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Optimization of process parameters for osmotic dehydration of banana slices using response surface methodology

Deep P Patel, Kusum Meghwal, Maya Sharma and Sanjay Kumar Jain

Abstract
Syrup concentration (40, 50 and 60 °Brix), process temperature (35, 45 and 55 °C) and duration of osmosis (30, 60, 90, 120, 150, 180, 210, 240, 270 and 300 min) for osmotic dehydration of banana (Grand Naine) slices were optimized for the maximum water loss and optimum sugar gain by using response surface methodology. The ripe banana slices of 20 mm thickness were immersed in sugar syrup at constant temperature water bath having syrup to banana slices ratio of 5:1 (w/w). The slices were removed from bath at pre-decided time, rinsed with water and weighed. Initial moisture content of papaya samples were 75.5-78.5 per cent (wb), which was reduced to 61.1-68.02 per cent after osmotic dehydration in various experiments showing water loss and sugar gain were found in the range of 9.41-28.63 and 3.86-6.79 per cent, respectively. The water loss and sugar gain data were statistically analyzed and regression equations of second order were found the best fit for all the experimental data. Maximum water loss of 13.49 per cent with optimum sugar gain of 4.81 per cent was predicted for the 45 °Brix syrup concentration at 47 °C for syrup to fruit ratio as 5:1 in 210 min of osmotic dehydration.

Keywords: banana, osmotic dehydration, RSM, water loss, sugar gain

Introduction
Convective drying stabilizes the product by decreasing its water activity and moisture content, but the quality of the fruit and particularly their original flavor, texture and color are sensitive to heat treatment (Boudhrioua *et al.*, 2003) [5]. Freeze dehydration of fruit gives high quality food products but at the same time it attributes the high cost of processing (Gharsallaoui *et al.*, 2007) [8].
Osmotic dehydration is a partial dehydration process, often considered more as a treatment, to give the product a quality improvement over the conventional dehydration process. The osmotic treatment involves soaking of a food in hypertonic solution of sugar and/or salt for specific times under controlled temperature condition. The process involves two counter-current mass transfers, a loss of water from the food to the solution and the simultaneous migration of solids from solution to the food. Such mass transfer phenomena are governed by pretreatment, osmotic solution, product and osmotic environment related factors. The method has two major advantages when compared with other dehydration methods. The quality of osmotically dehydrated products is better and shrinkage is considerably lower as compared to products from conventional dehydration processes. Secondly, the technique helps to conserve the overall energy relative to other dehydration procedures (Bekele, 2010) [3]. An attempt was made to optimize the selected 3 process parameters for maximum water loss and optimum (targeted) sugar gain during the osmotic dehydration of banana slices.

Materials and Methods
A widely grown 'Grand Naine' variety of banana (*Musa spp.*) was selected for the osmotic dehydration experiments. Physiologically matured banana fruits were procured from local market. Ripened banana of uniform size, colour and firm texture were washed, peeled and cut in uniform thickness of 2 cm.

Osmotic dehydration of banana slices: The initial moisture content of the fresh, as well as osmotically dehydrated banana slices was determined using AOAC (2000) [2] method. The slices of 2 cm size were osmotically dehydrated using sugar syrup with syrup to sample ratio of 5:1. The beakers were placed inside the constant temperature water bath.

The syrup in the beakers was manually stirred at regular intervals to maintain uniform temperature. One beaker was removed from water bath at pre-decided time and the samples were immediately rinsed in flowing water and placed on tissue paper to remove surface moisture. The samples were weighed and their moisture contents determined.

The selection of the levels for processing parameters was made by preliminary experiments in which 5 levels of each process parameters *viz*., sugar concentration (40, 50, 60, 70, 80 °Brix), processing temperature (30, 40, 50, 60, 70 °C) and duration of osmosis (2, 4, 6, 8, 10 h) were studied as suggested by earlier research workers (Rosa and Giroux, 2001; Torreggiani and Bertolo, 2001; Nieto *et al.*, 2001; Ozen *et al.* 2002; Jain and Verma, 2003) [13, 15, 11, 12, 9] for various fruits. The results of preliminary experiments revealed the levels of process variables and the same are given in Table 1.

