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Optimization of osmotic dehydration of tomatoes slices in salt and sucrose solutions using response surface methodology

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Abstract - *Response Surface Methodology (RSM) was used to investigate the effect of temperature solution, immersion time, salt concentration and sucrose concentration on the water loss and solid gain during osmotic dehydration of tomatoes slices. The optimum conditions for maximum WL% and SG % are: T=55°C, t =240 min and C=74% for the sucrose and T=55°C, t= 240 min and C= 5% for the salt. The value of water content of the monomolecular layer (X_m),is 17.07 % at 25°C.*

Résumé – *La Méthodologie des Surfaces de Réponses (MSR) a été utilisée pour étudier l'effet de la solution à la température, de la durée de trempage, et de la concentration des solutions de sel et de saccharose sur la perte en eau et du gain en solide au cours de la déshydratation osmotique des tranches de tomates. Les conditions optimales correspondant aux pourcentages élevés de perte en eau et de gain en solides sont: T=55°C, t =240 min et C%=74% pour le saccharose et T=55°C, t=240 min et C%=5% pour la solution saline. La teneur en eau de la couche mono moléculaire (X_m) est de 17.07% à 25°C.*

Keywords: Tomatoes - Osmotic Dehydration – Response Surface Methodology – Salt – Sucrose.

1. INTRODUCTION

In Algeria, the area devoted to the cultivation of tomatoes (*Solanumlycopersicum L.*) is estimated at 33 000 ha, which gives an average production of 11 million quintals. Tomatoes are preserved traditionally as sprinkled slices with salt, sun-dried and used in some traditional dishes such as couscous and sauces. Industrially tomatoes aretransformed into concentrated pulp.

Several studies focused on the osmotic dehydration of tomatoes slices in solutions of sucrose or salt had confirmed that the osmotic dehydration is an effective pretreatment for drying tomatoes because it allows better retention of lycopene and a decrease drying time [1-6].

The objective of this study is to dehydrate tomatoes slices by osmosis in solutions of sugar and salt using the response surface methodology. Desorption isotherms were also studied to determine the water content of the monomolecular layer and the energy required to evaporate the water during drying.

2. MATERIALS AND METHODS

2.1 Materials

The tomatoes were purchased from a local market. The initial moisture content of fresh tomatoes was 82%. Tomatoes slices blanched in boiling water for 30 seconds are

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immersed in a solution of sodium metabisulfite at 1% during 1 second and then in a solution of cornstarch at 2.5% for 3 to 4 minutes.

2.2 Osmotic dehydration

Tomatoes slices were osmotically dehydrated in salt and sucrose solutions at different temperatures (25, 45 and 60°C), concentrations (52, 67 and 74%) and times (15, 90 and 240 min). The ratio of tomatoes slices to solution was 1:20. Samples were withdrawn at periodic intervals during 6h.Excess solution from the surface was blotted and dried using paper towels. In the continuous method, each sample was weighed and returned to the osmotic solution to continue the drying process. After 6h the moisture content of the sample was determined in a infrared humidimeter, each experimental treatment was performed in triplicate runs. Water loss and solid gain during osmotic dehydration were calculated using following equation [7].

$$\mathrm{WFL} = \frac{s_1 t\, \mathrm{WFL}_\infty}{1 + s_1 t} \tag{1}$$

$$\mathrm{SG} = \frac{s_2 t\, \mathrm{SG}_\infty}{1 + s_2 t} \tag{2}$$

2.3 Extraction and determination of lycopene content

Lycopene was extracted from tomatoes using petroleum ether according the method described by Chen *et al.* [8]. The color was measured in a 1 cm cell at 503 nm using petroleum ether as blank.

