Assessment of mass exchange during osmotic dehydration of dragon fruit

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Abstract: Water loss, weight reduction, solute gain and water activity were investigated during osmotic dehydration of dragon fruit in binary solution of sucrose and sodium chloride using Response Surface Methodology. Experiments were designed according to Central Composite Rotatable Design (CCRD) with four factors Temperature (30 ℃ to 50 ℃), sucrose concentration (45% to 55% w/w), NaCl concentration (2.5% to7.5% w/w) and time (150 to 390 min). Experiments were conducted in a temperature controlled environmental chamber with solution to sample ratio of 10/1 (w/w).
Analysis of variance (ANOVA) was performed to check the adequacy of the fitted models. The response surface plots showing the interaction of variables were developed in Design-Expert 9.0.1. For every response linear variables were found more significant than quadratic variables. Optimum conditions for maximum water loss and weight reduction and minimum solid gain and water activity corresponds to temperature of 30 ℃, sucrose concentration of 55%, NaCl concentration of 6% and time of 270 min. Desirability was 0.81. At this condition, water loss, solid gain, weight reduction and water activity were recorded as 46.35 g/100 g, 5.48 g/100 g, 40.87 g/100g and 0.843 respectively.

Keywords: osmotic dehydration, dragon fruit, water loss, solid gain, weight reduction

Citation: Bobade, S. S., P. K. Nema, and S. Nandi. 2016. Assessment of mass exchange during osmotic dehydration of dragon fruit. Agricultural Engineering International: CIGR Journal, 18 (2):286-296.

1 Introduction

Dragon fruit (*Hylocereus Undatus*), a member of the *Cactaceae* family is native to Mexico, Central and South America (Haber, 1983). Dragon fruit is cultivated on a large scale in Malaysia, Vietnam, Thailand, Israel and Taiwan (Haj Najafi et al., 2014). Dragon fruit is a non-climacteric fruit covered with rosy-red skin studded with green scales and its white flesh contains many small black seeds (Bellec et al., 2006). In the fruit, the most important fruit pigments are betacyanins and betaxanthins (Wybraniec et al., 2001). Dragon fruit has a pH ranging between 4.7 to 5.1 and a Brix value ranging between 11 to 19 °Brix (Gunasena et al., 2007). Therefore it comes under a variety of acidic fresh-cut fruits such as papaya, watermelon, so that these fruit stored at temperatures ranging between 5 ℃ and 25 ℃ (Freitas and Mitcham, 2013). Dragon fruit is preferred due to its attractive color (Adnan et al., 2011), pleasant taste (Castellar et al., 2006), high content of nutrients (Tze et al., 2012) and cancer-preventing effects (Yusof et al., 2012).

Osmotic dehydration (OD) is a very reasonable method for dehydration of plant tissues and very effective for a number of fruits and vegetables (Torreggiani, 1993). It is also considered as an important method for conservation of tropical fruits and development of new products (Ponting, 1973). It is mainly used for product having porous structure. Osmotic dehydration reduces water activity of food materials so microbial growth is inhibited (Yadav and Singh, 2012).

The OD process has been used as a pre-treatment to the traditional processes of food processing such as drying and freezing (Falade et al., 2007). Rate of removal of moisture content of the material and changes in its chemical composition depends upon a number of

Received date: 2015-11-25 **Accepted date:** 2016-01-25
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different factors such as osmotic agent (Singh et al., 2010), temperature (Conway et al., 1983), concentration of the osmotic solution (Conway et al., 1983; Biswal and Maguer, 1989; Lenart, 1992), immersion time (Conway et al., 1983), the size and geometry of material (Lerici et al., 1985), the solution-to-material mass ratio (Ponting, 1973; Flink, 1979; Chaudhari et al., 1993) and agitation (Yadav and Singh, 2012). Combined solutions of sugar and salt can be used to enhance water removal with low solids gain by the products (Lerici et al. 1985; Sereno et al., 2001; Mercali et al., 2011).

As osmotic dehydration is counter current mass transfer process, water removal from the material is always combined by diffusion of solutes from the osmotic solution into the tissue. This solid gain not only changes the taste of material but also create resistance to water loss and mass exchange. There are several factors affecting osmotic dehydration of material in such situations, it is more important to determine the optimum conditions that give maximum water loss as well as weight reduction with minimum solid gain during osmotic dehydration.

