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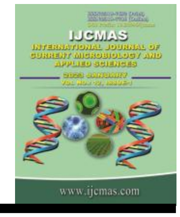

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Optimization of Process Parameters for Osmotic Dehydration of Aloe Vera (*Aloe barbadensis* Miller) Gel Using Response Surface Methodology

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A B S T R A C T

The process parameters for osmotic dehydration of aloe vera gel were optimized using response surface methodology. Sugar syrup temperature (30-50^{o}C), syrup concentration (10-40 ^{o}Brix) and duration of osmosis (60-240 min) were the factors investigated with respect to water loss (WL) and sugar gain (SG). The Box-Behnken design of three variables and three levels including 17 experiments formed by 5 central points was used. The solution to sample ratio of 5/1 (w/w) was used. With respect to water loss and solid gain, the linear, quadratic and interaction effects of three variables were analysed. For each response, second order polynomial models were developed using multiple linear regression analysis. Analysis of variance (ANOVA) was performed to check the adequacy and accuracy of the fitted models. The response surfaces and contour maps showing the interaction of process variables were constructed. The optimum operating conditions were found to be syrup temperature of 31.44^{o}C, syrup concentration of 25.35^{o}Brix and osmosis time of 128.73 min. At this optimum point, water loss and solid gain were predicted to be 39.69 % and 4.45 % respectively.

Introduction

Aloe vera (*Aloe barbadensis* Miller) is a member of the family Liliaceae, which comprises more than 360 different species found in the arid regions of Africa, Asia, Europe and the Americas. It is widely used as a natural treatment and alternative therapy for various types of diseases and several studies have suggested the healing, cosmetic and nutritional benefits of aloe vera (Chang *et al.,* 2006).

The parenchyma cells of aloe vera leaves contain a transparent mucilaginous jelly which is referred to as aloe vera gel consisting of long chain polysaccharides. The gel is a colourless, odourless and hydrocolloid with several natural beneficial substances. Aloe vera gel, like most natural juices, from fruit and vegetable, is an unstable product when extracted and is subject to discolouration and spoilage from contamination by microorganisms.

Unfortunately, because of improper processing procedure, many of these so-called aloe products contain very little or virtually no active ingredients (Ramachandra and Rao, 2006). In view of the known wide spectrum of biological activities possessed by the leaves of the aloe vera plant and its wide spread use, it has become very important to evolve a better method of preservation for increasing the shelf life and maintaining the quality of aloe vera gel and this can be achieved by some type of processing e.g. heating, dehydration (Chang *et al.,* 2006). The main purpose of drying products is to allow longer periods of storage, minimize packaging requirements and reduce shipping weights.

Osmotic dehydration is widely used to remove part of the water content of fruit to obtain a product of intermediate moisture or as a pre-treatment before further processing. Osmotic dehydration is also used to treat fresh produce before further drying to improve sensory, functional and even nutritional properties. The shelf life quality of the final product is better than without such treatment due to the increase in sugar/acid ratio, the improvement in texture and the stability of the colour pigment during storage. Osmotic dehydration combined with other drying technologies provides an opportunity to produce novel shelf stable types of high quality pineapple products for the local as well as export market.

Response surface methodology (RSM) is a statistical procedure frequently used for optimization studies. It uses quantitative data from an appropriate experimental design to determine and simultaneously solve multivariate problems. Equations describe the effect of test variables on responses, determine interrelationships among test variables and represent the combined effect of all test variables in any response. This approach enables an experimenter to make efficient exploration of a process or system. Therefore, RSM has been frequently used in the optimization of food processes (Corzo *et al.,* 2008; Changrue *et al.,* 2008; Mestdagh *et al.,* 2008; Shi *et al.,* 2008; Altan *et al.,* 2008).

Only limited efforts have so far been made to process aloe vera into dehydrated product. An expanding interest currently exists for osmo-convective dehydrated aloe vera in the domestic and world market. No attempt has been made to to optimize the osmotic process parameters for aloe vera. The purpose of the present work was to study the effect of osmotic process parameters on water loss and solid gain and also to optimize these parameters for developing higher quality finished product.

