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Optimization of assisted ultrasound osmotic dehydration of strawberries slices in sucrose solutions using response surface methodology

A. Ferradji[1 *], M. Bouchareb[1], N.E.H. Bouhous[1] and A. Malek[2]

[1] Département de Technologie Alimentaire
Ecole Nationale Supérieure Agronomique
Avenue Hassan Badi, El Harrach, Alger, Algérie

[2] Centre de Développement des Energies Renouvelables, CDER
B.P. 62, Route de l'Observatoire, 16340 Bouzaréah, Alger, Algérie

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Abstract - *Response surface methodology, according to Central Composite Design (CCD), was used to determine the optimum processing conditions giving maximum water loss and minimum solid gain during osmotic dehydration of strawberries in sucrose solution. The independent variables of osmotic dehydration were temperature (40-65°C), processing time (60-180 min), sugar concentration (45-65% w/w) and ultrasound (0-45 kHz). Moisture sorption isotherms of the strawberries slices were determined at 25 and 40°C by balancing the atmosphere with relative humidity fixed by dilute solutions of sulphuric acid. The optimum conditions were found to be: temperature = 55°C, time = 180 min, sucrose concentration = 60°Brix and ultrasound at 25 kHz. At this optimum point, water loss% and solid gain% were found to be 84.80%, and 11.16% respectively. The value of water content of the monomolecular (Xm), is 9.95% at 25°C and 6.40% at 45°C.*

Résumé - *La Méthodologie des Surfaces de Réponse, suivant le Composite Central Design(CCD), a été utilisée pour optimiser les paramètres influençant la deshydratation osmotique des tranches de fraises dans une solution de saccharose en vue d'obtenir une perte en eau maximale et un gain en solide minimal. Les variables indépendantes de la deshydratation osmotiques ont la température (40-65°C), le temps (60-180 min), la concentration de solution de saccharose (45-65%) et l'ultrason (0-45 kHz). Les isothermes de sorption des tranches de fraises ont été déterminées à 25°C et 40°C à différentes humidités relatives fixées par des solutions d'acide sulfurique diluées. Les conditions optimales sont comme suit: température = 55°C, temps = 180 min, concentration de saccharose = 60°Brix et ultrason = 25 kHz. A ces conditions optimales, les pourcentages de perte en eau et de gain en solide sont respectivement de 84.80%, and 11.16%. Les teneurs en eau de la couche monomoleculaire (Xm) à 25°C et 40°C sont respectivement de 9.95 % et 6.40 %.*

Keywords: Strawberries - Osmotic deshydratation - Response Surface Methodology - Ultrasound - Sucrose.

1. INTRODUCTION

The cultivation of strawberries in Algeria occupies a surface area of 50118 ha with a annual production estimated at 422242 tons. The strawberries are generally consumed at fresh state and often used in pastry.

Several studies were carried out on the osmotic dehydration of slices strawberries and of fruits in sucrose solution, (Venkatachalapathy *et al.*, 1999; Raghavan *et al.*, 2001; Bchir *et al.*, 2011; Ferradji, 2015).

* a_ferradji@yahoo.fr

Recently some studies are focused on the ultrasound used as pretreatment before osmotic dehydration of slices strawberries (Garcia–Noguerra *et al.*, 2010).

The objective of this study is to dehydrate the slices of strawberries by osmotic dehydration assisted by ultrasound using Response Surface Methodology. The desorption isotherm are also investigated to determine the water content of monolayer and the energy required to evaporate the water during the final drying.

2. MATERIALS AND METHODS

2.1 Materials

Fresh strawberries of Camarosa cultivar and sugar, the osmotic agent, were purchased locally. The osmotic solution was prepared by mixing the sugar with proper amount of pure water. The pretreatment of strawberries slices was carried out using the ultrasonic bath (Transonic TI-H Fisher bioblock), (figure 1).

