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Response Surface Methodology of Osmotic Dehydration for Sapota Slices

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Abstract

Osmotic dehydration is a widely accepted pre-treatment method of partial removal of water by submersing fruits in sugar/salt solution. The goal of this work was to optimize the process parameters during osmotic dehydration of sapota. Water and sugar transfer were quantitatively investigated during osmotic dehydration of sapota slices using response surface methodology with the temperature (40–60 ^{o}C), processing time (120 - 240 min), sugar concentrations (40–60 ^{o}B) and solution-to-sample to ratio was constant (5:1) being the independent process variables. Quadratic regression equations describing the effects of independent process variables on the water loss (WL), solid gain (SG) and water reduction were developed. It was found that concentration of sugar solution and temperature were the most significant factors affecting the water loss and sugar gain during osmotic dehydration of beet root followed by temperature. Effect of temperature and time were more pronounced for SG than the concentration of sugar solution. The osmotic dehydration process was optimized for water loss and sugar gain. The optimum conditions were found to be: temperature 47.36 ^{o}C; immersion time 167.85 min; sugar concentration 53.53 ^{o}B. At these optimum values, water loss and solid gain were found to be 27.72% and 8.25%. Conformance of experimental results with the empirical model was evaluated using correlation coefficient (R) which was found for the proposed model as, R = 0.997 for water loss and sugar gain, respectively.

Keywords: Sapota, Modeling, Optimization, Osmotic dehydration, Response surfacemethodology.

1. Introduction

Osmotic dehydration involves the immersion of food material in highly concentrated osmotic solution which, in turn imposes an osmotic pressure gradient to withdraw excess water from the material, thereby reducing its water activity. Osmotic dehydration is characterized by the sugar gain and water loss for both quantitative modeling and knowledge of the kinetics of mass transfer while, the quality of osmotic dehydration are evaluated as the percentage of water loss and sugar gain (Chenlo*et al.*, 2007).

Sapota (*Achrassapota*L.) commonly known as chiku, is mainly cultivated in India as a table fruit, while in South-East Mexico, Guatemala and other countries it is commercially grown for the production of chuckle, which is a gum like substance obtained from latex of this fruit and it is mainly used for preparation of chewing gum. Sapota contains high fiber content (2.60g/100g) and hence the dried powder can be consumed as a fiber supplement. The fruit is rich in sugar, dietary fibre, carotene, known for its antioxidant properties and laxative substances, possessing various nutraceutical properties. Thus, sapota powder can be considered as a complete food rich in vitamins, carbohydrates, fibers and other bioactive compounds (Ganjyal*et al.*, 2003 and Ganjyal *et al.*, 2005). Dehydration is an important operation for preserving sapota. The moisture content of sapota was reduced from 76 % to 10-15% (w.b.) using solar drying within 76 h with time savings of 27 % over sun drying. Ganjyal et al. (2003) studied convective drying (15 to 35 h) followed by vacuum drying (14 to 31 h) in which the moisture content of sapota was reduced from 72-78 % (w.b.) to 8.5 to 12.5 % (w.b.)

Response Surface Methodology (RSM) is an important tool in process optimization and product quality improvement. RSM is a collection of experimental design and optimization techniques that enables the researcher to determine the relationship between the response and the independent variables. RSM is typically used for mapping a response surface over a particular region of interest, optimizing the response or for selecting operating conditions to achieve target specifications. A lot of work has been done in the area of osmotic drying of fruit and vegetable but a very limited amount of work has been done in the area of osmotic dehydration of sapota.The objectives of the present study are to determine the effect of process parameters on sugar gain and water loss during osmotic dehydration of sapota and to optimize these parameters using RSM.

2. Materials and Methods

The methodology involved osmotic dehydration with different syrup concentration, determination of water loss and sugar gain, and optimization of response parameters with RSM.

2.1 Sample Preparation and Experimental Method

The fresh sapota fruits were procured from the Department of Horticulture, Dr. Panjabrao Deshmukh Krishi Vidyapeeth, Akola, India. They were sorted visually for uniform maturity and size; then were washed with tap water and surface dried with a filter paper. The average initial moisture content was determined by using oven method at 70 °C for 18 h (AOAC, 1990).