Response surface methodology (RSM) is a tool for process as well as product quality improvement. RSM is a collection of different experimental design and optimization techniques that enables the researcher to determine the relationship between the different response and the independent variables. RSM is typically used for mapping a response surface over a specific region of interest, improving the response or for selecting experimental conditions to achieve desired target or specifications or consumer requirement. The main advantage of RSM is the reduced number of experimental runs that provide sufficient information for statistically valid results (Myers and Montgomery, 1995; Khuri and Cornell, 1996).Several researchers used response surface methodology for optimizing osmotic dehydration of fruits and vegetables such as cashew apple (Azoubel, 2003); cherry tomato (Azoubel and Murr, 2004); papaya (Fernandes et al., 2006); banana (Oliveira et al., 2006); melons (Teles et al., 2006); sugar beet (Jokic et al., 2007); diced green peppers (Ozdemir et al., 2008); carrot (Singh et al., 2010); banana (Mercali et al., 2011).

In order to develop a high quality product and to improve the shelf life of dragon fruit, it is essential to do further processing of fruit. Osmotic dehydration is important for further processing of fruit. Objectives of this study are:

a) To study the effect of concentration and temperature of solution and immersion time on osmotic dehydration of dragon fruit;

b) To optimize experimental conditions by using RSM to maximize water loss and weight reduction as well as to minimize solid gain and water activity

2. Materials and methods

2.1 Materials

Fresh Dragon fruit (Hylocereus undatus) were obtained from Indian National Army (INA) market, New Delhi. The fruits were chosen at commercial maturity according to their similarity of color, size and absence of surface defects. Samples were stored at 5℃ and relative humidity of 80%-90% prior to the experiments.

Fruits were washed; the skin were manually peeled with a knife and then were cut into 1.5 ± 0.1 cm cubes manually using a sharp stainless steel knife. Osmotic solutions were prepared by mixing proper amount of food grade sucrose and salt in pure water.

2.2 Osmotic dehydration

Osmotic dehydration was carried out in a batch system. The osmotic solution and the fruit cubes were contained in 250 ml beaker which were placed inside a temperature controlled environmental chamber. Beakers were covered with an aluminium foil to prevent evaporation from the osmotic solution. During each experiment, the ratio of osmotic solution to sample was 10:1 (w/w). At each sampling time (150-390 min) which is determined according to the experimental design, the samples were taken out from the osmotic solution, drained properly, then gently blotted with adsorbent paper and placed on a filter paper in order to remove adhering water and then weighed.

In osmotic dehydration there is greater influence of solutes so that during experiment over a single or one solute, a binary solution of salt and sugar was used as the osmotic solution. Sucrose and salt concentration of the solutions, in the range of 45%-55% (w/w) and 2.5%-7.5% (w/w), respectively, were adjusted according to the experimental design. The effect of temperature and time were also investigated and the experiments were conducted between temperatures of 30 ℃-50 ℃ for time period between 150min-390 min. as per experimental design as shown in Table 1.

Table 1 Independent variables used for optimization of osmotic dehydration

Independent variable	Coded value	Unit	Levels -1	0	+1
Temperature	A	℃	30	40	50
Sucrose concentration	B	%	40	50	55
Salt concentration	C	%	2.5	5	7.5
Time	D	min	150	270	390

2.3 Determination of moisture and solid content

The sample was dried in a hot air oven (Alfa Instruments - 011D) at 105 ℃ for 24 h (AOAC, 1990). The moisture and solid content was then calculated by assuming that the loss in weight of the sample was only due to the loss of moisture alone.

2.4 Determination of mass transfer parameters

Water loss (WL), solid gain (SG) and weight reduction (WR) were expressed in g/g initial matter in order to account for initial weight differences between samples. Mass transfer parameters as a function of contact time in the solution were determined under the assumption that the solutes in the fruit samples did not diffuse into the solution. These were calculated as follows (Jokic et al., 2007).

$$\mathrm{WL}\ (\%) = \frac{\mathrm{M_o} - \mathrm{M_i}}{\mathrm{W_o}} \times 100$$

$$\mathrm{SG}\ (\%) = \frac{\mathrm{S_i} - \mathrm{S_o}}{\mathrm{W_o}} \times 100$$

$$\mathrm{WR}\ (\%) = \mathrm{WL} - \mathrm{SG}$$

Where,

M_o is the moisture content of fresh sample, g;

M_i is the moisture content of osmotically treated sample, g;

S_o is the solid content of fresh sample, g;

S_i is the solid content of osmotically treated sample, g;

W_o is the total weight of fresh sample, g.