Determination of water loss and sugar gain: The overall mass transport data namely, weight reduction (WR), water loss (WL), sugar gain (SG) and soluble solid concentration were used to indicate the overall exchange of solute and water between banana slices and sugar syrup. The WL and SG were calculated using the following mass balance equations. The WL can be defined as the net loss of water from banana slices at time (θ) on the initial mass basis.

$$WL = \frac{W_i X_i - W_\theta X_\theta}{W_i} \times 100 \quad \ldots (1)$$

The sugar gain is the net uptake of sugar by the slices on an initial weight basis. It was computed using the following expression:

$$SG = \frac{W_\theta (1 - X_\theta) - W_i (1 - X_i)}{W_i} \times 100 \quad \ldots (2)$$

Where

WL = water loss (g per 100 g mass of the sample)

SG = sugar gain (g per 100 g mass of sample)

W_θ = mass of slices after time θ, g

W_i = initial mass of slices, g

X_θ = water content as a fraction of mass of slices at time θ, fraction

X_i = water content as a fraction of initial mass of slices, fraction

The Box- Behnken design of three variables and three levels including 17 experiments formed by 5 central points was used (Box and Behnken, 1960) [7]. The osmotic dehydration was assumed to be affected by three independent variables *viz.*, syrup temperature (T), concentration (C) and duration of osmosis (θ). The experimental design of independent parameters and layout are shown in Table 1 for these three levels and three variables under the response surface methodology (Jain *et al*., 2011) [10]. All these variables were closely controlled and accurately measured during experimentation. The dependent variables also referred to as responses, Y_k (*i.e.*, the per cent of water loss and sugar gain) were measured experimentally.

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

$$Y_k = f_k (r_1, r_2, r_3, \ldots. r_m)$$

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

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

Where, Y_k is response (i.e. water loss or sugar gain) β_{ko}, β_{ki}, β_{kii} and β_{kij} are constant coefficients and x_i, the coded independent variables are linearly related to T, C and θ. Therefore, the designs are given in terms of standardized coded variables (x_i), which in any particular application are linearly related to r_i by the following equations.

$$x_i = \frac{(r_i - \bar{r}_i)}{d_i} \quad \ldots (4)$$

Where

r_i = Actual value in original units,

$\bar{r}_i$ = Mean of high and low levels of r_i

d_i = Spacing between the low and high levels of r_i

In present study, n = 2 and m = 3 and hence Eqn. 4 can be written as

$$Y_k = f_k (T, C, \theta) \quad \ldots (5)$$

Where

T = Sugar syrup temperature, °C,

C = Sugar syrup concentration, °Brix,

θ = Osmotic dehydration duration, min

Therefore, Eqn. 3 taken the following form as,

$$Y_k = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \quad \ldots (6)$$

Table 1: Experimental layout for 3 variables and 3 levels response surface analysis

Batch No.	Syrup temperature (°C)		Sugar concentration (%)		Duration of osmosis (min)	
	X_1 (coded)	X_1 (uncoded)	X_2 (coded)	X_2 (uncoded)	X_3 (coded)	X_3 (uncoded)
1	-1	35	-1	40	0	180
2	1	55	-1	40	0	180
3	-1	35	1	60	0	180
4	1	55	1	60	0	180
5	-1	35	0	50	-1	60
6	1	55	0	50	-1	60
7	-1	35	0	50	1	300
8	1	55	0	50	1	300
9	0	45	-1	40	-1	60
10	0	45	1	60	-1	60
11	0	45	-1	40	1	300
12	0	45	1	60	1	300
13	0	45	0	50	0	180
14	0	45	0	50	0	180
15	0	45	0	50	0	180
16	0	45	0	50	0	180
17	0	45	0	50	0	180

Optimization of process variables: The response surface analysis involved fitting experimental values of WL/SG to general quadratic polynomial equation and subsequently optimizing the values with suitable optimization software (RSM). Design-Expert version 11.0 software was used to evaluate coefficients of the Eq. 6. The sum of squares for the models (regression) and total error were computed. The regression sums of squares were divided into 3 parts, namely, linear, quadratic and cross products as the terms appeared in Eq. 6. The significance of these sources of sum of squares was determined by computing the F-value and comparing with the tabulated value for respective degrees of freedom under particular probability level.

Determination of optimum condition of independent variables *viz*. syrup temperature, syrup concentration and duration of osmosis involved calculation of stationary points and exploration of the nature (shape) of the surface around these points. At the stationary point, the slope of response surface is zero in all directions (Box and Draper, 1959) [6].