Osmotic dehydration was done in sugar solutionwith different concentrations such as 40, 50 and 60 °B. The concentration of the solution was by measured using hand refractometer at room temperature. The sample/solution ratio was constant 1:5 (w/w) to limit the decrease of ratio of sample to solution. It did not allow significant dilution of the medium from the release of water and subsequent decrease of the osmotic driving force during the process. The sapota slices were weighed and submerged in the sugar solution at 40, 50 and 60^0C. The temperature was maintained constant using hot water temperature bath. The samples were removed from the solution at different time intervals (60, 150 and 240 min). After removing from the sugar syrup, the samples were drained and the excess solution at the surface was removed with filter paper for subsequent weight measurement (Tiwari and Jalali 2004).

2.2 Sugar Gain and Water Loss

The water loss and sugar gain were calculated and given in Eq (1) (Kaleemullah*et al.*, (2002):

$$WL = \frac{W_{si} X_{swi} - W_{s\theta} X_{sw\theta}}{W_{si}} \times 100 \quad (1)$$

$$SG = \frac{W_{s\theta}(1 - X_{sw\theta}) - W_{si}(1 - X_{swi})}{W_{si}} \times 100 \quad (2)$$

where, WL = water loss (g water per 100 g initial mass of sample), SG = sugar gain (g solids per 100 g initial mass of sample). W_{si}= initial mass of sample, g, $W_{s\theta}$= mass of the sample after time θ, g, X_{swi} = water content as a fraction of the initial mass of the sample and $X_{sw\theta}$ = water content as a fraction of the syrup at time θ.

2.3 Design of Experiments

The optimized processing parameters of osmotic dehydration were considered for analysis and the maximized water loss and targeted sugar gain were determined by using RSM. The Box- Behnken Design (BBD) of three variables and the three levels including 17 trials formed by 5 central points were used (Box and Behnken, 1960) for designing the experiments of osmotic dehydration using three factors, viz., temperature, concentration and osmotic duration.Three levels of each variable(Table 1).

Table 1: Details of different treatments for osmotic dehydration.

Parameters	Notations	Process variables (Coded)		
		-1	**0**	**+1**
Concentration of syrup, ^{o}B	A	40	50	60
Temperature of solution, ^{o}C	B	40	50	60
Time of immersion, min.	C	60	150	240

The osmotic dehydration was assumed to be affected by three independent variables (regressor or factors), r_i, *viz.*, syrup temperature (A), syrup concentration (B) and duration of osmosis (C). The dependent variables referred as responses $Y_{k.}$ The independent variables (r_i), the coded variables (x_i), uncoded variables and their coded and uncoded levels are shown in Table 1. It is assumed that the mathematical function f_k (k = 1, 2, 3,...n), exists for each response variable, Y_k in terms of the processing factors, r_i (i = 1, 2, 3,...m), such as $Y_k = f_k(r_1, r_2, r_{3,} .... r_m)$

The exact mathematical representation of the function (*f*) is either unknown or extremely complex. However, second order polynomial equation (given in Eq. 3) of the following form was assumed to relate the response, $Y_k$and the factors, r_i

$$Y_k = \beta_{ko} + \sum_{i=1}^{i=3} \beta_{ki} x_i + \sum_{i=1}^{i=3} \beta_{kii} x_i^2 + \sum_{i=1}^{i=2} \sum_{j=i+1}^{j=3} \beta_{kij} x_i x_j \qquad (3)$$

where, $Y_k$is response (i. e. water loss or sugar gain) β_{ko} $\beta_{ki,}$ β_{kii} and β_{kij} are constant coefficients and x_i and x_J are the coded independent variables that are linearly related to A, B and C.

2.4 Optimization

During optimization of industrial processes, usually several response variables describing the quality characteristics and performance measurements of the systems, are to be maximized while some are to be minimized.One approach uses a constrained optimization procedure, the second is to superimpose the contour diagrams of the different response variables, and the third approach is to solve the problem of multiple responses through the use of a desirability function that combines all responses into one measurement. It is based on the idea that the “quality” of a product or process with complex characteristics is not acceptable, when one of its parameters is outside of “desired” limits. The method finds operating conditions ‘x’ that provide the “most desirable” response values. In the present study, desirability functions were developed for maximizing water loss and minimizing sugar gain.

3. Results and Discussion

3.1 Effect of variables on water loss and sugar gain

The effects of variation in water loss and sugar gain were studied by changing syrup temperature, syrup concentration and osmosis duration in experimental studies and it is presented in Table 2. A second order polynomial equation Eqn. (3) was fitted with the experimental data.