2.4 Experimental design

Response surface methodology was used to optimize the water loss and solid gain of tomatoes slices pretreated by osmotic dehydration in salt and sucrose solutions. The effects of independent variables, temperature (25-60 °C), concentration (45 – 74%) and immersion times (15- 240 min) on water loss and solid gain were studied using the Central Composite Design. The coded and uncoded levels of different process variables are indicated in **Table 1**. The second response surface model used to fit the experimental data has the following form:

$$\begin{aligned} Y = {} & b_0 + b_1 X^1 + b_2 X^2 + b_3 X^3 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 \\ & + b_{12} X_1 X_2 + b_{13} X_1 X_3 + b_{23} X_2 X_3 \end{aligned} \tag{3}$$

Where Y is the response (WL and SG) and b_0, $b_{12} \ldots b_{23}$, are constant coefficients of intercept, linear, quadratic, and interaction terms. X_1, X_2 and X_3 are coded independent variables [9, 10]. Analysis was conducted using Statistica v.8. The quality of the fitted model was evaluated by the analysis of variance (ANOVA).

Table 1: The coded and uncoded levels of different process variables used in Composite Central Design (CCD)

Independent variables	**Symbol**		**Levels**	
	Uncoded	Coded	Uncoded (salt)	Uncoded (sucrose
Concentration (%)	C	-1	5	50
		0	10	60
		1	15	70
Time (min)	t	-1	60	60
		0	120	120
		1	240	240

Temperature (°C)	T	-1	25	25
		0	45	45
		1	60	55

2.5 Sorption isotherm

The model used for sorption isotherms was the GAB model (Guggenheim, Anderson and Boer) [11]. The GAB equation is:

$$X_{eq} = \frac{X_m \times C \times K \times a_w}{(1 - K \times a_w) \times (1 - K \times a_w + C \times K \times a_w} \tag{4}$$

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 {Eq. (5)} and {Eq. (6)}.

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

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

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 ($RMS\%$):

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

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

Table 2: Experimental conditions and observed response values of CCD

Salt									**Sucrose**				
coded				**uncoded**					**uncoded**				
Test	**C**	**t**	**T**	**C**	**t**	**T**	**WL%**	**SG%**	**C**	**t**	**T**	**WL%**	**SG%**
1	1	-1	1	15	60	55	49.02	9.09	70	60	55	65.27	13.22
2	-1	0	0	5	120	45	45.3	5.02	50	120	45	72.54	16.07
3	1	1	-1	15	240	35	39.5	7.75	70	240	35	61.53	8.14
4	0	0	1	10	120	55	55.45	5.93	60	120	55	75.99	8.01
5	-1	1	1	5	240	55	63.02	3.32	50	240	55	79.49	12.35

To be continue (**Table 2**)

Salt									Sucrose				
Coded				**Uncoded**					**Uncoded**				
Test	**C**	**t**	**T**	**C**	**t**	**T**	**WL%**	**SG%**	**C**	**t**	**T**	**WL%**	**SG%**
6	-1	1	-1	5	240	35	38.92	5.61	50	240	35	57.59	10.23
7	-1	-1	1	5	60	55	35.18	6.72	50	60	55	63.14	7.77
8	-1	-1	-1	5	60	35	17.84	7.30	50	60	35	50.17	11.51
9	0	-1	0	10	60	45	33.16	9.67	60	60	45	57.87	12.62
10	0	0	0	10	120	45	46.5	7.31	60	120	45	67.23	12.78
11	0	0	0	10	120	45	50.23	6.77	60	120	45	69.21	12.66
12	0	1	0	10	240	45	48.71	7.81	60	240	45	74.28	12.9
13	0	0	1	10	120	55	54.67	5.95	60	120	55	76.81	8.39
14	1	-1	-1	15	60	35	22.33	8.44	70	60	35	51.74	14.22
15	1	0	0	15	120	45	47.78	6.83	70	120	45	74.28	16.92
16	1	1	1	15	240	55	65.12	7.65	70	240	55	86.85	14.21
17	1	-1	1	15	60	55	46.69	9.08	70	60	55	64.41	12.09
18	-1	0	0	5	120	45	45.49	6.01	50	120	45	68.45	15.02
9	1	1	-1	15	240	35	42.14	7.69	70	240	35	62.14	8.09
20	0	0	-1	10	120	35	35.91	6.82	60	120	35	57.07	7.65
21	-1	1	1	5	240	55	65.43	3.30	50	240	55	78.79	12.48
22	-1	1	-1	5	240	35	38.64	5.69	50	240	35	59.63	10.31
23	-1	-1	1	5	60	55	38.38	6.63	50	60	55	62.69	7.99
24	-1	-1	-1	5	60	35	19.09	7.46	50	60	35	50.93	11.55
25	0	-1	0	10	60	45	31.46	9.83	60	60	45	58.1	12.79
26	0	0	0	10	120	45	42.98	7.13	60	120	45	69.57	12.43
27	0	0	0	10	120	45	43.13	6.81	60	120	45	71.34	12.77
28	0	1	0	10	240	45	52.03	7.47	60	240	45	75.45	12.86
29	0	0	1	10	120	55	56.85	5.91	60	120	55	76.28	8.23
30	1	-1	-1	15	60	35	22.37	8.37	70	60	35	49.56	12.75
31	1	0	0	15	120	45	47.61	7.12	70	120	45	75.74	17.03
32	1	1	1	15	240	55	64.66	6.68	70	240	55	86.34	15.48

