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Optimization of Osmotic Dehydration of Apples Slices in Dates Syrup Using the Response Surface Methodology

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ABSTRACT

Osmotic dehydration of apple slices in continuous kinetic was studied using dates syrup at different concentration (52-74%), temperature (30-60°C), time (15-240 min). The response surface methodology was used to optimize effects of temperature, syrup concentration and immersion time in osmotic dehydration of apples slices in date syrup. A composite central design was used with water loss (WL%) and solid gain (SG %) as responses. The models obtained for all the responses were significant ($p<0.05$). The optimal conditions for maximum water loss and solid gains (75.52 and 17.92%) correspond to 60°C for a concentration of 74% (w/w) during 240 min.

Key words: Syrup, dates, apples, osmotic dehydration, optimization, RSM

INTRODUCTION

Date production in Algeria occupies a first place ranking in the Saharan agriculture, mainly by economic interest. The Algerian palm has a total area of 170,000 hectares which represents 18.7 million palm trees. Algeria, because of its more than 18 million palm trees and 800 varieties, occupies an important place among the producers and exporters of dates in the world and occupies the first place in terms of quality, through the famous variety "Deglet Nour." Production of dates in Algeria has increased to 8.5 million quintals in 2013, against 7.8 million in 2011 and 6.5 million in 2010. The main byproducts dates are pasta, flour, syrup, vinegar, yeast, alcohol and confectionery. Osmotic dehydration is an immersion of fruits or vegetables pieces in concentrated solutions. Immersion of food pieces in concentrated solutions of sucrose or other sugars promotes the removal of the water whose the percentage of loss can reach 70% without change of phase and limit the entry of the solute in the product. Several studies have reported the importance of osmotic dehydration as a pretreatment of fruit and vegetable pieces in solutions of sucrose and glucose syrup before final convective drying (Azoubel and Murr, 2004; Kowalska *et al.*, 2008; Garcia-Segovia *et al.*, 2010; Bchir *et al.*, 2011). On the other way solutes of higher molecular weight were selected carefully for osmotic dehydration to ensure the higher rate of water removal with little uptake of solute (Saurel *et al.*, 1994; Kuntz, 1995). Among different types of solutions, sucrose was ideal as osmotic agents for OD of fruits. Response surface methodology has been used by several investigators for optimizing food process operations (Mudahar *et al.*, 1990; Vijayanand *et al.*, 1995; Ravindra and Chattopadhyay, 2000). To our knowledge, no study has been conducted on the osmotic dehydration of fruits using date syrup. So the purpose of this study is the use of date syrup as an osmotic agent for the dehydration of apple slices optimized by response surface methodology.

MATERIALS AND METHODS

Vegetable materiel: Figure 1 shows the ripening of dates on tree of Timjouhart variety. We noted that the unripe dates are green. During ripening the tannin disappears and the color becomes red then dark and the flesh becomes soft, syrupy and translucent.

Determination of physical characteristics of dates: The evolution of color of dates during maturation was observed visually. Consistency of dates was evaluated calculating quality index using Eq. 1:

$$r = \frac{\text{Total sugar}}{\text{Water content}} \qquad (1)$$

Determination of chemical characteristics of dates and of dates syrup: Content water was determined using an infrared humidmeter. The dosing of sugars was carried out using the method of Lane-Eynon (Guiraud, 1982). Water activity (a_w) was determined by an activimeter. Purity index of syrup was evaluated by the Eq. 2 proposed by Mathlouthi and Reiser (1995):

$$P(\%) = \frac{\text{Total sugar}}{\text{Total soluble solids}} \times 100 \qquad (2)$$

Preparation of apples samples: The apples were purchased from a local market. The initial moisture content of fresh apples was 82%. The fruits were washed, peeled, pitted and cut into slices of 4.5 cm diameter and 0.5 cm thick. Immediately after cutting, the apple pieces are immersed in a ascorbic acid solution at 2 g L^{-1}.

Preparation of syrup date: Soft dates of Timjouhart variety were used to prepare the syrup. The percentage of dates in water was fixed at 25%. The first extraction was performed at 80°C for 12 h. After filtration, the juice was recovered and the residue undergoes a second extraction at 80°C for 8 h. After another filtration the obtained date juice at 12° Brix was decolorized using activated carbon (4%) and was concentrated to 70° Brix.