Table 2: Observed water loss and sugar gain under varying processing parameters.

S. No.	Syrup concentration, °B	Syrup temperature, °C	Duration of osmosis, min	Water loss, %	Sugar gain, %
1	40	40	180	22.8	6.36
2	60	40	180	30.1	9.26
3	40	60	180	26.16	8.02
4	60	60	180	35.56	11.7
5	40	50	120	19.6	6.36
6	60	50	120	26.88	9.56
7	40	50	240	25.15	7.51
8	60	50	240	34.41	10.87
9	50	40	120	20.82	6.2
10	50	60	120	25.61	8.51
11	50	40	240	27.41	7.51
12	50	60	240	32.08	9.69
13	50	50	150	27.59	8.02
14	50	50	180	27.59	8.02
15	50	50	180	27.59	8.02
16	50	50	180	27.59	8.02
17	50	50	180	27.59	7.95

This equation was obtained using step-down regression method, where factors with F-values less than one were rejected as described by Snedecor and Cochran (1967). The data for water loss and sugar gain were analysed by stepwise regression method and it is presented in Table 3. The quadratic model was fitted with the experimental data and the statistical significance for linear, quadratic and interaction terms were calculated for water loss and sugar gain as presented in Table 3. The R^2 value was calculated by least square technique and it was found to be 0.999 and 0.999, which shows a good fit of model with the data. The model F value of 391.5 and 103.9 implies that the model is significant ($P < 0.0001$). The linear terms (A, B & C) were significant ($P < 0.0001$). The lack of fit F value was non-significant which, indicated that the developed model was adequate for predicting the response. Moreover the predicted R^2 of 0.997 and 0.991 was in reasonable agreement with adjusted R^2 of 0.999 and 0.998 for water loss and sugar gain. This revealed that the non-significant terms have not been included in the model. Therefore, this model could be used in predict the design space.

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

Variables/ Factors	DF	F Values			
		WL, %		SG, %	
		SS	F Values	SS	F Values
Model	9	280.2589	3304.22**	36.33301	1039.9**
A-Concentration	1	138.1122	14654.92**	21.58245	5559.4**
B- Temperature	1	41.7698	4432.14**	9.223513	2375.9**
C-Duration	1	85.41245	9063.02**	3.062813	788.9**
AB	1	1.1025	116.98**	0.1521	39.2*
AC	1	0.9801	104.00**	0.0064	1.6*
BC	1	0.0036	0.38*	0.004225	1.1*
A2	1	1.318064	139.86**	2.160059	556.4**
B2	1	1.180506	125.26**	0.059375	15.3*
C2	1	10.9888	1166.01**	0.084007	21.6*
Lack of Fit	3	0.05445	6.30^NS^	0.019175	3.2^NS^
R^2		0.9997		0.9982	
Pred. R^2		0.9995		0.9982	
Adj R^2		0.9968		0.9912	
C.V, %		0.35		0.74	

*** Significant at 0.0001, ** Significant at 0.001, * Significant at 0.05, NS - Non significant

The high value of coefficient of determination (R^2 = 0.997, 0.999) obtained for response variable indicated that the developed model for water loss and sugar gain were highly significant. The result of analysis of variance indicated that the linear terms of syrup temperature, syrup concentration and duration of osmosis were highly significant at 1% level (Table 3). The presence of quadratic terms of syrup concentration and duration of osmosis indicated curvilinear nature of response surface. The quadratic terms of syrup concentration and duration of osmosis were also highly significant at 1% level while quadratic term of temperature was non-significant. The interaction terms of syrup temperature and duration of osmosis as well as syrup concentration and duration of osmosis were significant at 5% level.

The regression equation describing the effects of process variables on water loss and sugar gain in terms of actual values of variable is given in Eq. (4) and Eq. (5)

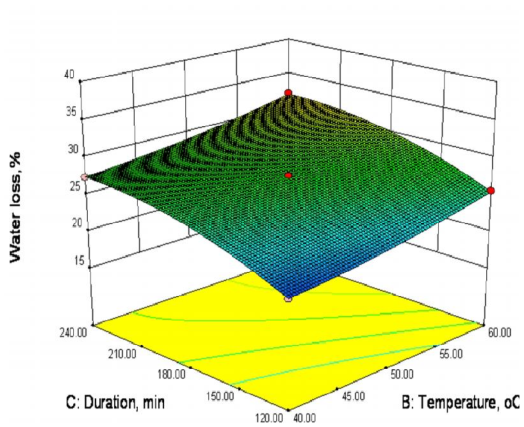
$$WL = 18.34-0.56C- 0.56T+0.17\theta+0.0053CT+0.00082C\theta-5x10^{-5}T\theta +0.0056C^2 +0.005T^2-0.00045\theta^2 \quad (4)$$