C = Concentration; t = Time; T = Temperature

3. RESULTS AND DISCUSSIONS

3.1 Response surface analysis

The thirteen two (32) generated experiments with the values of various responses to different experimental combination for coded variables are given in **Table 2**. A large variation in the WL and SG for osmotic dehydration, carried out in sucrose and salt solutions, was observed for different experimental combinations. The experiments were conducted in accordance with the CCD design to find the optimal combination of concentration, time and temperature for maximum water loss and minimum for solid gains. We noted that water loss was higher when sucrose was used as osmotic agent and that solids gain was low for the salt solution.

3.2 Fitting models

The results of analysis of variance carried out to estimate the quality of the fitted second order response surface model are shown in **Table 3 and 4**. The sign and magnitude of the coefficients allows interpreting the effect of the variable on the response. The negative sign of coefficient indicates that when the level of the variable increases the response decreases, while the positive sign indicates an increase in the response.

The Model F-value of WL and SG which was respectively 197.59 and 129.99 for the sucrose and 95.50 and 80.48 for the salt, implies that the model is significant. The Lack of Fit F-value 2.00 (WL) and 1.11 (SG) for sucrose, and 2.00 (WL) and 2.01 (SG) for salt implies that the Lack of Fit is not significant.

Values of $"\mathrm{Prof} > \mathrm{F}"$ less than 0.0500 indicate that model terms are significant. In this study for sucrose $C, t, T, C\times t, C\times T, t\times T, C^2, t^2$ and T^2 are significant model terms. The high coefficient of determination (R^2) which is of 0.987 (WL) and 0.981 (SG) shows that the fit of the model is good.

In conclusion the final models for the response variable WL and SG are following

$$\begin{aligned} WL\% = 70.187 + 1.722\times C + 2.139\times C^2 + 7.410\times t - 4.188\times t^2 + 9.130\times T \\ - 3.640\times T^2 + 1.081\times C\times t + 0.756\times C\times T + 2.341\times t\times T \end{aligned} \tag{8}$$

$$\begin{aligned} SG\% = 12.701 + 0.843\times C + 3.538\times C^2 + 0.0270\times t + 0.710\times t^2 + 0.509\times T \\ - 4.906\times T^2 - 0.806\times C\times t + 0.756\times C\times T + 2.341\times t\times T \end{aligned} \tag{9}$$

Significant model terms for the salt solution indicated in **Table 4** are respectively for WL and SG: $C,\ t,\ T,\ C\times t,\ C\times T,\ t\times T, C^2,\ t^2$ and T^2. The values of R^2 which are respectively 0.987 and 0.981 for WL and SG show that the fit of the model is good. The final models for the two response (WL and SG) are following

$$WL\% = 51.60 + 2.45\times C + 9.32\times t + 10.43\times T - 1.30\times C\times t - 2.87\times T^2 \tag{10}$$

