression of relationship to the response with variables is shown below:

Table 2: Experimental conditions and observed response values of CCD



Run N°	A	B	C	WL	WR	SG
1	0	-1	-1	4.62	6.79	4.17
2	-1	1	0	2.72	4.17	2.72
3	1	-1	1	47.83	52.88	44.56
4	-1	0	-1	3.51	4.97	2.86
5	-1	-1	-1	2.76	4.54	2.56
6	1	1	1	44.52	50.65	36
7	0	-1	1	32.92	41.81	26.37
8	-1	-1	1	7.59	12.14	6.99
9	**0**	**1**	**1**	**53.28**	**57.88**	**27.18**
10	-1	1	-1	1.36	2.05	1.3
11	-1	1	1	7.48	10.74	6.95
12	-1	0	0	5.28	7.39	4.29
13	1	-1	-1	17.37	25.3	17.27
14	0	0	0	14.91	21.88	13.39
15	0	0	1	32.92	41.81	26.37
16	1	-1	0	27.37	37.37	26.77
17	**1**	**1**	**1**	**57.42**	**60.83**	**46.48**
18	1	1	-1	34.84	41.55	27.97
19	-1	0	1	9.36	12.21	6.87
20	0	-1	0	12.72	16.51	9.5
21	0	0	0	14.91	21.88	13.39
22	0	1	-1	27.01	39.34	23.48
23	0	0	0	14.91	21.88	13.39
24	1	0	0	34.78	40.76	31.2
25	0	0	-1	4.92	6.9	4.17
26	0	1	0	36.5	49.69	30.46
27	1	0	-1	19.57	25.93	18.89
28	1	0	1	47.83	52.88	44.56
29	-1	-1	0	4.14	6.96	4.04
30	0	0	0	14.91	21.88	13.39
31	0	0	0	14.91	21.88	13.39
32	0	0	0	14.91	21.88	13.39

The mathematical expression of relationship to the response with variables is shown below:

$$\begin{aligned} WL = {} & 16.24 + 15.89 \times A + 6.06 \times B + 10.00 \times C + 3.92 \times A \times B \\ & + 5.33 \times A \times C - 0.67\, B \times C - 2.20 \times A^2 + 5.63 \times B^2 + 3.80 \times C^2 \end{aligned} \tag{4}$$

$$\begin{aligned} WR = {} & 23.49 + 17.83 \times A + 6.28 \times B + 10.28 \times C + 3.58 \times A \times B \\ & + 4.08 \times A \times C - 2.08\, B \times C - 4.95 \times A^2 + 6.38 \times B^2 + 2.72 \times C^2 \end{aligned} \tag{5}$$

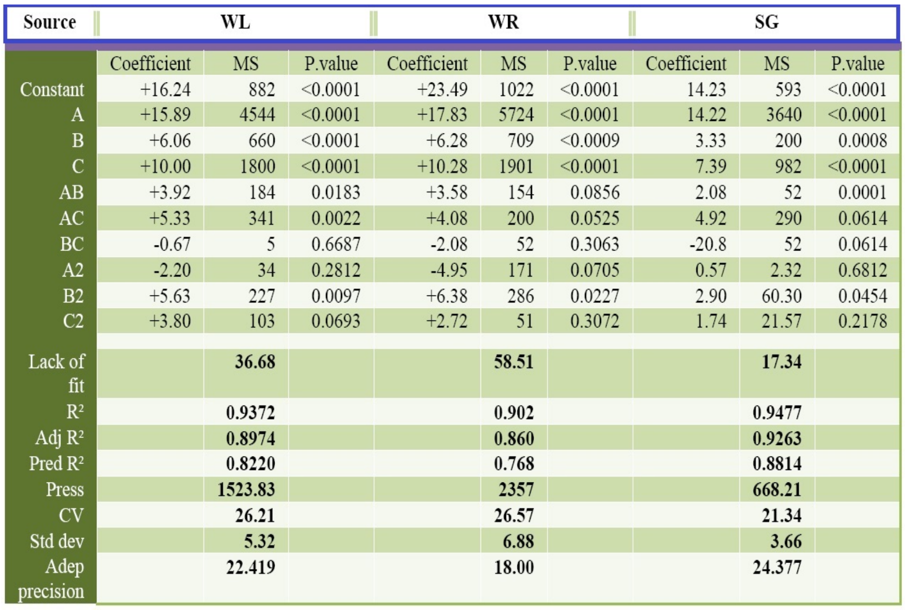
$$GS = 14.23 + 14.22 \times A + 3.33 \times B + 7.39 \times C + 2.08 \times A \times B + 4.92 \times A \times C + 2.08\, B \times C + 0.57 \times A^2 + 2.90 \times B^2 + 1.74 \times C^2 \quad (6)$$