Materials and Methods

Selection of Raw Materials

A widely grown 'Aloe barbadensis Miller' variety of aloe vera was selected for all the experiments. Fresh aloe vera leaves were obtained from Banki Research Farm, Sisarama, Department of Forest, Udaipur (Rajasthan). The two years old and matured aloe vera leaves were selected, cut and transported to the working place in a covered polyethylene bag to avoid oxidation or contamination. The commercial sugar, being cheap and easily available, was used as an osmotic agent.

Sample and Solution Preparation

The aloe vera leaves were thoroughly washed under tap water to remove adhering impurities. Aloin (a yellow colour liquid) which is laxative or purgative if consumed in large amount was extracted by cutting the base of the leaves and allowing them to drain vertically for 1 hour. The epidermis was separated from gel (pulp) and gel was then cut into 10 x 10 x 10 mm ± 1 cubes with the help of sharp stainless steel cutter. The cubes were washed extensively with distilled water to remove the exudates from their surfaces. Sugar syrups of various concentrations were prepared by dissolving required amount of sugar in distilled water.

Osmotic dehydration of aloe vera cubes

The initial moisture content of raw and aloe vera cubes was determined by oven drying as described. The cubes of 1 cm size were osmotically dehydrated using sugar syrup with syrup to fruit 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 placed on tissue paper for 5 min to eliminate excess syrup from the surface. The samples were weighed and their moisture contents determined. The water loss and sugar gain were also calculated. The selection of the levels for processing parameters was made by preliminary experiments in which 5 levels of syrup temperature (30, 40, 50, 60, 70 °C), six levels of osmotic duration (1, 2, 4, 6, 8, 10 h) and eight levels of syrup concentration (10, 20, 30, 40, 50, 60, 70, 80 ^{o}Brix), were studied for various fruits and logical judgment. 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, mass reduction, water loss (WL), sugar gain (SG) and soluble solid concentration were used to indicate the overall exchange of solute and water between aloe vera cubes 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 aloe vera cubes at time (θ) and was calculated on the initial mass basis as

$$WL = \frac{W_{si} X_{swi} - W_{s\theta} X_{sw\theta}}{W_{si}} \times 100 \qquad \ldots 1$$

Solid gain was calculated as a net uptake of solids by food material on an initial mass basis as

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

Where,

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

SG = solid gain (g solids per 100g 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

$X_{sw\theta}$ = water content as a fraction of the syrup at time θ

Product quality

Two responses considered for optimization of osmotic dehydration were water loss and sugar gain. In an osmotic dehydration process, the higher the water loss the better is the dehydration process. However, high solid gain affects the final product quality and sensory characteristics. When high levels of solids are incorporated into the fruit during the osmotic dehydration, significant sensory alterations can occur and the final product may present a taste that is very different from the overall acceptability. Thus, considering the importance of the ‘sugar gain’ in product quality, acceptability and marketability of osmo-convectively dried product, this factor was used as targeted constraint for optimization of the input parameters of osmotic dehydration. For this purpose, it was necessary to fix the level of sugar gain in the final product, so that it is acceptable by the consumers. For this purpose, the osmo-dehydrated products (of 8 different sugar gains) obtained by varying sugar syrup concentration (10, 20, 30, 40, 50, 60, 70, 80 ^{o}Brix) and keeping other parameters constant viz. sample to sugar syrup ratio as 1:5 sugar syrup temperature as 50 ^{o}C and duration of osmosis as 6 h and followed by convective drying (70 ^{o}C, 2 m/s) were used to evaluate the product quality by sensory evaluation (BIS-1971). A nine point hedonic scale was employed for the sensory attributes evaluated. The data on the sensory attributes like overall acceptability (w.r.t. sugar gain) were analysed by analysis of variance (ANOVA). The acceptable (optimum) sugar gain was used as a targeted constraint (one of the response) for optimization of osmotic dehydration of aloe vera gel.