Numerical optimization: Numerical optimization technique of the Design-Expert version 11.0 software was used for simultaneous optimization of the multiple responses. The desired goals for each factor and response were chosen. The possible goals are: maximize, minimize, target, within range, none (for responses only). All the independent factors (T, C and θ) were kept minimized from an economical point of view while the responses *viz*. water loss was kept maximized and sugar gain was kept targeted.

Graphical optimization: Graphical optimization technique of design-expert software was carried out for obtaining the desired attributes in dried product. For graphical optimization, super- imposition of contour plots for all responses was done with respect to process variables using Box Behnken model of design expert version 11.0 software. The superimposed contours of all responses for syrup temperature, syrup concentration and duration of osmosis and their intersection zone for maximum water loss and targeted sugar gain indicated the ranges of variables which were considered as the optimum range for best product in terms of responses.

Verification of optimum responses: The optimum responses were verified by conducting the osmotic dehydration experiment under optimum conditions. The responses such as water loss and sugar gain at optimum processing conditions were compared with the values which were predicted by the mathematical model.

Optimum level of SG: Dehydrated banana should have a typical taste, flavour, colour and texture. To test these organoleptic characteristics, sensory evaluation was done on the basis of numerical sensory card based on BIS: 6273 (Part III) - 1975. The osmo-dehydrated products were prepared having 1, 2, 3, 4, 5 and 6% SG. The product was served for the evaluation to 20 panelists in the age range of 18 to 45 years. The overall acceptability of the dehydrated product is mainly governed by the taste of the product, so it was used to obtain the desired level of SG in the osmo-dehydrated products. The SG in osmotic dehydrated banana slices were given to the consumer panel and paired “t test” was conducted by assuming null hypothesis (Ho) as there is no significant difference between the mean score due to variation in SG of 2 levels.

Result and Discussions

Optimum level of SG: During the preliminary experiments it was found that WL and SG during osmotic dehydration depend on temperature and concentration of syrup and duration of osmotic process. However, equilibrium could not be reached even after 300 min of osmotic dehydration. Although, the SG in samples increased with increase in temperature and concentration of syrup. Therefore, it can be inferred that sweetness of the product was enhanced at higher concentration of sugar syrup. For this reason it was felt necessary to optimize SG in the final product for better acceptability.

Table 2: Effect of sugar gain in osmo-dehydrated banana samples on mean sensory score

Product code	Sugar gain, %	Mean score	t-value	Remark given by consumer panel	Tabulated value at 5% level of significant
01	1	72.85	-	Good	$T_{0.05}$ (19) = 2.09
02	2	75.55	3.53*	Good	
03	3	76.65	1.97ns	Good	
04	4	77.85	1.68ns	Good	
05	5	81.50	6.13*	Excellent	
06	6	76.35	4.67*	Good	

ns: non-significant *significant at 5% level

To decide the optimum level of SG in the final product, the samples with varied SG levels were given to the consumers and they were asked to mention the quality grade scores as suggested in BIS (1971) [4]. The effect of SG in osmo-dehydrated banana slices on acceptability by the consumers is shown in Table 2. It can be observed from Table 2 that with increase in SG from 1 to 4 per cent, the t-value was non-significant i.e., there was no significant change in the taste of the product. When SG was increased from 5 to 6 per cent, there was significant change in the taste at 5 and 6 per cent level. Therefore, it was concluded that SG of 5 per cent was superior to 6 per cent, since the mean score of 5 per cent was 81.50, while the mean sensory score of 6 per cent SG was 76.35.

Optimization for WL: A second order polynomial equation (Eq. 6) was fitted with the experimental data presented in Table 3. Equation 8 gives the predicted WL % as a function of syrup temperature (x_1), concentration (x_2) and duration of osmosis (x_3) expressed in coded form. This equation was obtained using step-down regression method where factors with F-values less than one were rejected as suggested by Snedecor and Cochran (1967) [14]. High value of coefficient of determination (R^2=0.9974) obtained for response variable indicated that the developed model for WL accounted for and adequately explained 99.74% of the total variation.

