Optimization of Osmotic Dehydration of Medlars Slices in Sucrose Solution Using Response Surface Methodology

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Abstract: Response surface methodology, according to CCD (central composite design), was used to determine the optimum processing conditions giving maximum water loss and minimum solid gain during osmotic dehydration of medlars in sucrose solution. The independent variables of osmotic dehydration were temperature (25-65 °C), processing time (20-240 min), sugar concentration (45%-65% w/w) and blanching time (0-180 s). The optimum conditions were found to be: temperature = 55 °C, time = 180 min, concentration = 60° Brix and blanching time = 30 s. At this optimum point, water loss, weight reduction and solid gain were found to be 74.12% and 7.136%, respectively.

Key words: Optimization, osmotic dehydration, medlars, sucrose, response surface methodology.

1. Introduction

Medlar (*Eriobotrya japonica* Lindeley) belongs to the Rosaceae family. The varieties of medlars grown in Algeria are: DrTrabut, Champagne, Tanaka, Vanilla and Saint-Michel. The medlars pieces are traditionally preserved using the candying by dipping in solution of sugar concentrated. The candying facilitates the penetration of solids into fruits and limits the loss in water, whereas the osmotic dehydration facilitates the loss in water and limits the gain of solids [1]. 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 [2, 3]. Solute transference to the fruit causes an increase of soluble solids content of the fruit which decreases the phenolase activity, hence reducing the susceptibility of the fruit to the enzymatic browning during air

dehydration [4]. Response surface methodology is often used to optimize the osmotic dehydration of fruits [5-7]. The objective of this study is to determine the effects of temperature, time, sucrose concentration and blanching time on the water loss and solute gain during osmotic dehydration of medlars using response surface methodology and also their desorption isotherm.

2. Materials and Methods

2.1 Materials

Fresh medlar and sugar, the osmotic agent, were purchased locally. The osmotic solution was prepared by mixing the sugar with proper amount of pure water. Osmotic dehydration: medlars were washed, destoned and cut into two slices. The samples were pretreated in boiling water during 0, 30, 60, 120 and 180 s, and then cooled in water to stop the effect of blanching. The samples were also immersed during 60 s in sodium metabisulfite solution at 1% in order to preserve the color of fruits. Ratio of sample/déshydration solution

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was 1:4. The osmotic dehydration was conducted in a 500 mL Erlenmeyer flask, which is placed in a thermostatically controlled water bath shaker. Medlars slices were weighed and placed into dehydrating vessel X_1 containing sugar solution (200 mL) of varying concentrations (45%-65% w/w). All the experiments were done in triplicate. At each sampling time (30, 60, 120, 180 and 240 min), the medlars slices were taken out and excess water is removed with adsorbent paper and weighed. The effect of temperature was also investigated and the experiments were conducted between temperatures of 25-65 °C. For each experiment, an agitation speed of 200 rpm was used. The average moisture content of the samples was determined by infraredhumimeter. WL (water loss) and SG (solid gain) were calculated according to the expressions [8]:

$$WL = \frac{S1 \times t \times PEe}{1 + S1 \times t} \quad (1)$$

$$SG = \frac{S2 \times t \times GSe}{1 + S2 \times t} \quad (2)$$

2.2 Experimental Design

Response surface methodology was used to optimize the WL and SG of medlars slices pretreated by osmotic dehydration in sucrose solutions. The effects of independent variables, temperature (25-65 °C), concentration (45%-65%), immersion times (30-240 min) and blanching time (0-180 s), on water loss and solid gain were studied using the CCD (central composite design). The coded and uncoded levels of different process variables are indicated in Table 1.

The second order response surface model used to fit the experimental data has the following form:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 + b_{12}X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 \quad (3)$$

where, Y is the response (WL and SG) and $b_0, b_{12}, \dots, b_{23}$, are constant coefficients of intercept, linear, quadratic, and interaction terms. X_1, X_2 and X_3 are coded independent variables [9]. Analysis was conducted using Statistica v.8. The quality of the

Table 1 Coded and uncoded values of variables and their levels.