Optimization of input parameters for osmotic dehydration

The process parameters (sugar syrup concentration, syrup temperature and duration of osmosis) were optimized for maximum of water loss and optimum (targeted) sugar gain.

Design of experiments

The method of response surfaces deals with the problem of seeking the conditions of an experiment, which are optimal, *i.e.*, most desirable. The techniques applied here are standard techniques, which have been described in greater detail by Myers (1971). The Box- Behnken design of three variables and three levels including 17 experiments formed by 5 central points was used (Box and Behnken, 1960). The osmotic dehydration was assumed to be affected by three independent variables (regressor or factors), r_i, *viz.*, sugar syrup temperature (T), sugar syrup concentration (C) and duration of osmosis (□). The experimental design of independent parameters and layout are shown in Tables 1 for these three levels and three variables under the Response Surface Methodology (Mullen and Ennis, 1979; Pokharkar 1994; Ranmode 2009). The dependent variables, also referred as responses, Y_k (*i.e.*, the percentage of water loss and sugar gain) were measured experimentally.

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

$$Y_k = f_k(r_1, r_2, r_3, \ldots r_m) \quad \ldots 3$$

The second order polynomial equation of the following form was assumed to relate the response, Y_k and the factors, r_i as

$$Y_k = \beta_{ko} + \sum_{i=1}^{i=3} \beta_{ki} x_i + \sum_{i=1}^{i=3} \beta_{kii} x_i^2 + \sum_{i=1}^{i=2} \sum_{j=i+1}^{j=3} \beta_{kij} x_i x_j \quad \ldots 4$$

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 and x_J are the coded independent variables that are linearly related to T, C and θ. In practice, the levels of the independent variables change from one application to another. Therefore, the general 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 equation:

$$x_i = \frac{(r_i - \bar{r}_i)}{d_i} \quad \ldots 5$$

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. (3) can be written as

$$Y_k = f_k(T, C, \theta) \quad \ldots 6$$

Where, T = sugar syrup temperature, °C, C = sugar syrup concentration, °Brix, θ= osmotic dehydration duration, Y_k = water loss or sugar gain, percent, in osmotic dehydration. Hence, Eqn. (3.8) takes the following form as

$$Y_k = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \quad \ldots 7$$

Response Surface Methodology (RSM) was applied to the experimental data using the package, Design-Expert version 8.0.4.1 (Statease Inc, Minneapolis, USA, Trial version).

Numerical optimization

Numerical optimization technique of the Design-Expert software was used for simultaneous optimization of the multiple responses. The desired goals for each factor and response were chosen. The goals may apply to either factors or responses. 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 economical point of view while the responses viz. water loss was kept maximized and sugar gain was kept targeted.

Graphical optimization

Graphical optimization was also carried out for the process parameters for osmotic dehydration of aloe vera gel for obtaining the best product. For graphical optimization, super imposition of contour plots for all responses was done with respect to process variables using Design-Expert 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 could be considered as the optimum range for best product quality in terms of responses. The optimum combinations of product and process variables for osmotic dehydration of aloe vera gel were derived by averaging those ranges of variables.

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 to the values predicted by the mathematical model.

Results and Discussion

Product quality

Ten judges were given the aloe vera samples having the various levels of sugar gain as shown in Table 2. The judges were asked to taste the samples and give the marks according to their liking. The details of the mean sensory score for the sweetness characteristics of the product as well as the result of these tests are presented in Table 2.

It can be observed from Table 2 that, as the sugar gain increased from 1.41 to 7.56 per cent, the mean sensory scores were increased up to 4.59 per cent and then decreased.

From the analysis of variance, it could be seen that the coefficient of variance among the different judges was 4.87 per cent, which is less than 10 percent indicating coherence amongst the score attributed by the judges. The F value was significant and the CD (5 %) indicated that the product with 4.59 per cent sugar gain was most liked by the judges.