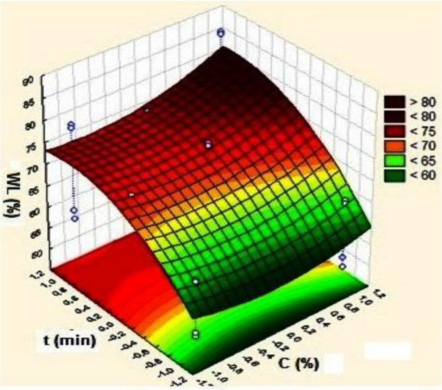
$$\begin{aligned} SG\% = 7.10 + 1.08\times C - 0.98\times t - 0.37\times T + 0.806\times C\times t + 0.40\times C\times T \\ + 0.36\times t\times T - t^2 + 0.90\times C^2 - 0.81\times T^2 \end{aligned} \tag{11}$$

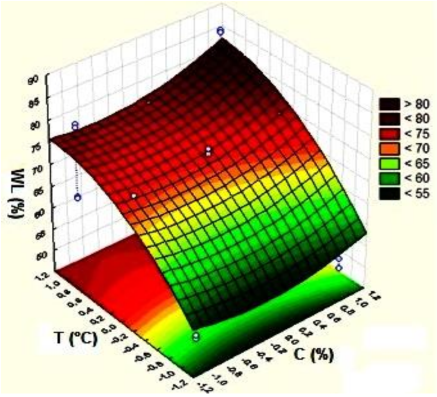
3.3 Response surfaces and contour plots

The effect of independent variables (temperature, concentration, time) on the dependents variable (WL and SG) is indicated by the response surfaces plots developed from equations models mentioned above (figure 1, 2). We observed that the surface plots confirm that the linear term of temperature, concentration, and time had a very significant effect on the water loss and solid gain.

Table 3: Analysis of variance for water loss (WL) and solids gain (SG) in the osmotic dehydration of slices tomatoes in sucrose solution

	Water loss				**Solid gain**			
Source	**Coef.**	**Sum of Squares**	**F**	**P**	**Coef.**	**Sum of Squares**	**F**	**P**
Model	70.19	3181.79	197.59	< 0.0001	12.70	232.03	129.99	< 0.0001
C (%)	1.72	59.31	33.15	< 0.0001	0.84	14.32	71.75	< 0.0001
t (min)	7.41	1098.31	613.85	< 0.0001	0.027	0.015	0.074	0.7888
T (°C)	9.13	1595.38	891.66	< 0.0001	0.51	4.96	25.00	< 0.0001
C× t	1.08	18.73	10.47	0.0038	-0.81	10.42	52.52	< 0.0001
C×T	0.76	9.17	5.12	0.0338	0.93	13.71	69.12	< 0.0001
t×T	2.34	87.75	49.04	< 0.0001	1.67	44.59	224.82	< 0.0001
C^2	2.14	23.85	13.33	0.0014	3.54	65.25	329.00	< 0.0001
t^2	-4.19	91.41	51.09	< 0.0001	0.071	0.026	0.13	0.7194
T^2	-3.64	68.28	38.16	< 0.0001	-4.91	123.97	625.09	< 0.0001
Residue		39.36				4.36		
Lack of fit		14.58	2.00	0.1304		1.08	1.11	0.3907
Pure Error		24.79				3.29		
Total		3221.15				236.40		
R^2		0.9878				0.9815		
Adjust-R^2		0.9828				0.9740		
Predict- R^2		0.9768				0.9526		