2.6 Measurement of water activity

Water activity (a_w) of the osmotically dehydrated samples was measured using a water activity measurement device (Aqua Lab, 4TE) with an accuracy of ±0.001 at 25 ℃. All the experiments were done in triplicate and the average value was taken for calculations.

2.7 Experimental design

The response surface methodology was used to estimate the effects of the process variables such as temperature (A), sucrose concentration (B), salt concentration (C) and treatment time (D) on water loss (WL), solid gain (SG), weight reduction (WR) and water activity (a_w), during the osmotic dehydration of dragon fruit. Process variables were selected as independent variables by means of literature review and preliminary experiments.

A Central Composite Rotatable Design (CCRD) was used for designing the experimental data. The design included 30 different experiments as shown in Table 2. Experiment is designed with eight star points and six replicate in the design. The center runs used for estimating the experimental error and a measure of lack of fit. Curvatures of the model were estimated by adding star points to the factorial design. Different designs generated with independent variables are used as condition values for different runs. Coded values analogous with the natural values of each variable and CCRD are shown in Table 1.

The following second order polynomial model was fitted to the data. Four models of the following form were developed to relate four responses (Y) such as WL, WR, SG and a_w to four process variables:

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_4 D + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 + \beta_{44} D^2 + \beta_{12} AB + \beta_{13} AC + \beta_{14} AD + \beta_{23} BC + \beta_{24} BD + \beta_{34} CD$$

Where,

β_0 is the fixed response at the central point.

$\beta_1, \beta_2, \beta_3, \beta_4$ are linear regression coefficients.

β_{11}, β_{22}, β_{33}, β_{44} are quadratic regression coefficients.

β_{12}, β_{13}, β_{14}, β_{23}, β_{24}, β_{34} are intersection regression coefficients.

A, B, C, D are independent variables.

The mathematical models were estimated for every response by using multiple linear regression analysis. Modelling was done with a linear, squared, quadratic and interaction models. Analysis of variance (ANOVA) was used to find significant terms in the model for each response. Significance was judged by determining the probability level that the F-test calculated from the data is less than 5%. The model capabilities were tested by R^2, adjusted-R^2, predicted-R^2 and prediction error sum of squares (PRESS) (Myers and Montgomery, 1995). A good model will have a large predicted R^2, and a low PRESS.

After experiment mapping of the fitted responses was achieved using Design Expert Version 9.01 software. Optimization of different independent variables for maximization of WL, WR and minimization of SG, a_w was obtained on basis of desirability function in the same version of Design Expert 9.01.

Table 2 Central composite rotatable design of RSM and effect on independent variables

Run	T, ℃	Sucrose Conc., %	NaCl Conc., %	Time, min	WL, %	SG, %	WR, %	a_w
1	-1	-1	-1	-1	26.22	2.84	23.38	0.971
2	1	-1	-1	-1	30.17	3.8	26.37	0.939
3	-1	1	-1	-1	34.65	3.51	31.14	0.961
4	1	1	-1	-1	36.95	4.65	32.30	0.926
5	-1	-1	1	-1	36.9	4.14	32.76	0.884
6	1	-1	1	-1	38.12	5.21	32.91	0.862
7	-1	1	1	-1	40.65	4.18	36.47	0.874
8	1	1	1	-1	41.35	5.82	35.53	0.843
9	-1	-1	-1	1	37.68	5.16	32.52	0.923
10	1	-1	-1	1	39.01	6.68	32.33	0.929
11	-1	1	-1	1	41.92	5.57	36.35	0.901
12	1	1	1	1	45.37	6.91	38.46	0.895
13	-1	-1	1	1	41.07	5.91	35.16	0.837
14	1	-1	1	1	43.95	7.15	36.80	0.839
15	-1	1	1	1	45.05	6.12	38.93	0.821
16	1	1	1	1	48.6	7.55	41.05	0.805
17	-2	0	0	0	36.33	3.98	32.35	0.899
18	2	0	0	0	47.65	5.91	41.74	0.877
19	0	-2	0	0	39.93	5.72	34.21	0.884
20	0	2	0	0	46.1	7.26	38.84	0.841
21	0	0	-2	0	36.56	4.01	32.55	0.964
22	0	0	2	0	49.02	6.21	42.81	0.795
23	0	0	0	-2	16.73	1.9	14.83	0.962
24	0	0	0	2	36.45	7.51	28.94	0.873
25	0	0	0	0	45.38	5.62	39.76	0.877
26	0	0	0	0	44.51	5.16	39.35	0.881
27	0	0	0	0	47.12	5.72	41.40	0.881
28	0	0	0	0	46.43	5.36	41.07	0.871
29	0	0	0	0	44.32	5.11	39.21	0.885
30	0	0	0	0	47.78	5.92	41.80	0.884