The highest sensory score attributed to the product indicated that osmo-convectively dried aloe vera product having 4.59 per cent sugar gain may acquire higher liking by the consumer. Therefore the input parameters namely, syrup temperature; syrup concentration and duration of osmosis (Table 1) were optimized (as discussed in methodology) on the basis of maximum water loss and targeted sugar gain (4.59 %).

Effect of variables on water loss

The initial mass of the aloe vera samples taken in the study were in the range of 50.10-50.77 g and the final mass after osmotic dehydration was in the range of 27.81-35.58 g (Table 3). Similarly initial moisture content of aloe vera gel samples was in the range of 98.13-99.16 % (wb) which was reduced to 86.24- 96.57 % after osmotic dehydration in various experiments causing WL and SG of 29.63 – 50.97 % and 0.65 – 6.25 % respectively.

A second order polynomial equation [Eqn. (4)] was fitted with the experimental data presented in Table 3 for water loss. Eqn. (8) gives the predicted water loss, per cent 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 described by Snedecor and Cochran (1967). The data for water loss were analysed for stepwise regression analyses as shown in Table 4. The quadratic model was fitted to the experimental data and statistical significance for linear, quadratic and interaction terms was calculated for water loss as shown in Table 4.

The R^2 value was calculated by least square technique and found to be 0.9966 showing good fit of model to the data. The model F value of 377.23 implies that the model is significant ($P < 0.01$). The linear terms (T, C & θ) are significant ($P < 0.01$). The lack of fit F value was non significant which indicates that the developed model was adequate for predicting the response. Moreover the predicted R^2 of 0.9831 was in reasonable agreement with adjusted R^2 of 0.9940. This revealed that the non-significant terms have not been included in the model. Therefore this model could be used to navigate the design space.

The result of analysis of variance of Eqn (8) indicated that the linear terms of syrup temperature, syrup concentration and duration of osmosis were highly significant at 1 % level (Table 4).

The presence of quadratic terms of concentration of syrup and duration of osmosis indicated curvilinear nature of response surface. The quadratic terms of concentration of syrup and duration of osmosis were also highly significant at 1 per cent level while interaction terms of temperature of syrup and duration of osmosis as well as concentration of syrup and duration of osmosis were significant at 5 per cent level.

The comparative effect of each factor on water loss would be observed by the F values in the ANOVA (Tables 4) and also by the magnitudes of coefficients of the coded variables. The F values indicated that concentration of syrup was the most influencing factor followed by duration of osmosis and temperature of syrup was least effective over water loss. The regression equation describing the effects of process variables on water loss in terms of coded values of variable is given as

$$WL = 43.55 + 3.28x_1 + 5.52x_2 + 4.85x_3 + 0.61x_1x_3 + 0.57x_2x_3 - 0.97x_2^2 - 2.71x_3^2 \quad \ldots 8$$

Replacing x_1, x_2 and x_3 with (T-40)/10, (C-25)/15 and (θ –150)/90 respectively [Eqn. (5)] in Eqn. (8), the water loss in real terms of syrup temperature, concentration and duration of osmosis can be given by

$$WL = 8.53 + 0.23T + 0.52C + 0.12\theta + 6.73\times10^{-4}T\theta + 4.25\times10^{-4}C\theta - 4.32\times10^{-3}C^2 - 3.35\times10^{-4}\theta^2 \quad \ldots 9$$

The linear positive terms [Eqn.(8)] indicated that water loss 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.

To visualize the combined effect of two variables on the water loss, the response surface and contour plots (Fig 1 A, B and C) were generated for the fitted model as a function of two variables while keeping third variable at its central value. The water loss increased rapidly in the early stages of osmosis, after which the rate of water loss from aloe vera gel sample into sugar syrup gradually slowed down with time. Rapid removal of water in early stages of osmosis has been reported for apple (Conway *et al.*, 1983), carrots (Uddin *et al.*, 2004), etc.