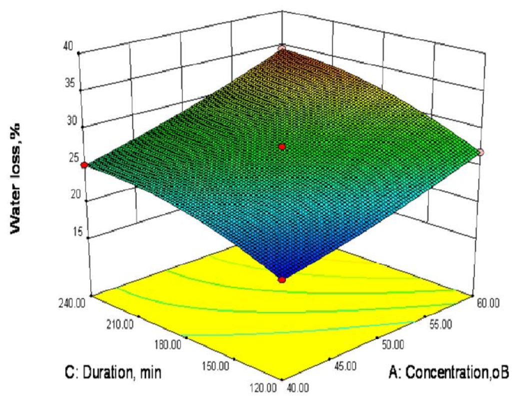
$$SG = 17.15-0.66C-0.099T+0.02\theta +0.002CT+6.67x10^{-5} C\theta - 5.4^{5}+0.007C2 +0.001T^{2}-3.9x10^{-5} \quad (5)$$

The linear positive terms indicated that water loss and sugar gain increased with increase in syrup temperature, syrup concentration and duration of osmosis. The presence of positive interaction terms between syrup temperature and duration of osmosis as well as syrup concentration and duration of osmosis indicated that increase in their levels increased water loss. The negative values of quadratic terms of syrup concentration and duration of osmosis indicated that higher values of these variables further reduced water loss and sugar gain.

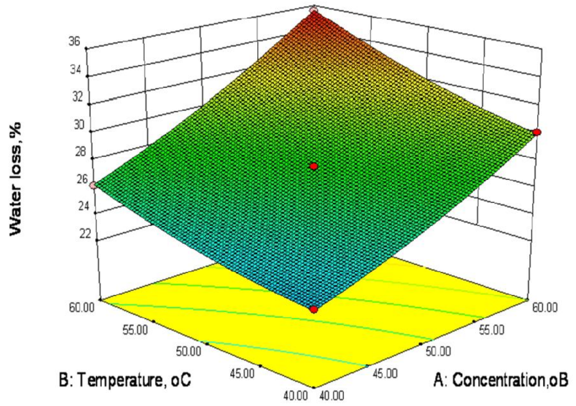
To visualize the combined effect of two variables on the water loss and sugar gain, the response surface and contour plots (Fig.1 A-C and Fig. 2 A- C) were generated for the fitted model as a function of two variables while keeping third variable at its central value. The water loss and sugar gain increased rapidly in the early stages of osmosis, after which the rate of water loss from sapota slices sample into sugar syrup and sugar gain from sugar syrup to sapota slices sample gradually slowed down with time. The rapid removal of water and sugar gain in the early stages of osmosis has been reported for carrots (Uddin *et al.,* 2004), etc.

The higher temperatures seem to accelerate water loss (Fig.1 A-B) through swelling and plasticizing of cell membranes as well as the better water transfer characteristics on the product surface due to lower viscosity of the osmotic medium (Uddin *et al.*, 2004). The water loss increased with concentration of syrup (Fig. 1 A and C) as well as with duration of osmosis (Fig 1 B and C) over the entire osmotic dehydration process. The similar findings have been reported for osmosis of other fruits (Sílvia 2010 and Pokharkar and Prasad, 1998).