3. Results and discussion

ANOVA was used to find out the effect of different variables on responses. The response surface plots were generated to visualize the intersection effect of two variables on the same response.

3.1 Fitting models and analysis of variance

The earlier data from different design run of osmotic treatment of dragon fruit was used as a base for further optimization of osmotic dehydration conditions. Results of different trials of osmotic dehydration of dragon fruit are shown in Table 2. These results were used to determine changes in different responses as per changes in variables. An analysis of variance was completed in response surface methodology to determine the important effects of independent process variables on every response. ANOVA for water loss, solid gain, weight reduction and water activity respectively at $p < 0.01$ is shown in Tables 3 and 4. D_f indicates degree of freedom of each different variable.

Table 3 ANOVA for water loss and weight reduction

		Water loss			Weight reduction		
Source	***Df***		**Sum of squares**	**F value**		**Sum of squares**	**F value**
Model	14	45.92	14.18	< 0.0001	40.44	1042.24	< 0.0001
A	1	1.75	73.57	0.0003	1.16	32.25	0.0055
B	1	2.24	120.42	<0.0001	1.97	93.06	< 0.0001
C	1	2.86	196.31	<0.0001	2.39	136.71	<0.0001
D	1	4.04	392.69	<0.0001	2.87	198.15	<0.0001
AB	1	0.039	0.024	0.9326	-0.0087	0.0012	0.9843
AC	1	-0.17	0.45	0.7154	-0.19	0.60	0.6649
AD	1	0.19	0.58	0.6794	0.14	0.34	0.7454
BC	1	-0.64	6.50	0.1777	-0.58	5.41	0.2048
BD	1	-0.18	0.55	0.6873	-0.13	0.26	0.7752
CD	1	-0.90	12.85	0.0654	-0.76	9.30	0.1025
A^2	1	-0.88	21.14	0.0222	-0.75	15.26	0.0417
B^2	1	-0.62	10.60	0.0911	-0.88	21.04	0.0195
C^2	1	-0.68	12.61	0.0677	-0.59	9.45	0.1000
D^2	1	-4.73	613.12	<0.0001	-4.54	564.31	< 0.0001
Residual	15		48.77			46.14	
Lack of Fit	10		38.77	0.2409		39.64	0.1151
Pure Error	5		10			6.50	
R^2		0.9668			0.9576		
Adj R^2		0.9357			0.9180		
Pred R^2		0.8380			0.7816		
PRESS		237.72			237.69		
C.V.%		4.46			5.00		

Table 4 ANOVA for solid gain and water activity

		Solid gain			Water activity		
Source	***Df***		**Sum of squares**	**F value**		**Sum of squares**	**F value**
Model	14	5.48	52.94	< 0.0001	0.88	0.061	< 0.0001
A	1	0.59	8.40	< 0.0001	-0.00742	0.00132	< 0.0001
B	1	0.27	1.76	0.0013	-0.010	0.00248	< 0.0001
C	1	0.47	5.38	<0.0001	-0.042	0.043	<0.0001
D	1	1.17	32.95	<0.0001	-0.020	0.0099	<0.0001
AB	1	0.048	0.036	0.5796	-0.00263	0.00011	0.0384
AC	1	0.026	0.011	0.7587	1.16×10^{-16}	0.000	1.0000
AD	1	0.045	0.032	0.5995	0.0066	0.0007	< 0.0001
BC	1	-0.056	0.051	0.5127	1.084×10^{-16}	0.000	1.0000
BD	1	-0.057	0.053	0.5035	-0.003375	0.000182	0.0106
CD	1	-0.13	0.29	0.1317	-0.00075	0.000009	0.5265
A^2	1	-0.13	0.48	0.0570	0.00225	0.000139	0.0223
B^2	1	0.25	1.77	0.0012	-0.00412	0.000467	0.0003
C^2	1	-0.091	0.23	0.1767	0.000125	0.00000043	0.8893
D^2	1	-0.19	1.01	0.0090	0.00962	0.00254	< 0.0001
Residual	15		1.69			0.000321	
Lack of Fit	10		1.16	0.4832		0.000188	0.7002
Pure Error	5		0.52			0.000133	
R^2		0.9691			0.9948		
Adj R^2		0.9402			0.9899		
Pred R^2		0.8634			0.9793		
PRESS		7.46			0.0013		
C.V.%		6.27			0.52		