Table.1 Experimental layout for 3 variables and 3 levels(coded) response surface analysis

Treatment no.	Syrup temperature, °C	Syrup concentration, °Brix	Duration of osmosis, min
	X_1	X_2	X_3
1	1(50)	1(40)	0(150)
2	1(50)	-1(10)	0(150)
3	-1(30)	1(40)	0(150)
4	-1(30)	-1(10)	0(150)
5	1(50)	0(25)	1(240)
6	1(50)	0(25)	-1(60)
7	-1(30)	0(25)	1(240)
8	-1(30)	0(25)	-1(60)
9	0(40)	1(40)	1(240)
10	0(40)	1(40)	-1(60)
11	0(40)	-1(10)	1(240)
12	0(40)	-1(10)	-1(60)
13	0(40)	0(25)	0(150)
14	0(40)	0(25)	0(150)
15	0(40)	0(25)	0(150)
16	0(40)	0(25)	0(150)
17	0(40)	0(25)	0(150)

Note: Data in parentheses indicate decoded (actual) variables

Table.2 ANOVA of sensory evaluation done for products having various levels of sugar gain

Product code	Sugar gain, %	Mean score
01	1.41	**6.10**^{d}
02	2.93	**7.10**^{c}
03	4.59	**8.20**^{a}
04	5.90	**7.50**^{b}
05	6.50	**7.30**^{b}
06	7.04	**7.20**^{b,c}
07	7.47	**7.10**^{c}
08	**7.56**	**7.10**^{c}

**The values superscripted by similar letters are non-significantly different from each other*
F-cal = 27.2 (Sig.), CD (5%) = 0.313, CV = 4.87%

Table.3 Experimental data for water loss and sugar gain under different treatment conditions

S. No.	Syrup temp., °C	Syrup conc., °Brix	Duration of osmosis, min	Initial mass, g	Final mass, g	IMC (wb), %	FMC (wb), %	Water loss, %	Sugar gain, %
1	50	40	150	50.29	27.86	98.62	86.24	50.85	6.25
2	50	10	150	50.33	30.61	99.01	95.11	41.17	1.98
3	30	40	150	50.24	30.14	98.61	89.47	44.93	4.93
4	30	10	150	50.37	33.88	98.80	96.54	33.87	1.13
5	50	25	240	50.28	28.10	98.91	88.03	49.71	5.60
6	50	25	60	50.19	32.92	98.91	91.73	38.74	4.33
7	30	25	240	50.38	31.51	98.13	89.78	41.98	4.52
8	30	25	60	50.12	35.03	98.13	92.56	33.43	3.33
9	40	40	240	50.69	27.81	99.16	87.82	50.97	5.84
10	40	40	60	50.77	32.54	99.16	92.00	40.19	4.29
11	40	10	240	50.46	32.26	98.22	94.00	38.13	2.05
12	40	10	60	50.10	35.58	98.22	96.57	29.63	0.65
13	40	25	150	50.72	31.39	98.27	89.21	43.06	4.95
14	40	25	150	50.40	31.05	98.27	88.91	43.50	5.10
15	40	25	150	50.41	31.14	98.27	88.95	43.32	5.10
16	40	25	150	50.39	31.11	98.27	88.48	43.64	5.38
17	40	25	150	50.35	31.09	98.27	88.39	43.69	5.44

Table.4 Analysis of variance for water loss during osmotic dehydration of aloe vera gel

Source	Sum of squares	df	Mean sum of squares	F value
Model	557.27	7	79.61	**377.23****
T	86.12	1	86.12	**408.09****
C	243.81	1	243.81	**1155.30****
θ	188.13	1	188.13	**891.45****
T θ	1.47	1	1.47	**6.94***
C θ	1.32	1	1.32	**6.24***
C^2	3.99	1	3.99	**18.92****
θ^2	31.08	1	31.08	**147.26****
Residual	1.90	9	0.21	
Lack of Fit	1.63	5	0.33	**4.90**^{NS}
Pure Error	0.27	4	0.067	
Cor Total	559.17	16		
R^2	0.9966			
Adj. R^2	0.9940			
Pred. R^2	0.9831			
C.V. %	1.10			
SD	**0.46**			