Independents variables	Uncoded levels	Coded levels				
		- 2	- 1	0	1	2
Blanching time (s)	<i>A</i>	0	30	45	120	180
T (°C)	<i>B</i>	25	35	55	55	65
Time (min)	<i>C</i>	30	60	120	180	240
Concentration (%)	<i>D</i>	45	50	55	60	65

fitted model was evaluated by the ANOVA (analysis of variance).

2.3 Sorption Isotherm

The model used for sorption isotherms was the GAB (Guggenheim Anderson and Boer) model. The GAB equation is:

$$X_{eq} = \frac{X_m \times C \times K \times aw}{(1 - k \times aw)(1 - k \times aw + c \times k \times aw)} \quad (4)$$

where, X_{eq} is equilibrium moisture content, X_m is the monolayer moisture content, a_w is water activity, C is a constant related to the first layer heat of sorption and K is a factor related to a heat of sorption of the multilayer.

Both C and K are defined in Eqs. (5) and (6):

$$K = K' \times e^{\left(\frac{\Delta H_2}{RT}\right)} \quad (5)$$

$$C = C' \times e^{\left(\frac{\Delta H_1}{RT}\right)} \quad (6)$$

where, T is absolute temperature (K), R is the universal gas constant (8.314 J·mol⁻¹·K⁻¹).

ΔH_1 and ΔH_2 are heat of sorption functions:

$$\Delta H_1 = (H_m - H_q) \quad (7)$$

$$\Delta H_2 = (H_L - H_q) \quad (8)$$

where, H_L is latent heat of vaporization of the liquid water (43 kJ·mol⁻¹); H_m is total heat sorption of the monolayer (kJ·mol⁻¹); H_q is total heat sorption of the multilayer (kJ·mol⁻¹).

3. Results and Discussions

3.1 Response Surface Analysis

The 30 generated experiments with the values of various responses to different experimental combination for coded variables are given in Table 2.

Table 2 Experimental conditions and observed response values of CCD.

.Run	A	B	C	D	WL (%)	SG (%)
1	-1.00	1.00	-1.00	-1.00	59.47	11.24
2	-1.00	-1.00	1.00	-1.00	36.82	1.41
3	0.00	0.00	-2.00	0.00	64.58	7.68
4	0.00	2.00	0.00	0.00	67.56	10.64
5	-1.00	1.00	1.00	1.00	74.12	7.13
6	0.00	0.00	2.00	0.00	66.82	7.74
7	-1.00	-1.00	1.00	1.00	45.28	2.29
8	1.00	1.00	1.00	1.00	74.3	14.98
9	1.00	1.00	-1.00	-1.00	67.35	7.74
10	-1.00	1.00	1.00	-1.00	63.55	4.36
11	1.00	1.00	1.00	-1.00	66.88	11.44
12	1.00	1.00	-1.00	1.00	63.91	7.83
13	0.00	0.00	0.00	-2.00	42.26	7.69
14	1.00	-1.00	1.00	-1.00	44.62	4.79
15	-1.00	1.00	-1.00	1.00	70.61	9.14
16	1.00	-1.00	1.00	1.00	45.92	8.76
17	1.00	-1.00	-1.00	1.00	43.96	4.83
18	-2.00	0.00	0.00	0.00	58.99	3.56
19	0.00	-2.00	0.00	0.00	23.39	4.59
20	-1.00	-1.00	-1.00	-1.00	42.71	9.61
21	0.00	0.00	0.00	0.00	54.27	11.52
22	0.00	0.00	0.00	0.00	54.27	11.52
23	-1.00	-1.00	-1.00	1.00	43.62	6.92
24	0.00	0.00	0.00	0.00	54.27	11.52
25	1.00	-1.00	-1.00	-1.00	51.84	4.69
26	2.00	0.00	0.00	0.00	73.3	9.12
27	0.00	0.00	0.00	0.00	54.27	11.52
28	0.00	0.00	0.00	0.00	54.27	11.52
29	0.00	0.00	0.00	2.00	55.24	9.17
30	0.00	0.00	0.00	0.00	54.27	11.52

The experiments were conducted in accordance with the CCD to obtain the optimal combination of concentration, time and temperature for maximum water loss and minimum solid gains.