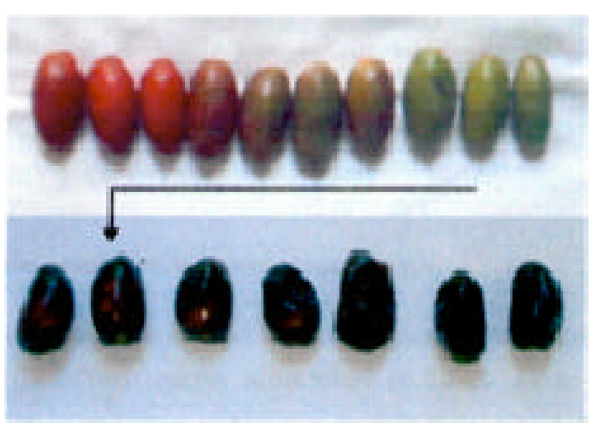

Fig. 1: Dates of Timjouhart variety at different states of maturity

Osmotic dehydration: Apples slices were osmotically dehydrated in dates syrup at different temperatures (30, 45 and 60°C), concentrations (52, 67 and 74%) and times (15, 90 and 240 min). Osmotic dehydration of slices apple in sucrose solution was carried out only for a comparison with the date's syrup. The ratio of apple slices to solution was 1:20. Samples were withdrawn at periodic intervals during 6 h. Excess solution from the surface was blot dried using paper towels. In the continuous method, each sample was weighed and returned to the osmotic solution to continue the drying process. After 6 h the moisture content of the sample was determined in a infrared moisture meter, each experimental treatment was performed in triplicate runs. Water loss and solid gain during osmotic dehydration were calculated using equations proposed by Azuara *et al.* (1998). Mass loss during osmotic dehydration is equal to water lost minus solids gained at the same time:

$$\text{WFL} = \frac{S_1 t \text{WFL}\infty}{1+S_1 t} \tag{3}$$

$$\text{SG} = \frac{S_2 t \text{SG}\infty}{1+S_2 t} \tag{4}$$

Experimental design: Osmotic dehydration of apple slices in syrup dates and sucrose solutions was studied in order to optimize the water loss and the solid gain. The chosen factors are the concentration, the time and the temperature. The Central Composite Design (CCD) allows us to conduct the study at three levels for each factor. The number of trials was set at seventeen (number of replication = 04). Equation of 2nd degree polynomial used to study the response surface is as following:

$$Y = b_0+b_1X^1+b_2X^2+b_3X^3+b_{11}X_1^2+b_{22}X_2^2+b_{33}X_3^2+b_{12}X_1X_2+b_{13}X_1X_3+b_{23}X_2X_3 \tag{5}$$

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 and X_3 are coded independent variables. The number of unknown is 10. Analysis was conducted using statistica V.8. The quality of the fitted model was evaluated by the analysis of variance (ANOVA). 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. Experimental design with the factors and responses for dates syrup and sucrose is presented in Table 2.

RESULTS AND DISCUSSION

Physico-chemical characteristics of dates of Timjouhart variety: The chemical composition of dates with some physical proprieties is given in Table 3. Results shows that the percentage of kernels is low and the value of r (total sugars/water content) reveals that the dates of Timjouhart variety are semisoft. Evaluation of r is an important criterion for the selection of varieties of dates destined for the preparation of syrup because the diffusion of sugars during extraction is high for soft and semisoft dates. Reducer's sugar content of flesh of dates was evaluated at 83.55%.