Multiple linear regression analysis of the experimental data produced second order polynomial models for predicting the value of WL, SG, WR and a_w. From ANOVA it can be seen that all the regression models developed for hypothesis were found to be statistically significant at 99% confidence level. The relative effect of each process variable on individual response was compared from the β values corresponding to that parameter. The sign of β coefficient explains the nature of the effect. For every response linear variables were found more significant than quadratic variables. The effects that are not significant ($p > 0.05$) were stepped down from the models without damaging the model hierarchy. The ANOVA of all responses also indicated that lack of fit was not significant for all response surface models at 95% confidence level. But at the same time to check the adequacy of developed model R^2, adjusted -R^2 and coefficient of variation (CV) was calculated. Though, in the study only higher values of R^2 do not always indicate that the regression model is the best one. Thus, it is preferred to use an adjusted -R^2 to evaluate the model adequacy and should be over 90%. Tables 3 and 4 show that R^2 and adjusted -R^2 values for the models did not differ with large value which implies that model is significant. The coefficient of variation (CV) indicates the relative dispersion of the experimental points whose values were 4.46%, 6.27%, 5.00% and 0.52% for water loss, solid gain, weight reduction and water activity, respectively. A low value PRESS and predicted R^2 analogous to fitted R^2 implies that the fitted model is suitable to predicting. Generally, a number closer to one is preferred and the predicted residual sum of squares

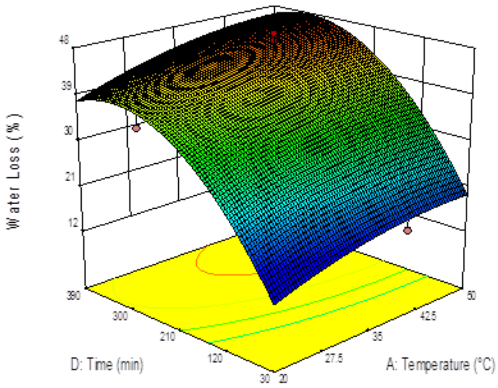
(PRESS) is a measure of how well the model fits each point in the design.

β value indicates the coefficient of equations of the proposed model. In linear variables higher β value of all the four independent variables indicates that increase in variable effects on the water loss. β value of time is 4.04 and higher in all the parameter which indicates that time has higher effect on water loss followed by NaCl concentration, sucrose concentration and temperature respectively. In weight reduction also time has a higher effect on response due to its higher β value, i.e. 2.87 as is shown in Table 3. Therefore, it implies that as time increases weight reduction also increases. As is shown in Table 4 β value of time is 1.17 which is higher in all the parameter which indicates that time has the highest effect on solid gain i.e. as time increases solid gain also increases which creates resistance for mass transfer. In water activity ANOVA it is found that NaCl concentration have higher negative value i.e. -0.042 which implies that as NaCl concentration increases, the water activity decreases.