3.2 Fitting Models

The results of analysis of variance, carried out to estimate the quality of the fitted second order response surface model for water loss and solid gain, are presented in Tables 3 and 4, respectively.

The model F -value of WL which is 76.81 implies that the model is significant. Values of “Prob > F ” less than 0.0500 indicate that model terms are significant. In this study, A , B , D , AD , BC , BC , BD , CD , A^2 , B^2 , C^2

Table 3 Analysis of variance for response surface quadratic model for the osmotic dehydration of medlars—water loss.

Source	Water loss			
	Coefficient	SS	F	p -value
Model	54.27	4,303.04	76.81	< 0.0001
A —Blanching time	2.13	109.31	27.32	0.0001
B —Temperature	11.40	3,122.69	780.37	< 0.0001
C —Time	0.52	6.51	1.63	0.2215
D —Concentration	2.27	123.49	30.86	< 0.0001
AB	-0.83	10.92	2.73	0.1193
AC	0.081	0.11	0.026	0.8731
AD	-2.11	70.90	17.72	0.0008
BC	1.69	45.56	11.39	0.0042
BD	1.439	32.78	8.19	0.0119
CD	1.69	45.63	11.40	0.0042
A^2	2.87	226.19	56.53	< 0.0001
B^2	-2.30	144.57	36.13	< 0.0001
C^2	2.76	209.00	52.23	< 0.0001
D^2	-1.48	59.84	14.96	0.0015
Lack fit		60.02		
R^2		0.9862		
Adj- R^2		0.9734		
Pred- R^2		0.9208		

Table 4 ANOVA for response surface quadratic model for the osmotic dehydration of medlars—solid gain.

Source	Solid gain			
	Coefficient	SS	F	p -value
Model	11.52	306.99	62.22	< 0.0001
A —Blanching time	1.00	24.16	68.56	< 0.0001
B —Temperature	1.78	75.83	215.17	< 0.0001
C —Time	-0.28	1.88	5.34	0.0355
D —Concentration	0.40	3.81	10.81	0.0050
AB	0.45	3.31	9.40	0.0078
AC	2.29	83.72	237.57	< 0.0001
AD	0.55	4.93	13.98	0.0020
BC	0.67	7.24	20.53	0.0004
BD	0.13	0.25	0.71	0.4129
CD	0.98	15.44	43.83	< 0.0001
A^2	-1.33	48.41	137.37	< 0.0001
B^2	-1.01	27.97	79.36	< 0.0001
C^2	-0.99	26.67	75.67	< 0.0001
D^2	-0.81	17.82	50.57	< 0.0001
Lack fit		5.29		
R^2		0.9831		
Adj- R^2		0.9673		
Pred- R^2		0.902		

and D^2 are significant model terms. The high coefficient of determination (R^2) which is 0.986 shows that the fit of the model is good.

$$WL = +54.27 + 2.13 \times A + 11.41 \times B + 0.52 \times C + 2.27 \times D - 0.83 \times A \times B + 0.081 \times A \times C - 2.1 \times A \times D + 1.69 \times B \times C + 1.43 \times B \times D + 1.69 \times C \times D + 2.87 \times A^2 - 2.30 \times B^2 + 2.76 \times C^2 - 1.48 \times D^2$$

The model F -value of SG which is 62.22 implies that the model is significant. Values of “Prob > F ” less than 0.0500 indicate that model terms are significant. In this study, A , B , C , AD , BC , BC , CD , A^2 , B^2 , C^2 and D^2 are significant model terms. The high coefficient of determination (R^2) which is 0.983 shows that the fit of the model is good.