Yield of dates syrup: It is noted that 100 kg of date's pulp treated in 200 L of water gives 479 L (20:1) of juice at 12° Brix having a density of 1.054. Consequently 479 liters or 504 kg of

Table 1: Coded and uncoded levels of different process variables used in CCD

		Levels	
Independent variables	Symbol	Coded	Uncoded
Temperature (°C)	T	-1	25
		0	40
		+1	60
Concentration (%)			
	C	-1	52
		0	67
		+1	74
Time (min)	t	-1	15
		0	90
		+1	240

Table 2: Experimental data for water loss (WL) and solids gain (SG) under different treatment conditions of temperature, sugar concentration and time

				Dates syrup (%)		Sucrose (%)	
Run	T (°C)	C %(w/w)	t (min)	WL	SG	WL	SG
1	0	1	-1	20.23	8.53	17.22	2.22
2	0	0	0	49.66	8.71	41.22	7.56
3	0	1	1	75.52	17.92	70.22	16.00
4	0	0	0	49.66	8.71	41.22	7.56
5	1	-1	0	34.55	7.71	32.55	7.11
6	-1	0	-1	11.33	3.88	11.22	2.78
7	-1	0	1	57.36	10.10	51.51	13.87
8	-1	-1	0	27.33	7.71	24.78	5.13
9	0	-1	-1	13.36	3.53	11.33	3.11
10	-1	1	0	35.87	13.87	32.33	10.11
11	1	0	-1	18.33	5.78	16.33	6.22
12	1	1	0	38.55	9.22	36.36	8.81
13	1	0	1	61.22	12.67	59.33	12.78
14	0	-1	1	55.57	11.11	49.55	7.56
15	0	0	0	49.66	8.71	41.22	7.56
16	0	0	0	49.66	8.71	41.22	7.56
17	0	0	0	49.66	8.71	41.22	7.56

Table 3: Physico-chemical characteristics of syrup dates

Constituents	Values
Moisture (%)	31
Sucrose (%)	0.70
Reducer sugars (%)	83.55
r	2.7
Weight of one date (g)	12.86± 2.56
Kernels (%)	11

date's juice contains approximately 60 kg of soluble solids substances. In order to produce 100 kg of concentrated date's juice at 66° Brix 110 kg of dates are required.

Table 4: Physico-chemical characteristics of Timjouhart variety dates

Constituents	Values
Degrees Brix (%)	66
pH	4.07
Density	1.15
Reducers sugars (%)	63.95
Sucrose (%)	0.24
Water activity	0.72
Purity (%)	97.3

Physico-chemical characteristics of syrup dates prepared from timjouhart variety: The physico-chemical proprieties of syrup dates are presented in Table 4. The dates syrup contains 64.20% of total sugars with 63.95% for reducing sugars and 0.24% for sucrose . The value of purity percentage (97.3%) shows that after clarification the rate of recovering of total sugars is high. Water activity of 0.72 does not allows the proliferation of yeast such *Debaryamyces* which have a important multiplication in fruits juice for high water activity (aw >0.80).

Mass transfer during osmotic dehydration: The experimental values presented in Table 2 show that the water loss was higher than the solid gain. These results are in agreement with those reported by other researchers (Ravindra and Chattopadhyay, 2000; Saurel *et al*., 1994). We noted that the water loss and solid gain both were higher for samples treated in syrup dates compared to those treated in sucrose solution at 45°C during 240 min. This result is explained by the rapid penetration of monosaccharide's date syrup (fructose and glucose) into apples slices. The uptake of fructose and sucrose improve excellently the quality of final product. Sucrose, fructose and glucose solutions were considered as excellent for osmotic dehydration of fruits (Lenart and Lewicki, 1987; Lazarides *et al*., 1995).

Response surface analysis

Fitting model: The seventeen 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, from 3.81 to 22.64 (g/100 g on dry weight) was observed for different experimental combinations. The experiments were conducted in accordance with the Central Composite Design (CCD) to find the optimal combination of temperature, concentration and time for maximum water loss and minimum solid gains . The results of analysis of variance carried out to estimate the quality of the fitted second order response surface model are shown in Table 5. The Model F-value of 61.36 reveals that the model is significant. Values of "Prob > F" less than 0.0500 indicate that model terms are significant. In this study, the significant model terms are T, C, t, T^2, C^2, t^2 and C×t for Water Loss (WL) and only C and t for Solid Gain (SG). The values of R^2 and adjusted R^2 which are, respectively 0.967 and 0.969 for WL and 0.925 and 0.864 for SG shown that the descriptive ability of the model is good. The value of predicted R^2 being of 0.949 and 0.945, respectively for WL and SG indicate that the predictive ability of the model is satisfactory. Figure 2 confirms the good agreement between the experimental and predicted values for WL and SG.