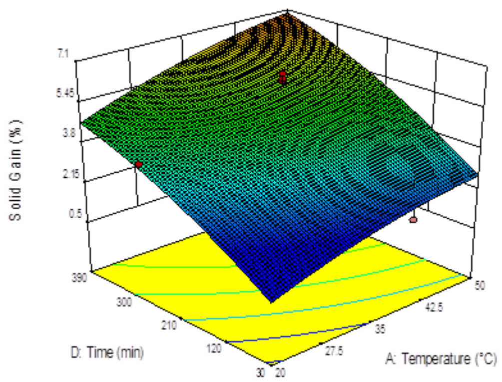
To compute the combined effects of the two independent factors on each response, the response surface plots were generated for each of the fitted models in function of two variables, while keeping other two variables constant. The effect of immersion time and temperature on each response is shown in Figure 1. At the initial stages of the process, as the high osmotic driving force between the concentrated solution and the fresh sample, the rate of water loss, solid gain and weight reduction is higher. After 300 min water loss as well as weight reduction reached nearly the equilibrium conditions and after that it reduces with respect to time because solid gain had increased with time and temperature and it blocks all the pores and which results in decrease in water loss as well as weight reduction after a certain period of time. Quick water loss in the initial stages of osmotic dehydration has been reported by some researchers (Genina-Soto et al., 2001; Eren and Kaymak-Ertekin, 2007).But at initial stages, increasing temperature increases water loss more than solid gain which causes an increase in weight reduction. This phenomenon is familiar to the diffusional differences between water and solutes as related to their molar masses (Torreggiani, 1993). As increase in both temperature and time increases solid gain linearly and weight reduction is also increased. Water activity is high at initial stages at lower temperature then as time and temperature increases water activity reduces as explained in Figure 1 (d). Decrease in water activity is due to increase in solid gain with respect to time. Effect of time is more effective in case of decrease in water activity in comparison with the effect of temperature.

At all processing temperatures, the effect of salt concentration on water loss, solid gain, weight reduction and water activity is greater than the effect of sucrose concentration as explained in Figure 2 and Tables 3 and 4. Combined effect of sucrose and NaCl concentration increases water loss, weight reduction and solid gain as is shown in Figure 2, which also explains that at higher concentration water loss is more and it increases as concentration of both solutes increases. While the increase in both sucrose and salt concentration increases water loss, solid gain and weight reduction, the effect of salt concentration is more distinct for solid gain and water activity than the sucrose concentration. This can be explained by the synergistic effect of binary solutions of sucrose and salt on reducing water activity. Similar results have been reported by a number of researchers (Sacchetti et al., 2001; Eren and Kaymak-Ertekin, 2007).

From the result it has been found that during osmotic dehydration water as well as solid transfer depends on the each process variable such as sugar temperature (a), sucrose concentration (b), salt concentration (c) and treatment time (d) in different manner. This significantly affects the quality characteristics of the final product. Desired products can be developed by applying certain levels of independent process variables. In order to obtain maximum water loss, osmotic dehydration should be conducted at elevated temperatures and low times, but the increase in solid gain must be present in this case. If it is aimed to lower the solid gain, lower temperatures and concentrations should be used preferably. Though, in this case, very long processing periods are required to reach the desired quantity of water removal and solid gain. Therefore, process parameters should be optimized for desired final product characteristics.



(a)



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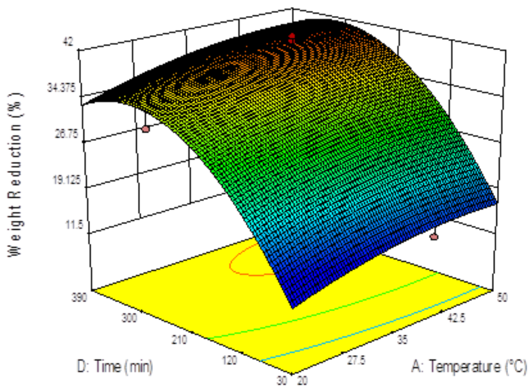

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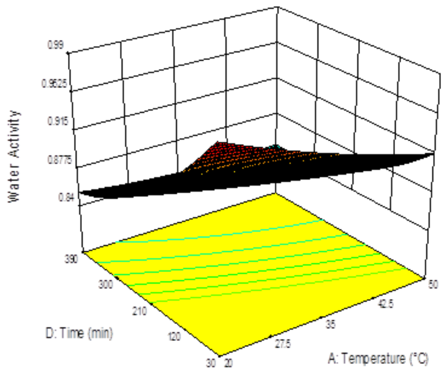
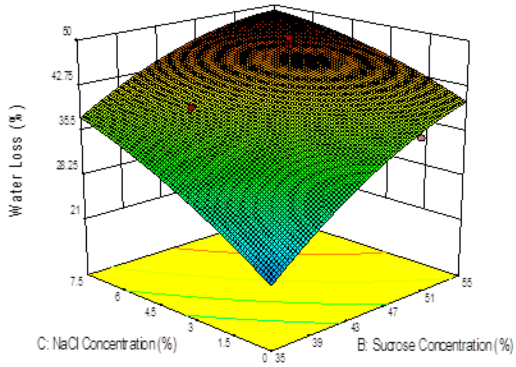
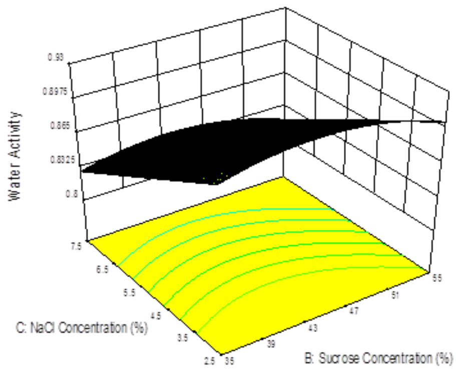