Fig. 1: Ultrasonic bath transonic TI-H (Fisher Bioblock)

2.2 Osmotic dehydration

Strawberries were washed, and cut in two slices. The samples immersed in sucrose solutions (45, 50, 65) were pretreated by ultrasound waves at 0, 25 and 45 kHz during 45 min with a power fixed at 100%. Ratio sample/dehydration solution was 1:4. The osmotic dehydration was conducted in a 500 ml Erlenmeyer flask, which is placed in thermostatically controlled water bath shaker. Strawberries slices were weighed and placed into dehydrating vessel containing sugar solution (200 ml) of varying concentrations (45– 65% w/w).

All the experiments were done in triplicate. At each sampling time 60, 120, 180 min, the strawberries 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 40 – 60°C.

For each experiment an agitation speed of 200 rpm was used. The average moisture content of the samples were determined by infrared humid meter. Water Loss (WL) and Solid Gain (SG) were calculated according to the expressions (Azuara, 1998).

$$WL = \frac{S_1 \times t \times PE_e}{1 + S_1 \times t} \quad (1)$$

$$SG = \frac{S_2 \times t \times GS_e}{1 + S_2 \times t} \quad (2)$$

2.3 Experimental design

Response surface methodology was used to optimize the water loss and solids gains of strawberries slices pretreated by osmotic dehydration in sucrose solutions. The effects of independent variables, temperature (40-60°C), concentration (45-65%), and immersion time (60-180min) and ultrasound at 45 kHz, on water loss and solid gain were studied using the Central Composite Design (CCD). The coded and uncoded levels of different process variables are indicated in **Table 1**.

Table 1: Coded and uncoded values of variablesand their levels

Independent variables	Uncoded levels	Coded values		
		-1	0	1
Concentration, %	A	40	50	60
Temperature, °C	B	40	50	60
Ultrason , kHz	C	0	25	45
Time, min	D	60	120	180

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

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_4 X_4 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 + b_{44} X_4^2 + b_{12} X_1 X_2 + b_{13} X_1 X_3 + b_{14} X_1 X_4 + b_{23} X_2 X_3$$

where Y is the response (WL and SG) and b_0, $b_{12} ...$ b_{23}, are constant coefficients of intercept, linear, quadratic, and interaction terms. X_1, X_2, X_3, X_4 are coded independent variables analysis was conducted using Design – Expert 9.

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

2.4 Sorption isotherm

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

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

where, X_{eq} is equilibrium moisture content**,** X_m is the monolayer moisture content, a_w is water activity, C 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 Equation 6 and 7.

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

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

where T, is absolute temperature (K), R, is the universal gas constant (8.314 J/mol.K), ΔH_1 and ΔH_2 are heat of sorption functions: $\Delta H_1 = (H_m - H_q)$; $\Delta H_2 = (H_L - H_q)$; H_L is latent heat of vaporization of the liquid water (43 kJ/mol); H_m is total heat sorption of the monolayer (kJ/mol). H_q is total heat sorption of the multilayer (kJ/mol).

The three GAB constants depend on product characteristics and temperature, they square root of the error (RMS%):

$$\text{RMS\%} = 100 \sqrt{\frac{\sum \left(\frac{X_{exp} - X_{cal}}{X_{exp}} \right)^2}{N}} \quad (7)$$

Where, X_{exp} and X_{cal} are the experimental and calculated moisture contents and N is the number of experimental points.

3. RESULTS AND DISCUSSIONS

The twenty nine (29) 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	Concentration of sucrose	Température	Ultrasound Fréquence	Time	WL%	SG%
1	1	0	0	-1	47.53	4.7
2	-1	0	-1	0	42.46	3.57
3	0	1	1	0	68.68	9.79
4	0	1	0	-1	40.63	3.89
5	-1	0	0	1	58.62	5.28
6	0	-1	0	1	60.11	7.14
7	0	0	0	0	60.26	5.05
8	0	0	1	-1	30.55	3.11
9	0	1	-1	0	63.63	4.96
10	1	1	0	0	68.81	10.65
11	0	0	1	1	67.53	5.93
12	0	0	-1	-1	40.02	4.00
13	0	1	0	1	75.23	14.85
14	0	0	-1	1	58.93	6.05
15	-1	0	0	-1	50.87	3.73
16	-1	-1	0	0	60.02	6.48
17	1	0	0	1	84.80	11.76
18	0	0	0	0	82.32	7.79
19	-1	0	1	0	75.79	7.86
20	0	-1	0	-1	56.62	12.76
21	-1	1	0	0	74.19	6.72
22	0	-1	1	0	70.4	5.88
23	1	0	1	0	75.46	8.29
24	0	-1	-1	0	52.97	4.06
25	1	0	-1	0	65.98	5.36
26	0	0	0	0	81.14	7.15
27	0	0	0	0	80.91	7.14
28	0	0	0	0	74.65	6.4
29	1	-1	0	0	79.81	6.5