Significance of regression: This test require for the error term to be normally and independently distributed with mean zero and given variance. This assumption is satisfied if the residuals are plotted approximately along a straight line and which is checked using a normal probability plot of residuals. Indeed we observed that the residuals are plotted approximately along a straight line

(Fig. 3). Therefore, the residuals are considered as normally distributed and the normality assumptions for both of the responses WL and SG are satisfied. The values of t-statistics of all independents variables presented in table are higher than the critical t-value ($t_{0.05,\,7}$ = 1.894). This result allows us to conclude that the independent variables the temperature (T), concentration (C) and the time (t), all contribute significantly to the model.

Significance of individual regression coefficient: 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). The test called t-statistics was applied to determine the coefficients which should be deleted in order to improve the quality of model . In this study we noted that the coefficients having a significant t-statistics (Table 6) are: b_0, b_1, b_2, b_3, b_{11}, b_{22}, b_{33} and b_{23} for WL and t, C and C^2 for SG.

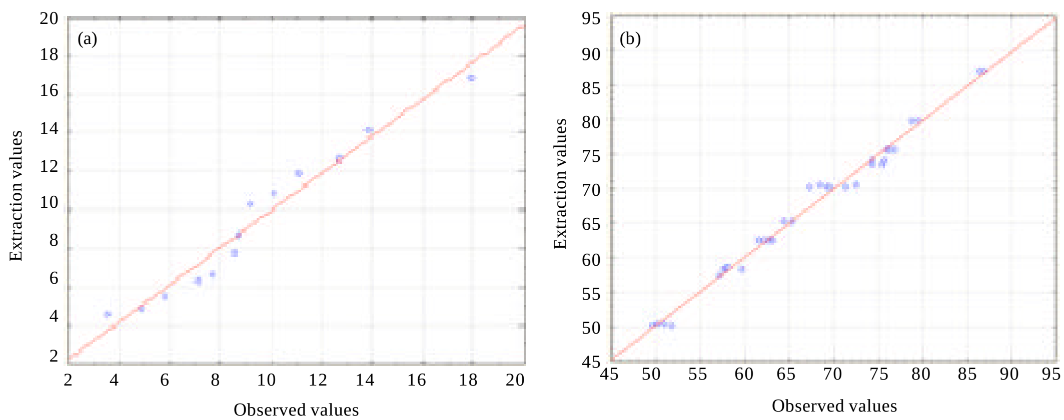

Fig. 2(a-b): Predicted vs. observed values for extraction WL and SG%

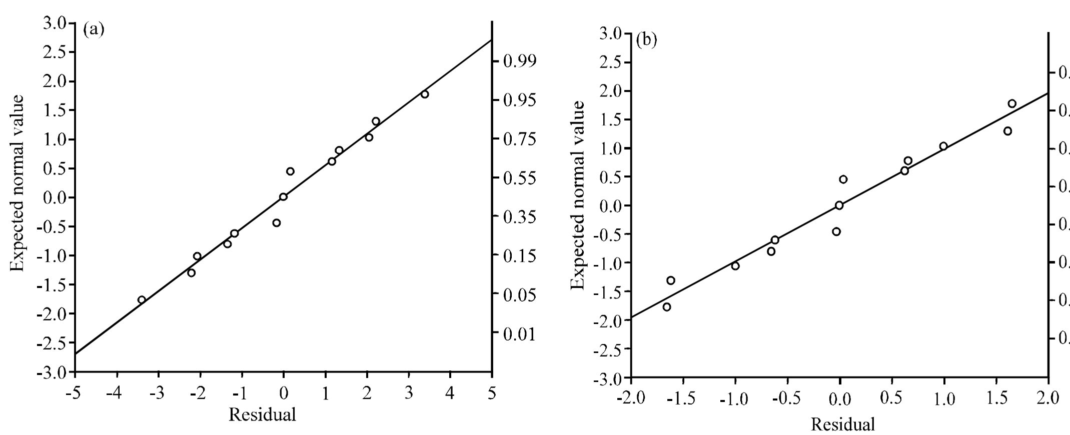

Fig. 3(a-b): Normal probability plot of residuals for WL and SG

Table 5: Analysis of variance of process variables as linear, quadratic and interactive terms on response variables