(d)

Figure 1 Effect of temperature and time on: (a) WL, (b) SG, (c) WR and (d) a_w



(a)



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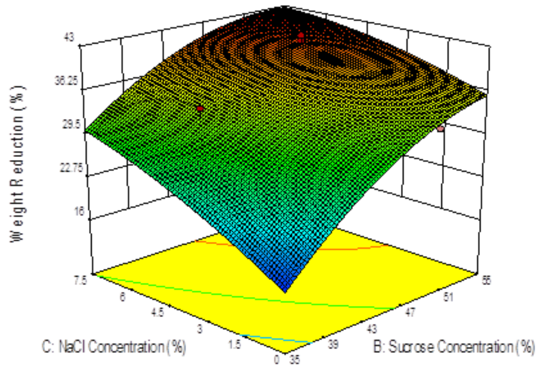
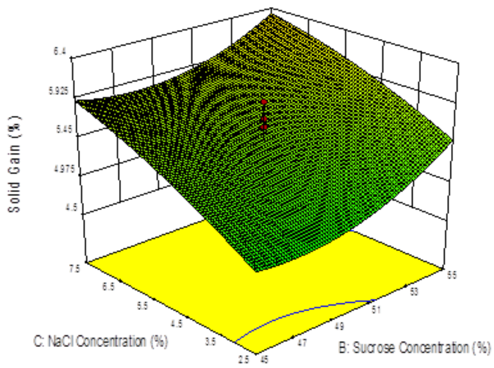


(c)



(d)

Figure 2 Effect of sucrose and NaCl concentration on: (a) WL, (b) SG, (c) WR and (d) a_w

3.2 Optimization

Optimum condition depending on variables for osmotic dehydration of dragon fruit were determined to obtain maximum water loss and weight reduction and minimum solid gain and water activity. Second order polynomial models obtained in this study were utilized for each response in order to determine the specified optimum conditions. In this study, temperature, processing time, sucrose and NaCl concentration were selected in the range of 30℃-50℃, 150-390 min, 45%-55% and 2.5%-7.5%, respectively.

Optimization of these different variables was done in Design Expert, 9.01 with model developed by these independent variables. After optimization 54 different results were obtained, from that one is selected with maximum desirability. The optimum conditions for the osmotic dehydration of dragon fruit are temperature 30 ℃, sucrose concentration 55%, NaCl concentration 6% and immersion time 270 min. with desirability of 0.81. At these conditions water loss 46.35 (g/100 g initial sample), maximum weight reduction 40.87 (g/100 g initial sample), solid gain 5.48 (g/100 g initial sample), water activity 0.843 is obtained.

4 Conclusion

Response surface methodology was used to analyse water loss, weight reduction, solid gain and water activity during osmotic dehydration of dragon fruit. Every response was calculated for different independent factor design and second order polynomial models were developed using multiple linear regression analysis. Analysis of variance (ANOVA) was performed to check the adequacy of the fitted models and during analysis linear variables were found more significant than quadratic variables. The response surface plots showing the interaction of independent variables were developed in Design-Expert 9.0.1. During the study optimum conditions were determined to improve process parameters of osmotic dehydration of dragon fruit and optimization was done by using the same software with maximum desirability. Optimum operating conditions were found to be temperature of 30℃, sucrose concentration of 55%, salt concentration of 6% and treatment time of 270 min with desirability 0.81. At this optimum point, water loss, solid gain, weight reduction and water activity were recorded as 46.35 (g/100 g initial sample), 5.48 (g/100 g initial sample), 40.87 (g/100 g initial sample) and 0.843 respectively.

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