3.1 Response surface analysis

Fitting models

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

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

Table 3: Analysis of variance for response surface quadratic model

for the osmotic dehydration of strawberries -water loss

Source	Coefficient	Water loss		
		SS	F	p-valeur
Model	75.86	5071.81	7.64	0.003
A-Concentration	5.04	304.42	6.42	0.0239
B-Temperature	0.94	10.53	0.22	0.6448
C-Ultrasound	5.37	345.83	7.29	0.0173
D-Time	11.58	1610.08	33.94	<0.0001
AB	-6.29	158.38	3.34	0.0891
AC	-5.96	142.21	3.00	0.1053
AD	7.38	217.86	4.59	0.0502
BC	-3.10	38.32	0.81	0.3840
BD	7.78	241.96	5.10	0.0404
CD	4.52	81.63	1.72	0.2107
A^2	-1.12	8.14	0.17	0.6849
B^2	-2.78	49.97	0.17	0.3221
C^2	-10.11	663.3	1.05	0.0022
D^2	-15.23	1505.51	13.73	<0.0001
Lack Fit		324.16	0.38	0.9014
R^2		0.884		
Adj-R^2		0.768		

Table 4: Analysis of variance (ANOVA) for response surface quadratic model for the osmotic dehydration of strawberries–solid gain

Source	Coefficient	Solid grain		
		SS	F	p-valeur
Model	6.71	186059	4.47	0.0042
A-Concentration	1.16	16.03	5.38	0.0360
B-Temperature	0.65	5.06	5.38	0.2138
C-Ultrasound	1.07	13.78	4.62	0.0495
D-Time	1.57	29.52	9.90	0.0071
AB	0.92	3.35	1.12	0.3072
AC	-0.34	0.46	0.16	0.6996
AD	1.38	7.59	2.55	0.1329
BC	0.75	2.27	0.76	0.3881
BD	4.15	68.72	23.05	0.0003
CD	0.19	0.15	0.050	0.8268
A^2	-3.02	0.03	0.01	0.9715
B^2	1.57	16.05	5.38	0.0360
C^2	-1.56	15.80	0.12	0.0372
D^2	0.23	0.35	3.40	0.7377
Lack Fit		41.74	0.38	0.9014
R^2		0.817		
Adj-R^2		0.634		

Water Loss

The Model F-value of WL which is 74.41 implies that the model is significant. Values of "Prob > F" less than 0.0500 indicate that model terms are significant.

In this study B, C, D, AB, BC, BD, D^2 are significant model terms. The high coefficient of determination (R^2) which is of 0.884 shows that the fit of the model is good.

The final equation model for WL:

$$\begin{aligned} WL = {} & +75.86 + 5.04 \times A + 0.94 \times B + 5.37 \times C + 11.58 \times D - 6.29 \times AB \\ & - 5.96 \times AC + 7.38 \times AD - 3.10 \times BC + 7.78 \times BD + 4.52 \times CD \\ & - 1.12\ A^2 - 2.78 \times B^2 - 10.11 \times C^2 - 15.23 \times D^2 \end{aligned}$$

Solids Gain

The Model F-value of SG which is 6.71 implies that the model is significant. Values of "Prob> F" less than 0.0500 indicate that model terms are significant.

In this study B, C, D, A^2, C^2 are significant model terms. The high coefficient of determination (R^2) which is of 0.817 shows that the fit of the model is good.