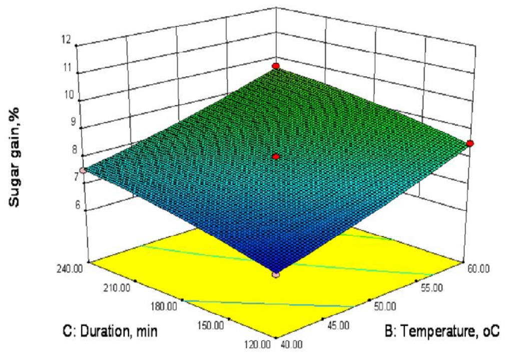


(A) At 50 °B concentration **(B) At 50 °C temperature**

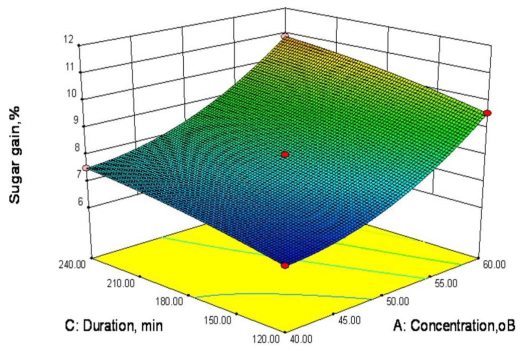


(c) At duration 120 min

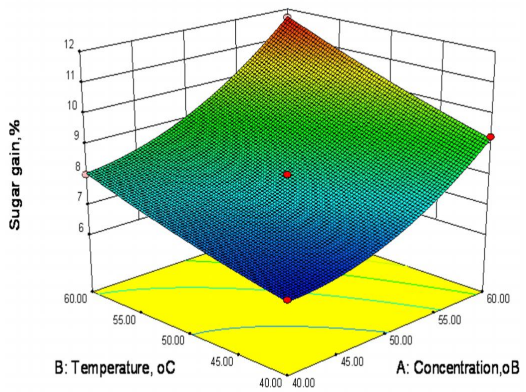
Fig. 1: The contour and response surface showing the effect of temperature, concentration and duration on water loss during osmotic dehydration of sapota slices.



(A)At 50 ^{o}B concentration



(B)At 50 ^{o}C temperature



(C)At 120 min duration

Fig. 2: The contour and response surface showing the effect of temperature, concentration and duration on sugar gain during osmotic dehydration of sapota slices.

The sugar gain increased with temperature rise (Fig. 2 A and Fig. 2B) of syrup. As explained for water loss, syrup temperature has an effect on the cell membrane permeability that could allow solute to enter by losing its selectivity. Increased concentration of the sugar syrup also led to increase in sugar gain (Fig. 2A and Fig. 2C) which might be due to increase of osmotic pressure gradient and consequent loss of functionality of cell plasmatic membrane that allows solute to enter. Similar results were found for pineapple (Sujata Jene, 2005), mango (Duduyemi Oladejo et al , 2013), cranberry (Shamaei et al, 2012).

It was observed from Fig.1 and Fig. 2 that the moisture loss as well as the sugar gain increased non-linearly with time at all concentrations. Both moisture loss and sugar gain were faster in the initial period of osmosis and then the rate decreased. This was because osmotic driving potential for moisture as well sugar transfer was kept on decreasing with time as the moisture keeps moving from sapota to solution and the sugar from solution to sapota. Progressive sugar uptake would result in the formation of high solid sub surface layer, which would interface with the concentration gradients across the sample solution interface and would set as barrier against removal of water and uptake of solid. Besides, rapid loss of water and uptake of solids near the surface in the beginning may result in structural changes leading to compaction of this surface layers and increased mass transfer resistance for water and solids. Similar trends have been reported for other fruits and vegetables during osmosis (Ghosh *et al.,* 2006).

3.2 Optimization of osmotic dehydration of Sapota slice

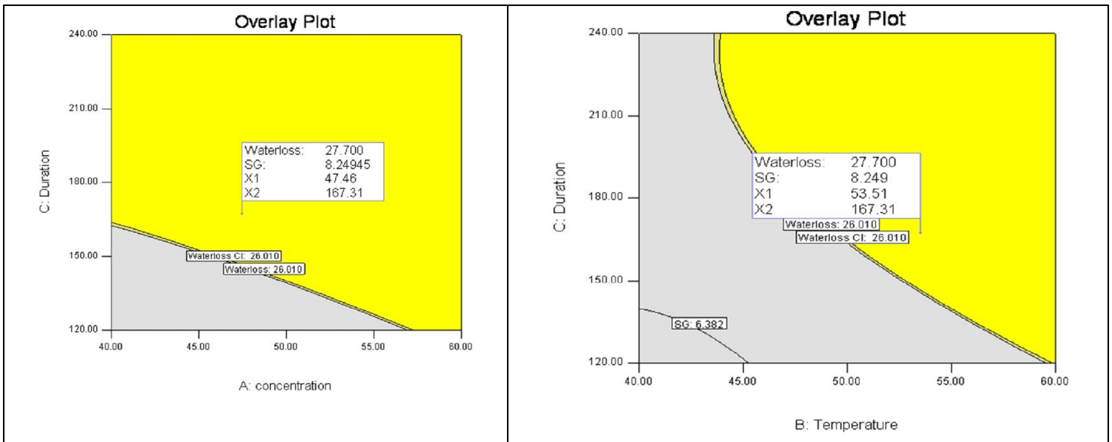
The constraints were set such that the selected variables (A, B and C) would be minimum from economical point of view for the most important product attribute and close to the optimum for the others (Jain *et al.*, 2011). The main criteria for constraints optimization were maximum possible water loss and targeted sugar gain of 8.25 percent as most important quality (sweetness) attribute. The desired goals for each factor and response are shown in Table 4. In order to optimize the process parameters for osmotic dehydration process by numerical optimization, which finds a point that maximizes the desirability function; equal importance of '3' was given to all the 3 process parameters and 2 responses. The goal setting begins at a random starting point and proceeds up the steepest slope on the response surface for a maximum value of water loss and targeted value of sugar gain.