$$WL = 12.37 + 4.30\,x_1 + 2.31\,x_2 + 5.51\,x_3 + 0.69\,x_1 x_2 + 2.24\,x_1 x_3 + 1.36\,x_2 x_3 + 1.37\,x_1^2 + 0.55\,x_2^2 - 1.24\,x_3^2 \quad \text{.........(7)}$$

(R^2 = 0.9974)

Replacing x1, x2 and x3 with (T-45)/10, (C-50)/10 and (θ–180)/120 respectively (Eq. 4) in Eq. 7, the water loss in real terms of syrup temperature, concentration and duration of osmosis can be given by

$$WL = 52.93 - 1.4842\,T - 0.8410\,C - 0.6356\,\theta + 0.0069\,T\,C + 0.00186\,T\,\theta + 0.00113\,C\,\theta + 0.0137\,T^2 + 0.00557\,C^2 - 0.000086\,\theta^2 \quad \text{........(8)}$$

The result of the analysis of variance of Eqn. 7 indicated that linear terms of syrup temperature, concentration and duration of osmosis were highly significant at 1% level of significance (Table 4).

The quadratic terms and the interaction terms of the temperature, concentration and duration were also significant. So, Eqn. 8 was considered as the final regression equation because there wasn't any non-significant term.

Table 3: Experimental data for water loss (WL) and sugar gain (SG) under different treatment conditions

Treatment No.	Coded value			Decoded value			Water Loss (%)	Sugar gain (%)
	Syrup temp., °C	Syrup conc. °Brix	Duration of osmosis, Min.	Syrup temp. °C	Syrup conc. °Brix	Duration of osmosis, Min		
	x_1	x_2	x_3	T	C	Θ		
1	-1	-1	0	35	40	180	8.22	3.06
2	1	-1	0	55	40	180	21.86	4.69
3	-1	1	0	35	60	180	11.36	4.15
4	1	1	0	55	60	180	21.76	5.37
5	-1	0	-1	35	50	60	5.04	1.23
6	1	0	-1	55	50	60	8.55	2.43
7	-1	0	1	35	50	300	10.31	4.23
8	1	0	1	55	50	300	24.67	5.83
9	0	-1	-1	45	40	60	5.26	1.72
10	0	1	-1	45	60	60	7.47	2.01
11	0	-1	1	45	40	300	12.18	4.87
12	0	1	1	45	60	300	17.43	6.36
13	0	0	0	45	50	180	12.01	4.67
14	0	0	0	45	50	180	11.88	4.44
15	0	0	0	45	50	180	12.03	4.74
16	0	0	0	45	50	180	11.68	4.25
17	0	0	0	45	50	180	12.11	4.59

Table 4: Analysis of variance for water loss during osmotic dehydration of banana slices

Source	Sum of squares	Df	Mean sum of squares	F value
Model	477.38	9	53.04	294.46*
T	147.66	1	147.66	1346.48
C	42.78	1	42.78	819.72*
Θ	242.55	1	242.55	237.49*
T C	1.90	1	1.90	10.57**
T θ	20.03	1	20.03	111.17*
C θ	7.40	1	7.40	41.07*
T^2	7.91	1	7.91	43.89*
C^2	1.31	1	1.31	7.27**
θ^2	6.52	1	6.52	36.22*
Residual	1.26	7	0.1801	
Lack of Fit	1.04	3	0.3477	6.38^{ns}
Pure Error	0.2179	4	0.0545	
Cor Total	478.65	16		
R-Squared	0.9974			
Adj R-Squared	0.9940			
Pred R-Squared	0.9644			
Std. Dev.	0.4244			
Mean	12.69			
C.V. %	3.34			

* Significant at 1% Level, ** Significant at 5% Level and ns - Non significant

The presence of quadratic terms of the variables in Eq. 8 indicated curvilinear nature of the response surface. It was observed that the syrup temperature contributed maximum for WL, followed by concentration of syrup and duration of osmosis. The presence of positive interaction terms between syrup temperature and concentration, temperature and duration of osmosis and concentration and duration of osmosis of Eqn. 8 indicated that an increase in their levels increased water loss. The negative coefficient for concentration, temperature and duration of osmosis indicated that increases in the level of these three variables decrease water loss.