where A, B and C, are the coded values of the test variables, temperature (°C), concentration (%w/w) and processing time (min), respectively. The results of multiple linear regressions conducted for the second order response surface model are given in **Table 3**.

The Model F-value of implies the model is significant. There is only a 0.01% chance that a 'Model F-Value' this large could occur due to noise. Values of 'Prob > F' less than 0.0500 indicate model terms are significant.

In this case A, B, C, AB, AC, $B2$ are significant model terms for WL, WR and SG.Values greater than 0.1000 indicate the model terms are not significant .

Table 3: Analysis of Variance (ANOVA) for response surface quadratic model for the osmotic dehydration of oranges pieces



Source	WL			WR			SG		
	Coefficient	MS	P.value	Coefficient	MS	P.value	Coefficient	MS	P.value
Constant	+16.24	882	<0.0001	+23.49	1022	<0.0001	14.23	593	<0.0001
A	+15.89	4544	<0.0001	+17.83	5724	<0.0001	14.22	3640	<0.0001
B	+6.06	660	<0.0001	+6.28	709	<0.0009	3.33	200	0.0008
C	+10.00	1800	<0.0001	+10.28	1901	<0.0001	7.39	982	<0.0001
AB	+3.92	184	0.0183	+3.58	154	0.0856	2.08	52	0.0001
AC	+5.33	341	0.0022	+4.08	200	0.0525	4.92	290	0.0614
BC	-0.67	5	0.6687	-2.08	52	0.3063	-20.8	52	0.0614
A2	-2.20	34	0.2812	-4.95	171	0.0705	0.57	2.32	0.6812
B2	+5.63	227	0.0097	+6.38	286	0.0227	2.90	60.30	0.0454
C2	+3.80	103	0.0693	+2.72	51	0.3072	1.74	21.57	0.2178
Lack of fit		**36.68**			**58.51**			**17.34**	
R^2		**0.9372**			**0.902**			**0.9477**	
Adj R^2		**0.8974**			**0.860**			**0.9263**	
Pred R^2		**0.8220**			**0.768**			**0.8814**	
Press		**1523.83**			**2357**			**668.21**	
CV		**26.21**			**26.57**			**21.34**	
Std dev		**5.32**			**6.88**			**3.66**	
Adep precision		**22.419**			**18.00**			**24.377**	

The sign and magnitude of the coefficients indicate the effect of the variable on the response. Negative sign of the coefficient means decrease in response when the level of the variable is increased while positive sign indicated increase in the response.

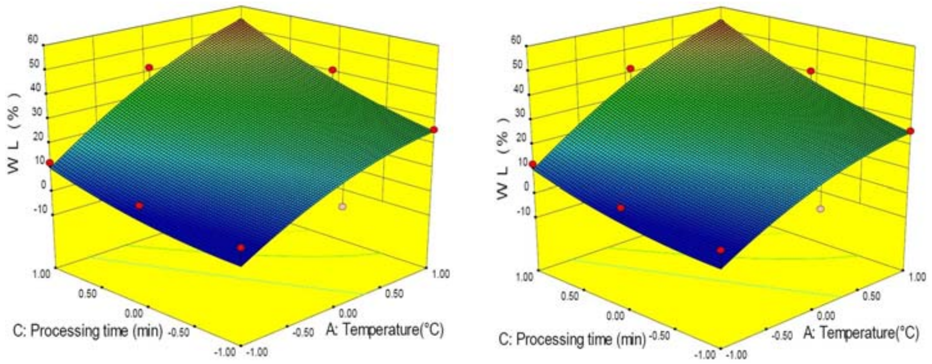
Significant interaction suggests that the level of one of the interactive variable can be increased that of other decreased for constant value of the response {Montgomery, (2004)}.