Table 4: Optimization criteria for different process variables and responses for osmotic dehydration of sapota slice.

Parameter	Goal	Lower limit	Upper limit	Importance	Solutions obtained
Temperature, ^oC	Minimize	40	60	3	47.36
Concentration, ^{o}Brix	Minimize	40	60	3	53.53
Duration, min	Minimize	120	240	3	167.85
Water loss, percent	Maximize	19.6	35.56	3	27.72
Sugar gain, percent	target = 8.25	6.2	11.7	3	8.25

A graphical multi response optimization technique was adopted to determine the workable optimum conditions for the osmotic dehydration of sapota slices. The contour plots for all responses were superimposed and regions that satisfy all the constraints were selected as optimum conditions. The criteria for constraint optimization are given in Table 4. These constraints resulted in feasible zone of the optimum solutions (yellow coloured area in the superimposed contour plots). The superimposed contour plots having common superimposed area for all responses for osmotic dehydration of sapota are shown in Fig. 3.

The superimposed contours of all responses for A and B (Fig. 3 A) and A and C (Fig. 3 B) and their intersection zone for maximum water loss and targeted sugar gain (8.25%) indicated the range of optimum values of process variables.



(A) At temperature of osmosis 47.46%

B) At concentration of syrup = 53.51 ^{o}Brix

Fig. 3: Superimposed contours for water loss (%) and sugar gain (%) for osmotic dehydration of sapota slices at varying (A) concentration of syrup and temperature of syrup and (B) concentration of syrup and duration of osmosis.

3.3 Verification of the model for osmotic dehydration of sapota slices

Osmotic dehydration experiments were conducted at the optimum process conditions (x_1 = 49 ^{o}C, x_2 = 48 ^{o}B and x_3 = 140 min) for testing the adequacy of model equations for predicting the response values.

The observed experimental values (mean of 3 experiments) and values predicted by the equations of the model are presented in Table 5. The experimental values were found to be very close to the predicted values for water loss and sugar gain, with the value of C.V. as 9.37 percent and 1.57%, respectively. Therefore, it could be concluded from above discussion that model are quite adequate to assess the behaviour of the osmotic dehydration of sapota.

Table 5: Predicted and experimental values of response at optimum process conditions for osmotic dehydration of sapota

Response	Predicted Value	*Experimental value (±SD)	C.V., %
Water loss, %	27.72	27.53 ± 0.30	9.37
Sugar gain, %	8.24	8.36 ± 0.12	1.57

* Average of three replications

4. Conclusion

RSM was used to determine the optimum operating conditions that yield maximum water loss and minimum sugar gain in osmotic dehydration of sapota. Analysis of variance has shown that the effects of all the process variables including temperature, time, and sugar concentration were statistically significant. Second order polynomial models were obtained for predicting water loss, and solid gain. The optimal conditions for maximum water loss and minimum solid gain correspond to temperature of 47.36 ^{o}C, processing time of 167.85 min , sugar concentration of 53.53 ^{o}B and solution to sample ratio of 5:1w/w in order to obtain water loss of 27.72% sugar gain of 8.25%. Conformance of experimental results with the empirical model was evaluated using correlation coefficient (R) which was found for the proposed model as, R = 0.999 and 0.997 for water loss and sugar gain, respectively.

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