The final equation model for SG:

$$
\begin{aligned}
SG =& + 6.71 + 1.16 \times A + 0.65 \times B + 1.07 \times C + 1.57 \times D - 0.92 \times AB \\
& + 0.34 \times AC + 1.38 \times AD + 0.75 \times BC - 4.15 \times BD + 0.19 \times CD \\
& - 3.02 \times A^2 - 1.57 \times B^2 - 1.56 \times C^2 - 0.23 \times D^2
\end{aligned}
$$

3.2 Response surfaces

The effects of independent variables (temperature, concentration, immersion time and the ultrasound on the dependents variables (WL and SG) are indicated by the response surfaces plots developed from equations models mentioned above (figures 2, 3). The results of analysis variance (**Table 2**) show that the effect of time ($p < 0.0001$) on the WL and SG was the most important factor followed by ultrasound ($p = 0.013$) and concentration ($p = 0.020$).

We noted also that the values of temperature lies between 40°C and 60°C have a weak effect on the WL and SG. For the pretreatment of slices strawberries carried out at 25 kHz the water loss percentage obtained is 84, 80%. However at 45 kHz with the same values of temperature and time the value of WL % is only 67.53 % (**Table 1**, see run 17 and 11).

This result is similar to value reported by Fernandes *et al.* (2008). These authors have reported that the weak percentage of WL % at 45 kHz was caused by extensive damage to the strawberries tissues .The sucrose penetrating into micro channels coat the inter tissues cellules surfaces and consequently prevent the leaving of water.

Venkatachalapathy *et al.* (1999) have also reported that the low value of WL% at 45kHz would be caused by the increase of energy absorption by solutions and consequently reduce the penetration of ultrasound waves into strawberries tissues.

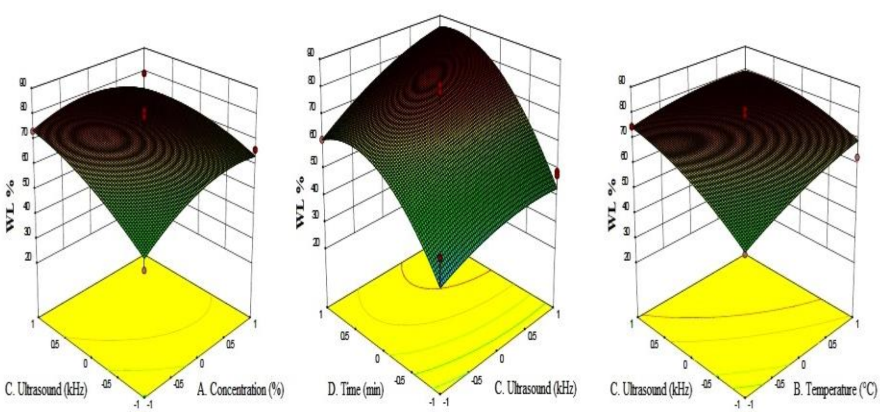

Fig. 2: Response surface and contour plots showing effect processing variables on the WL: ultrasound v/s concentration, immersion time v/s ultrasound temperature v/s ultrasound

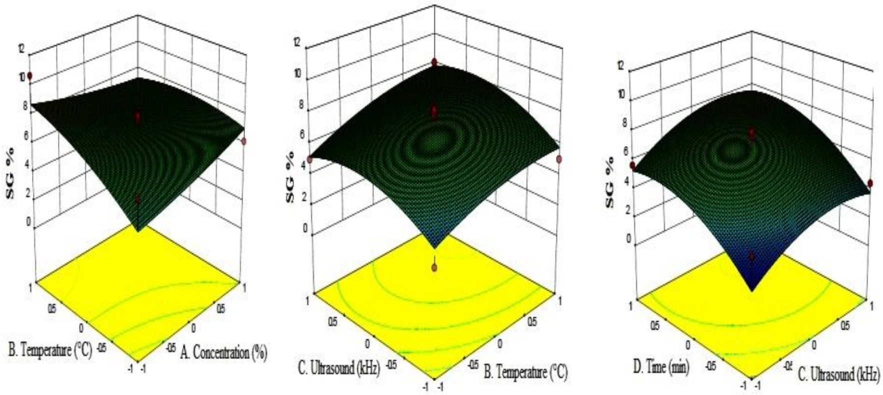

Fig. 3: Response surface and contour plots showing effect processing variables on the SG: temperature v/s concentration, ultrasound v/s temperature immersion time v/s ultrasound