		WL			SG		
Source	df	Mean of squares	F	P	Mean of squares	F	P
Model	9	585.66	117.78	0.000001	178.14	9.620	0.0003
T	1	34.114	5.024	0.059945	0.0040	0.0038	0.9522
T^2	1	453.44	66.78	0.000080	1.6381	1.5590	0.2519
C	1	194.73	28.68	0.001058	65.723	62.550	0.000098
C^2	1	112.97	16.63	0.004695	24.328	23.153	0.001940
t	1	4143.68	610.28	0.000000	88.644	84.365	0.000037
t^2	1	46.131	6.79	0.035088	2.9798	2.8359	0.13605
T×C	1	4.906	0.722	0.423412	0.3600	0.3426	0.57669
t×T	1	14.02	2.065	0.193808	1.9321	1.8388	0.21720
C×t	1	42.77	6.299	0.040404	0.8190	0.7794	0.40659
Residue	7	6.790			1.0507		
R^2		0.967			0.925		
Adj R^2		0.969			0.864		
Pred R^2		0.949			0.945		

Table 6: Values of the second-order polynomial regression coefficients

		WL				SG			
variables	Coefficients	Parameters	Std. err	t	p	Parameters	Std. err	t	p
	b_0	49.66000	0.997240	49.79745	0.000000	8.710000	0.458416	19.00022	0.000000
T	b_1	2.59500	0.788387	3.29153	0.013275	-0.022500	0.362409	-0.06208	0.952231
T^2	b_2	-9.84750	1.086717	-9.06170	0.000041	-0.623750	0.499547	-1.24863	0.251939
C	b_3	4.92000	0.788387	6.24059	0.000428	2.866250	0.362409	7.90887	0.000098
C^2	b_{11}	-5.73750	1.086717	-5.27966	0.001148	2.403750	0.499547	4.81186	0.001940
t	b_{22}	23.30250	0.788387	29.55717	0.000000	3.328750	0.362409	9.18505	0.000037
t^2	b_{33}	-2.75250	1.086717	-2.53286	0.039069	-0.841250	0.499547	-1.68403	0.136050
T× C	b_{12}	-1.13500	1.114948	-1.01798	0.342572	-0.300000	0.512524	-0.58534	0.576695
t× T	b_{13}	-0.78500	1.114948	-0.70407	0.504131	-0.695000	0.512524	-1.35603	0.217203
t× C	b_{23}	3.27000	1.114948	2.93287	0.021934	0.452500	0.512524	0.88288	0.406595

The final estimated regression model using the coded variables is expressed as follows:

$$WL = 49,\ 66\text{-}2,\ 59\times T\text{-}9,\ 847\times C+4,\ 92\times t\text{-}5,\ 73\times T^2+23,\ 30\times C^2\text{-}2,\ 752\times t^2+3,\ 27\times C\times t \quad (6)$$

$$SG = 8,\ 71+2,\ 866\times C+3,\ 362\times t\text{-}5,\ 27966\times C^2 \quad (7)$$

Response surfaces plots: Results of analysis of variance (Table 5) have revealed that for the Water Loss (WL%) the linear term of immersion time (t), concentration (C), temperature (T) had a significant effect, followed by the quadratic term of temperature (T^2), concentration and (C^2) the interaction between time and concentration (t×C). However for Solids Gain (SG%) we noted a significant effect only for the linear term of concentration (C) and time (t) followed by the quadratic term of concentration (C^2).

Effect of variables on water loss: Figure 4a-c shows that the water loss increased with increase in the immersion time, the temperature and the solution concentration. The Fig. 4b