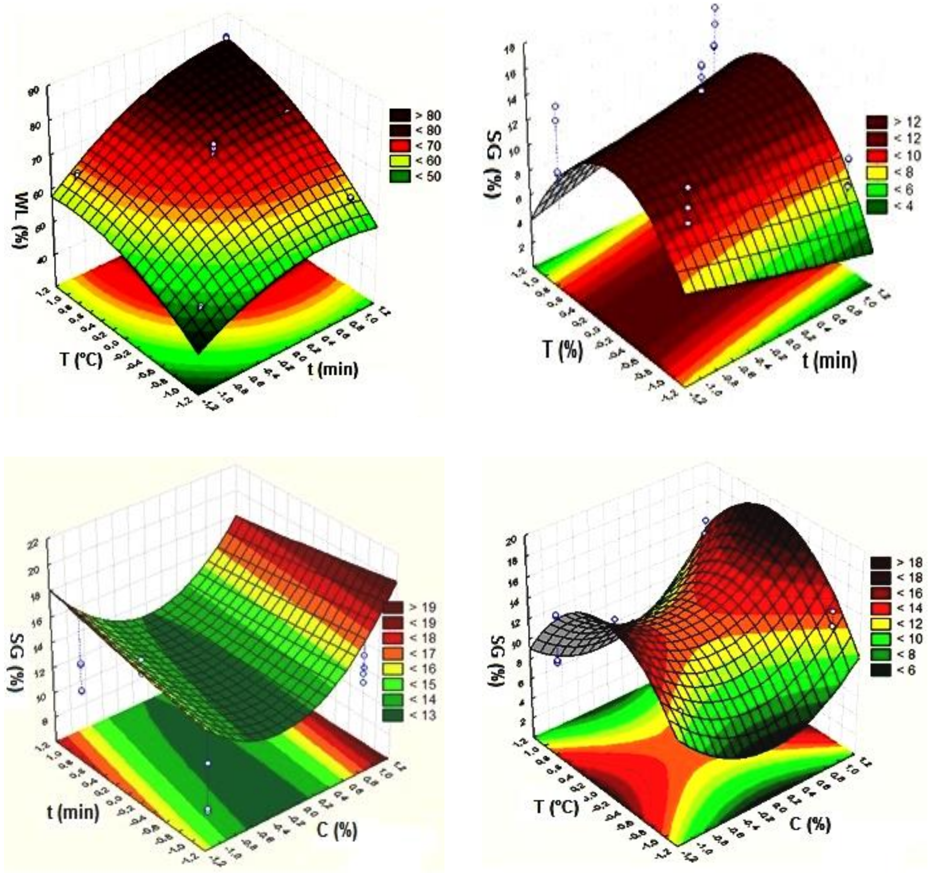


Fig.1: Response surface plots of WL and SG in sucrose solution

Table 4: Analysis of variance for water loss (WL) and solids gain (SG) in the osmotic dehydration of slices tomatoes in salt solution

	Water loss				**Solid gain**			
Source	**Coef.**	**Sum of Squares**	**F**	**P**	**Coef.**	**Sum of Squares**	**F**	**P**
Model	51.60	4164.93	95.50	< 0.0001	7.10	68.14	80.48	< 0.0001
C (%)	2.45	120.00	24.76	< 0.0001	1.08	23.41	248.89	< 0.0001
t (min)	9.32	1735.57	358.1	< 0.0001	-0.98	19.25	204.60	< 0.0001
T (°C)	10.43	2081.57	429.5	< 0.0001	-0.37	2.66	28.25	< 0.0001
C× t	-1.30	27.07	5.59	0.0274	0.31	1.55	16.48	0.0005
C×T	1.06	17.91	3.70	0.0676	0.40	2.51	26.70.	< 0.0001
t×T	0.49	3.83	0.79	0.3835	-0.36	2.06	21.89	0.0001
C^2	-0.75	2.89	0.60	0.4479	-0.90	4.25	45.15	< 0.0001
t^2	-0.76	3.01	0.62	0.4390	1.55	12.48	132.61	< 0.0001

T^2	-2.87	42.42	8.75	0.0073	-0.81	3.37	35.83	< 0.0001
Residue		106.61				2.07		
Lack of fit		39.50	2.00	0.1300		0.77	2.01	0.1285
Pure Error		67.10				1.30		
Total		4271.54				70.21		
R^2		0.9750				0.9705		
Adjust-R^2		0.9648				0.9585		
Predict- R^2		0.9547				0.9385		