** Significant at 1 % Level
* Significant at 5 % Level
NS - Non significant

Table.5 Analysis of variance for sugar gain during osmotic dehydration of aloe vera gel

Source	Sum of squares	df	Mean sum of squares	F value
Model	46.56	6	7.76	**243.48****
T	2.25	1	2.25	**70.75****
C	29.97	1	29.97	**940.43****
θ	3.66	1	3.66	**114.86****
T^2	0.16	1	0.16	**4.93**^{NS}
C^2	8.59	1	8.59	**269.55****
θ^2	1.31	1	1.31	**41.08****
Residual	0.32	10	0.032	
Lack of Fit	0.14	6	0.024	**0.55**^{NS}
Pure Error	0.17	4	0.044	
Cor Total	46.88	16		
R^2	0.9932			
Adj. R^2	0.9891			
Pred. R^2	0.9819			
C.V. %	4.28			
SD	**0.18**			

** Significant at 1% level
NS - Non significant

Table.6 Optimization criteria for different process variables and responses for osmotic dehydration of aloe vera gel

Parameter	Goal	Lower limit	Upper limit	Importance
Temp, °C	minimize	30	50	**3**
Concentration, °Brix	minimize	10	40	**3**
Duration, min	minimize	60	240	**3**
Water loss, percent	maximize	29.63	50.98	**3**
Sugar gain, percent	**target = 4.59**	**0.65**	**6.25**	**3**

Table.7 Solutions generated by the software for osmotic dehydration of aloe vera gel

No.	Temperature, °C	Concentration, °B	Duration, min	Water loss, %	Sugar gain, %
1	**31.44**	**25.35**	**128.73**	**39.69**	**4.45***

*Selected

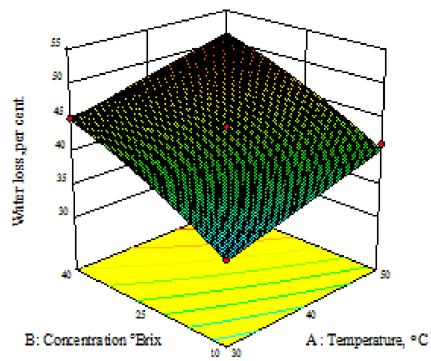
Table.8 Predicted and experimental values of response at optimum process conditions for osmotic dehydration of aloe vera gel

Response	Predicted value	*Experimental value (±SD)	C.V., %
Water loss, %	39.69	40.72(±1.53)	**3.76**
Sugar gain, %	**4.45**	**4.68(±0.368)**	**7.85**