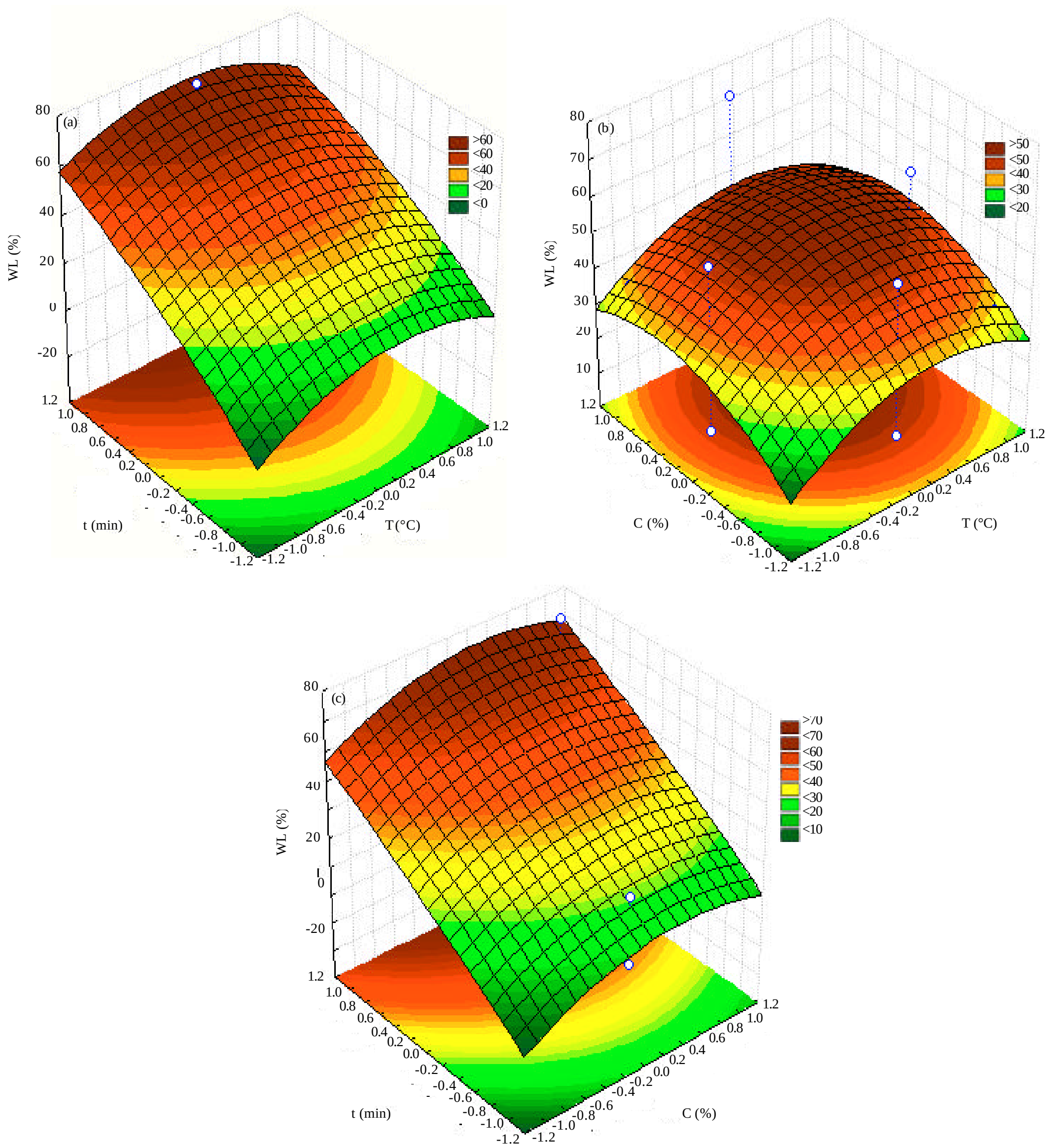

Fig. 4(a-c): Response surface and contour plots showing effect of processing variables on the WL, (a) Time vs. temperature, (b) Date syrup concentration vs. temperature and (c) Time vs. date syrup concentration

shows that the water loss (WL%) increased with the increase of temperature then decreased, indicating the existence of optimal temperature.

Effect of variables on solute gain: Figure 5a-c shows that the solute gain increases with increase in immersion time and concentration however, we noted a low effect of temperature on the solids gain.