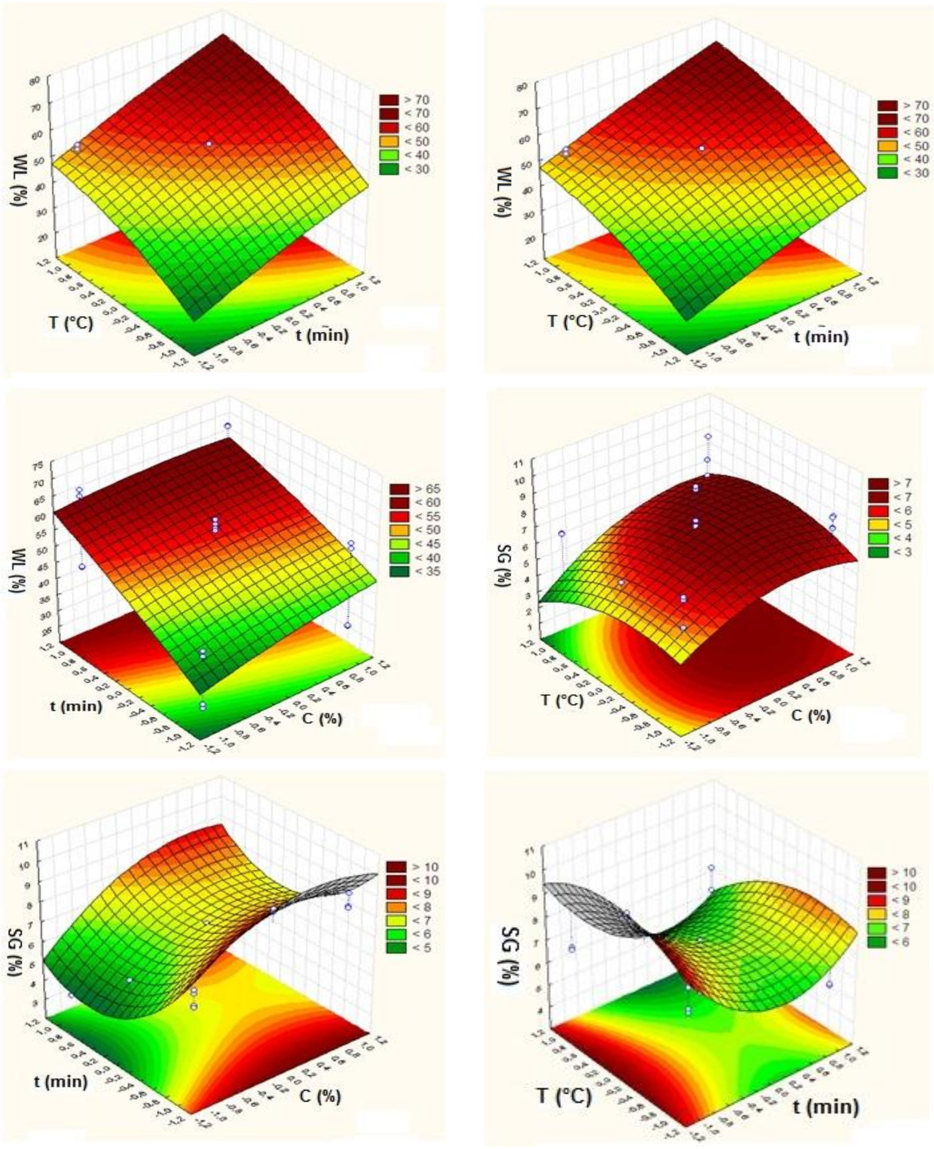


Fig. 2: Response surface plots of WL and SG in salt solution

3.4 Sorption isotherm of tomatoes slices at 25 and 40 °C

The experimental moisture sorption data for tomato slices at 25 and 40 °C are shown in figure 3.