Effect of variables on water loss

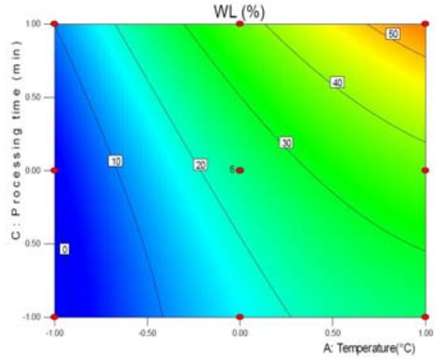
The magnitude of P and F values in **Table 3** indicates the maximum positive contribution of temperature, process time and concentration on the water loss during osmotic dehydration.

The figure 3 shows that the water loss increased with increase in temperature, in concentration and in processing time. The maximum value of water loss was observed for combination of higher temperature and concentration.

The quadratic terms of temperature have negative effect, and processing time and concentration solution have positive effect on water loss. The interactions of $A-B$, $A-C$ have positive effect, whereas the interactions of $B-C$ have negative effect on water loss.



Temperature Concentration



Time processing

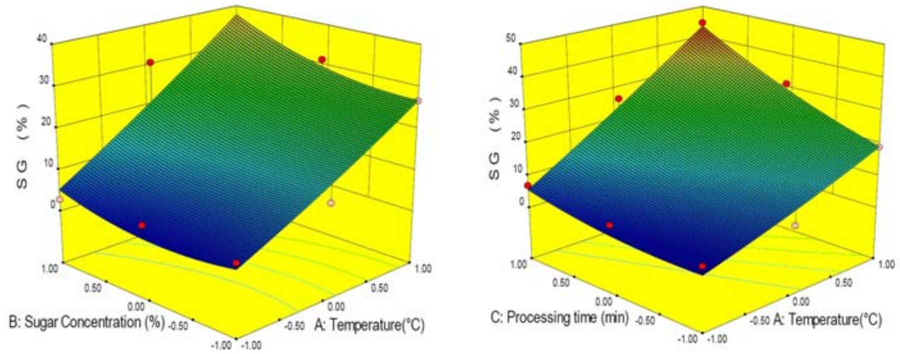
Fig. 3: Response surface plot and contour plot of the combined effect of the variables (temperature, concentration and time processing) on WL

Effect of variables on the solid gain

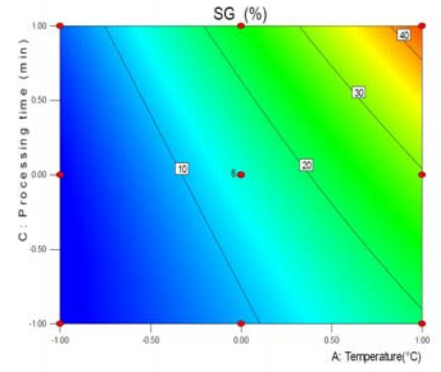
The linear effects of all variables show positive effect on solid gain. It implies increased solid gain with increase of process variables (Fig. 4).

The quadratic terms of processing time and concentration have negative effect on solid gain and temperature and solution to sample ratio has positive effect.

The interactive effects of $A-B$ and $A-C$ have positive effect on solid gain, whereas the interactive effects of $B-C$ have negative effect (**Table 3**).



Concentration Temperature



Time processing

Fig. 4: Response surface plot and contour plot of the combined effect of the variables (temperature, concentration and time processing) on SG.

4. CONCLUSION

In this study, RSM was used to determine the optimum operating conditions that yield maximum water loss and weight reduction and minimum solid gain in osmotic dehydration of oranges. Analysis of variance has shown that the effects of all the process variables including temperature, times, and sugar concentration were statistically significant.

Second order polynomial models were obtained for predicting water loss, solid gain and weight reduction. The optimal conditions for maximum water loss, weight reduction and solid gain (candying) correspond to temperature of 50^{o}C, processing time of 240 min, sugar concentration of 65% in order to obtain water loss of 60,83% (g/100 g fresh sample), solid gain of 46,48% (g/100 g fresh sample) and weight reduction of 57,42% (g/100 g fresh sample).

However the optimal conditions for maximum water loss and weight reduction and minimum solid gain (respectively 53.28 %, 57.88 % and 27.18 %) correspond to temperature of 40^{o}C, processing time of 240 min, and sugar concentration of 65 %.

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