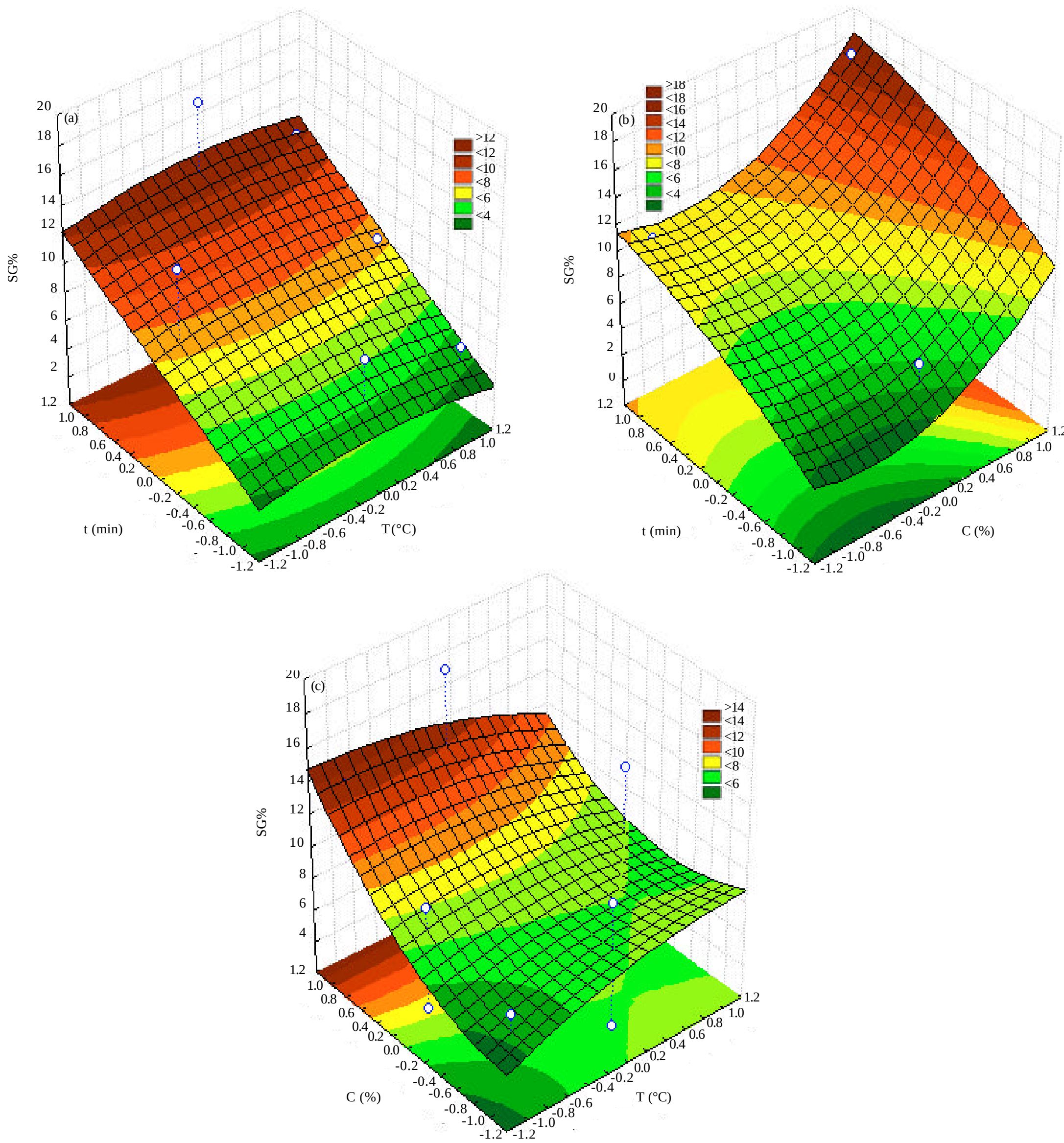

Fig. 5(a-c): Response surface and contour plots showing effect of processing variables on the SG, (a) Time vs. temperature, (b) Date syrup concentration vs. temperature and (c) Time vs. date syrup concentration

CONCLUSION

This study has shown that the "response surface methodology" is a effective way to determine the optimal conditions for water loss and solid gain during osmotic dehydration. Analysis of variance has shown that the effects of all the process variables including temperature, concentration and immersion time were statistically significant. Polynomial model was obtained for predicting WL and SG%. The optimal conditions for maximum water loss and solid gains (75.52 and 17.92%) correspond to 60°C for a concentration of 74° Brix during 240 min.

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