Optimization for SG: A second order polynomial equation (Eq. 6) was fitted with the experimental data presented in Table 3. Equation 10 gives the predicted SG % as a function of syrup temperature (x_1), concentration (x_2) and duration of osmosis (x_3) expressed in coded form. This equation was obtained using step-down regression method where factors with F-values less than one were rejected as suggested by Snedecor and Cochran (1967) [14]. High value of coefficient of determination (R^2=0.9910) obtained for response variable indicated that the developed model for SG accounted for and adequately explained 99.10% of the total variation.

$$SG = 4.54 + 0.7063\,x_1 + 0.4438\,x_2 + 1.74\,x_3 - 0.1025\,x_1 x_2 + 0.1000\,x_1 x_3 + 0.3000\,x_2 x_3 - 0.2653\,x_1^2 + 0.0447\,x_2^2 - 0.8427\,x_3^2 \quad \text{…...(9)}$$

(R^2 = 0.9910)

Table 5: Analysis of variance for sugar gain during osmotic dehydration of banana slices

Source	Sum of Squares	Df	Mean sum of Squares	F value
Model	33.56	9	3.73	85.55*
T	3.99	1	3.99	554.19*
C	1.58	1	1.58	91.56*
Θ	24.15	1	24.15	36.15*
T C	0.0420	1	0.0420	0.9643^{ns}
T θ	0.0400	1	0.0400	0.9179^{ns}
C θ	0.3600	1	0.3600	8.26**
T^2	0.2962	1	0.2962	6.80**
C^2	0.0084	1	0.0084	0.1935^{ns}
θ^2	2.99	1	2.99	68.62*
Residual	0.3051	7	0.0436	
Lack of Fit	0.1516	3	0.0505	1.32^{ns}
Pure Error	0.1535	4	0.0384	
Cor Total	33.86	16		
R-Squared	0.9910			
Adj R-Squared	0.9794			
Pre R-Squared	0.9213			
Std. Dev.	0.2088			
Mean	4.04			
C.V. %	5.17			

* Significant at 1%, ** significant at 5% level, ns - Non significant

and SG in real process parameters is given by

$$SG = -8.99512 + 0.34560\ T + 0.00750\ C + 0.01929\ \theta + 0.000255\ C\,\theta - 0.002653\ T^2 - 0.000059\ \theta^2 \qquad \ldots (10)$$

The result of analysis of variance of Eqn. 9 indicated that linear terms of temperature, concentration and duration of osmosis were highly significant at 1 per cent level (Table 5). The quadratic term of the duration of osmosis was also highly significant, while the temperature was significant at 5 per cent and the term concentration was non-significant. Moreover, interaction term of syrup concentration and duration was significant at 5 percent level, while the rest two interaction terms of syrup temperature - concentration and syrup temperature and duration were non-significant. The presence of quadratic terms of the variable in Eq. 10 indicated curvilinear nature of the response surface.

It was observed that the syrup temperature contributed maximum for SG, followed by duration of osmosis and concentration of syrup. Negative coefficient for the quadratic term of syrup temperature and duration indicated that increase in the level of these 3 variables decreased SG. However, the positive coefficients for the linear term of syrup temperature, concentration and duration suggested that an excessive increase in the levels of these variables resulted in significant increase in SG %.

Numerical optimization of osmotic dehydration of banana slices: The constraints were set such that the selected variables such as temperature (T), concentration (C), and duration of osmosis (θ) would be minimum from the economical point of view for the most important product attribute and close to the optimum for the others (Jain *et al.*, 2011) [10]. The main criteria for constraints optimization were maximum water loss and targeted sugar gain of 5 per cent as most important quality (sweetness) attribute (Ade-Omowaye *et al.*, 2002) [1]. The desired goals for each factor and response are shown in Table 6.

Table 6: Optimization criteria for different process variables and responses for osmotic dehydration of ripen banana slices

Parameter	Goal	Lower limit	Upper limit
Syrup temperature, ºC	Minimize	35	55
Syrup concentration, ºBrix	Minimize	40	60
Duration of osmosis, min	Minimize	60	300
Water loss, per cent	Maximize	4.86	24.61
Sugar gain, per cent	Target = 5.00	1.23	6.36

Graphical optimization: A graphical multi-responses optimization technique was adapted to determine the workable optimum conditions for the osmotic dehydration of banana slices. The contour plots for all responses were superimposed and regions (yellow regions) that best satisfy all the constraints were selected as optimum conditions. The criteria for constraint optimization are already given in Table 6. Superimposed contour plots having common superimposed area for all responses for osmotic dehydration of banana slices are shown in Fig. 1. Table 7 shows the software generated optimum conditions of independent variables with the predicted values of responses.