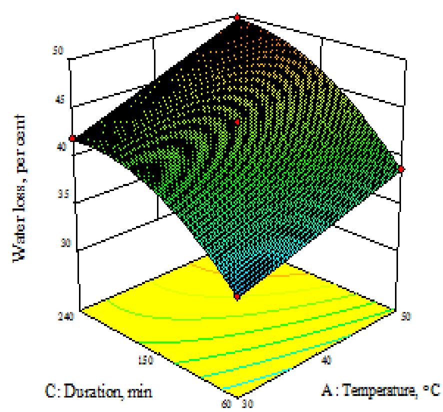
* Average of three replications

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

a. At 150 min duration of osmosis



b. At 25°Brix syrup concentration



c. At 40 °C syrup temperature

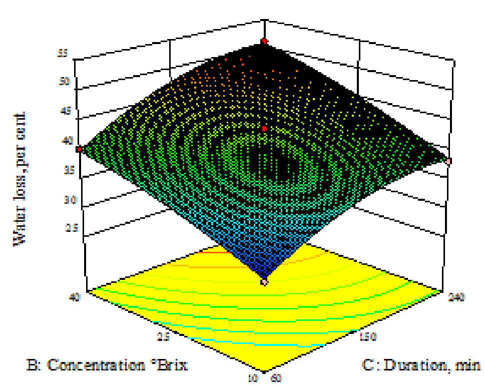
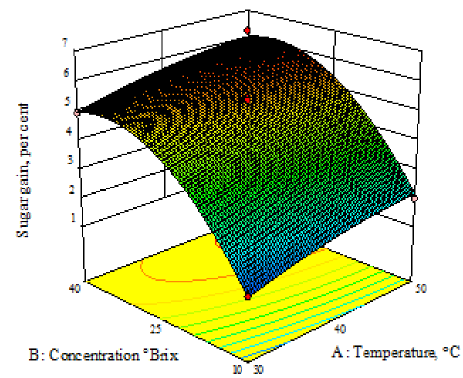
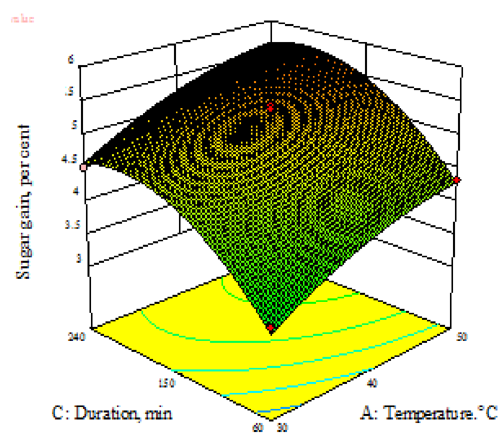


Fig.2 The contour and response surface showing the effect of temperature, concentration and duration on sugar gain during osmotic dehydration

a. At 150 min duration of osmosis



b. At 25 ^{o}Brix syrup concentration



c. At 40 ^{o}C syrup temperature

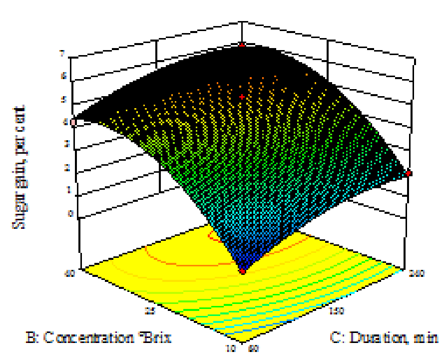
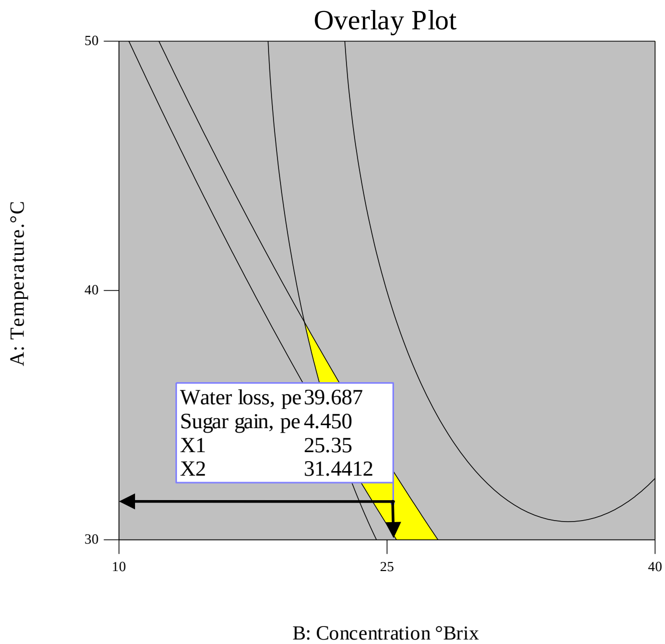
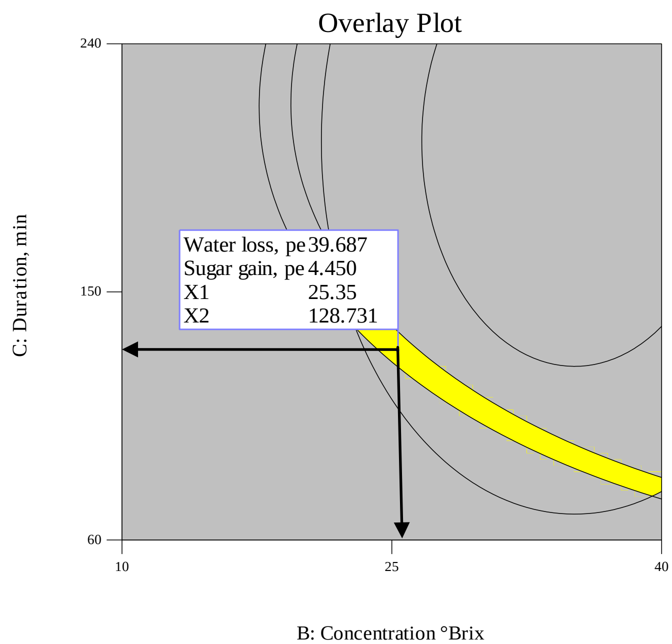