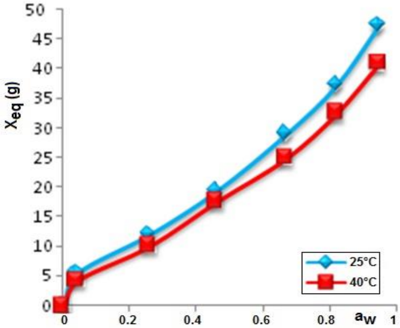


Fig. 3: Moisture adsorption isotherm of the tomatoes slices at 25 and 40°C

The sorption isotherms demonstrate an increase in equilibrium moisture content with increasing water activity, at constant temperature, and are sigmoid in shape for the two examined temperatures, characteristic of amorphous materials rich in hydrophilic components [12, 13]. 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 5**.

In this study it was found that for tomatoes slices the monolayer moisture level, determined by GAB equation, at 25°C and 40°C were respectively 17.07% and 16.66%. This moisture level should allows a long storage of tomatoes slices pretreated by osmotic dehydration [14], whose their appearance is presented in figure 4.

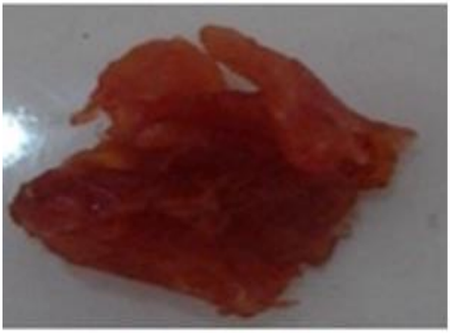


Fig. 4: Aspect of tomatoes slices dehydrated by osmosis

Table 5: Estimated GAB parameters for tomatoes slices dehydrated by osmosis

Temperature	X_m	C_0	K_0	ΔH_c	ΔH_k	R^2	RMS(%)
25°C	17.07	8.006	0.77	17.17	-229.51	0.99	2.62 %
40°C	16.66	7.49	069	13.54	-130.77	0.99	3.26%

Osmotic dehydration of tomatoes slices in salt and sucrose solution decrease slightly the lycopene content from 4.36 to 2.96 mg/100 g.

3.5 Calculation of the sorption heat

In order to determine the energy required for evaporation of water from tomatoes 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 shown that the heat of desorption of the mono-molecular layer (H_m = 16222 kJ/kg) is more important than that of the multilayer (H_q = 15111 kJ/kg). For example the energy necessary to evaporate the water from 1 kg of tomatoes slices pre-treated by osmotic dehydration can be evaluated using the following formulas: $Q = H_q \times m_e$, where H_q =heat desorption of the multilayer; m_e = amount of water to evaporate, which is calculated accordingto the following formula:

$$m_e = m_i \times \frac{m_{ci} - m_{cf}}{100 - m_{cf}} \tag{12}$$

Where m_i, mass of product = 1 kg; m_{ci}, rate of initial humidity = 29.56%, m_{cf}, rate of final humidity = 16.21%.Either, m_e = 0.159 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: $Q = 0.111\ \text{kWh}$.

4. CONCLUSIONS

In this work, the response surface methodology was used to investigate the optimum operating conditions that give maximum water loss and minimum solid gain during osmotic dehydration of tomatoes slices in sucrose and salt solutions. Analysis of variance has shown that the effects of the temperature, time, salt and sucrose concentration solution were statistically significant. Second order polynomial models were obtained for predicting water loss and solid gain.

The optimum conditions for maximum WL% and SG% are for sucrose solution: T =55°C, t =240 min and C =74% and for the salt solution T =55°C, t =240 min and C =5 %.

At these conditions the values of water loss and solid gain are respectively for the sucrose and the salt: 86.34 and 15.48 (g/100 g initial sample) then 65.43 and 3.30 (g/100 g initial sample).The value of water content of the monomolecular layer (X_m) was found to be 17.07% at 25°C.

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