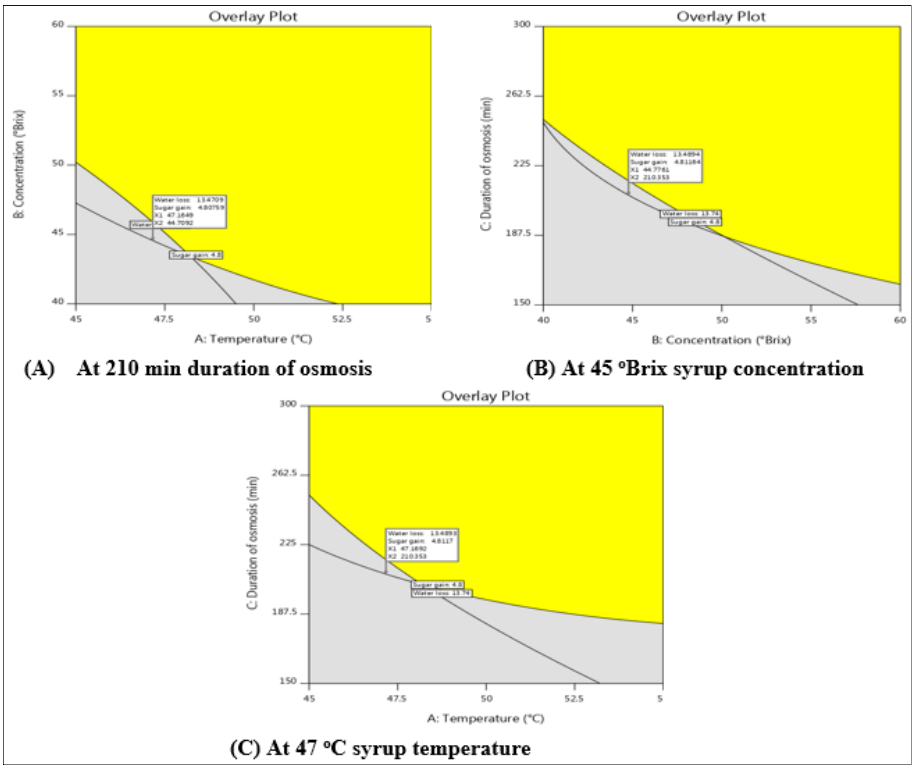

Fig 1: Overlay plots for water loss (%) and sugar gain (%) for osmotic dehydration of banana slices

Table 7: Optimized process parameters for osmotic dehydration of banana slices

No.	Temperature, ºC	Concentration, ºBrix	Duration, min	Water loss, %	Sugar gain, %
1	47.16 ≈ 47	44.77 ≈ 45	210.35 ≈ 210	13.49	4.81

Validation of the model for osmotic dehydration of banana slices: Osmotic dehydration experiments were conducted at the optimum process conditions (Temperature = 47 ºC, Concentration = 45 ºBrix and Duration = 210 min) for testing the adequacy of model equations for predicting the response values. The observed experimental values (mean of three values) and values predicted by the equations of the model are presented in Table 8.

Table 8: Predicted and experimental values of responses at optimized osmosis process conditions

Responses	Predicted value	Experimental value*	Std Dev (±)	C.V., %
Water loss, %	13.49	13.38	0.077	0.58
Sugar gain, %	4.81	4.67	0.098	2.11

* Average of three replications

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 0.58 and 2.11 per cent, respectively.

Conclusion

The effect of sugar syrup concentration, syrup temperature and duration of osmosis during the osmotic dehydration process was investigated and these were optimized. The regression equations of second order polynomial were found to predict the behaviour of osmotic dehydration process of banana slices. If the banana slices were osmo dehydrated in sugar syrup having 45 °C syrup temperature, 47 °Brix syrup concentration for 210 min, then 13.49 per cent of WL could take place with 4.81 percent SG which would be the good quality market acceptable product. During sensory analysis, osmo-dehydrated banana sample with 5 per cent sugar gain was gave excellent acceptability by panellists. It was also concluded that temperature of syrup was the most influencing factor for both the responses.

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