The final equation model for SG is as follows:

$$SG = +11.52 + 1.00 \times A + 1.78 \times B - 0.28 \times C + 0.40 \times D + 0.45 \times A \times B + 2.29 \times A \times C + 0.55 \times A \times D + 0.67 \times B \times C + 0.13 \times B \times D + 0.98 \times C \times D - 1.33 \times A^2 - 1.01 \times B^2 - 0.99 \times C^2 - 0.81 \times D^2$$

3.3 Response Surfaces and Contour Plots

The effects of independent variables (blanching time, temperature, concentration and immersion time) on the dependent variable (WL and SG) are indicated by the response surfaces plots developed from equations models mentioned above. We noted that for water loss, the temperature was the most important factor. The surface plots confirm that the linear term of temperature has a very significant effect on the water loss (Fig. 1). However, for solid gain, we noted that the quadratic term of all factors is significant. Fig. 2 shows that the solid gain increased with the increase of all factors then decreased, indicating the existence of optimal value.

4. Desorption Isotherms of the Medlars at 40 °C and 60 °C

The experimental moisture sorption data for medlars slices at 25 °C and 40 °C are shown in Fig. 3. The moisture sorption isotherms of the medlars slices dehydrated by osmosis were determined at 40 °C and 60 °C by balancing the atmosphere with relative

humidity fixed by dilute solutions of sulphuric acid. The results showed an increase in equilibrium moisture content with increasing water activity, at constant temperature, and are sigmoid in shape for the two examined temperatures. The values of RMS (root mean square) (%) are less than 10% and this allows us to conclude that the equations GAB can be used to predict the value of moisture in the equilibrium, and other parameters such as the moisture content of the mono-molecular layer and enthalpy of link to the monolayer and multilayer. The results of the analysis of the nonlinear regression of the adjustment of GAB equation in the experimental values are presented in Table 5.

In this study, it was found that for medlars slices, the monolayer moisture content, determined by GAB equation at 40 °C and 60 °C, were 9.07% and 8.4%, respectively.

5. RR (Rehydration Ratio)

Rehydration process was used to measure water absorption by dehydrated medlars. We noted that after 12 h, the water content of medlars rehydrated at ambient temperature increases from 48.3% to 89%. The rehydration allows the recovery of the shape and the size of the initial sample (Fig. 4). The RR is estimated at 0.31.

6. Conclusions

In this work, the response surface methodology was effective in determination of the optimum operating conditions giving maximum water loss and minimum solid gain during osmotic dehydration of medlars slices in sucrose solution. Analysis of variance has shown that the effects of the temperature, blanching time, immersion time and sucrose concentration were statistically significant. Second order polynomial models were obtained for predicting water loss and solid gain. The optimum conditions were found to be: temperature = 55 °C, time = 180 min, concentration = 60° Brix and blanching time = 30 s. At this optimum point, water loss, weight reduction and solid gain were found to be

74.12%, and 7.136%, respectively.

Fig. 1 Response surface and contour plots showing effect of processing variables on the WL: (a) temperature vs. blanching time; (b) immersion time vs. temperature and (c) temperature vs. concentration time.

Fig. 2 Response surface and contour plots showing effect of processing variables on the SG: (a) temperature vs. blanching time; (b) immersion time vs. temperature and (c) temperature vs. concentration.

Fig. 3 Moisture adsorption isotherm of the medlars slices at 40 °C and 60 °C.

Table 5 Estimated GAB parameters for medlars slices dehydrated by osmosis.

$T(^{\circ}\text{C})$	X_m	C_0	K_0	ΔH_c	ΔH_k	R^2
40	9.08	872	0.79	49.79	-172.42	0.98
60	8.45	496.1	0.37	29.45	-124.46	0.99

(a)

(b)

Fig. 4 Appearance of medlars slices (a) before and (b) after rehydration.

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