3.3 Desorption isotherms of the strawberries slices at 25 and 45°C

The experimental moisture sorption data for strawberries slices at 25 and 45 °C are shown in figure 3. The moisture sorption isotherms of the strawberries slices dehydrated by osmosis were determined at 40 and 60°C by balancing the atmosphere with relative humidity fixed by dilute solutions of sulphuric acid (a_w : 0.0016; 0.043; 0.355; 0.65; 0.812; 0.942)**.**

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 (%) 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 on the **Table 4**.

Table 4: Estimated GAB parameters for strawberries slices dehydrated by osmosis

T (°C)	X_m	C_0	K_0	ΔH_C (J/mole)	ΔH_K(J/mole)	R^2
25	9.95	4191	0.754	1013	1446.42	0.990
45	6.40	105161	0.835	546	-2394.46	0.978

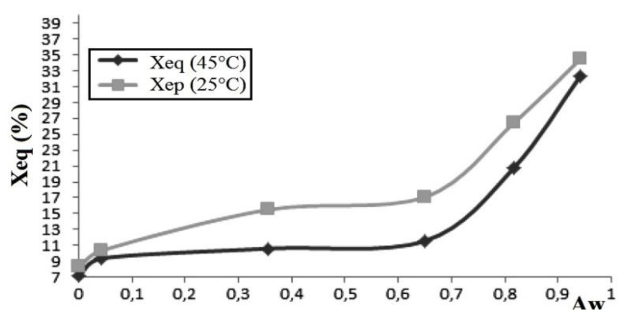

Fig. 4: Moisture adsorption isotherm of the strawberries slices at 25 and 45°C

3.4 Calculation of the sorption heat

In order to determine the energy required for evaporation of water from strawberries slices, during drying, the desorption heat of the mono-molecular layer (H_m) and the multilayer (H_q) were evaluated using respectively the parameters $\Delta H_1 = H_m - H_q$ and $\Delta H_2 = H_L - H_q$.

The results show that the heat of desorption of the mono-molecular layer, $H_m = 2419.24\ kJ/kg$, is more important than that of the multilayer, $H_q = 2362.97\ kJ/kg$.

The required energy to evaporate the water from 1 kg of strawberries slices pre-treated by osmotic dehydration can be evaluated using the following formula:

$$Q = H_q \times m_e \qquad (8)$$

where H_q = heat desorption of the multilayer; m_e = amount of water to evaporate calculated according to the following formula:

$$m_e = m_i \frac{mc_i - mc_f}{100 - mc_f} \qquad (9)$$

where m_i, mass of sample = 1 kg; mc_i, rate of initial humidity = 40.02%; mc_f, rate of final humidity = 8.13%. Either: m_e = 0.347 kg of water evaporated. Hence: $Q = 403.12\ kJ$. Since it was 1 kWh = 3.6×10^3 kJ the value of energy required will be: $Q = 0.227\ kWh$.

The strawberries slices after osmotic dehydration keep the primary colour with an improvement of aspect (figure 5).

Fig. 5: Aspect of Strawberries slices after osmotic dehydration

4. CONCLUSIONS

In this work, the response surface methodology was effective in determination the optimum operating conditions giving maximum water loss and minimum solid gain during osmotic dehydration of strawberries slices in sucrose solution.

Analysis of variance has shown that the effects of the temperature, immersion time and sucrose concentration and ultrasound were statistically significant.Second order polynomial models were obtained for predicting water loss and solid gain.

The optimum conditions were found to be: temperature = 50 °C, time = 120 min, concentration = 50°Brix, and pre treated at 25 kHz during 45 min. At this optimum point, water loss, weight reduction and solid gain were found to be 84.80 % and 11.16 %, respectively.

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