Fig.3 Superimposed contours for water loss (%) and sugar gain (%) for osmotic dehydration of aloe vera gel at varying (A) concentration of syrup and temperature of syrup and (B) concentration of syrup and duration of osmosis

a. At duration of osmosis = 128.73 min



b. At concentration of syrup = 25.35 ^{o}B



Higher temperatures seem to promote faster water loss 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.

Water loss increased with concentration of syrup as well as with duration of osmosis also over the entire osmotic dehydration process. In the osmosis of other fruits and vegetables, also such effect has been observed.

Effect of variables on sugar gain

The sugar gain during the osmotic dehydration was found to be dependent on the syrup temperature, concentration and duration of osmosis. A second order polynomial equation [Eqn. (4)] was fitted with the experimental data presented in Table 3. Eqn. (10) gives the predicted sugar gain, per cent 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 described by Snedecor and Cochran (1967). The data for sugar gain were analyzed for stepwise regression analysis as shown in Table 5. The quadratic model was fitted to the experimental data for sugar gain (Table 3) and statistical significance for linear and quadratic terms was calculated for sugar gain as shown in Table 5. The R^2 value was calculated by least square technique and found to be 0.9932 showing good fit of model to the data. The model F value of 243.48 implies that the model is significant ($P < 0.01$). The linear terms (T, C and θ) are significant ($P < 0.01$).

The lack of fit F value was non significant, which indicates that the developed model was adequate for predicting the response. Moreover the predicted R^2 of 0.9819 was in reasonable agreement with adjusted R^2 of 0.9891. This revealed that the non-significant terms have not been included in the model. Therefore this model could be used to navigate the design space.

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 per cent level (Table 5). The presence of quadratic terms of concentration of syrup and duration of osmosis indicated curvilinear nature of response surface. The quadratic terms of concentration of syrup and duration of osmosis were also highly significant at 1 per cent level while quadratic term of temperature was non significant.

The comparative effect of each factor on sugar gain could be observed by F values in the ANOVA (Table 5) and also by the magnitudes of the coded variables. The F values indicated that concentration of syrup was the most influencing factor followed by duration of osmosis and temperature of syrup was least effective over sugar gain.
The regression equation describing the effects of process variables on sugar gain in terms of coded values of variables is given as

$$SG = 5.19 + 0.53x_1 + 1.94x_2 + 0.68x_3 - 0.19x_1^2 - 1.43x_2^2 - 0.56x_3^2 \quad \ldots 10$$

(R^2 = 0.9932)

Replacing x_1, x_2 and x_3 with (T-40)/10, (C-25)/15 and (□ – 150)/90 respectively [Eqn. (3.9)] in Eqn. (5.24), the sugar gain in real terms of syrup temperature, concentration and duration of osmosis is given by

$$SG = -9.89 + 0.21T + 0.45C + 0.03\theta - 1.93\times10^{-3}T^2 - 6.35\times10^{-3}C^2 - 6.89\times10^{-5}\theta^2 \quad \ldots 11$$

The linear positive terms [Eqn. (10)] indicated that sugar gain increased with increase in syrup temperature, syrup concentration and duration of osmosis. The negative values of quadratic terms of temperature of syrup, concentration of syrup and duration of osmosis indicated that higher values of these variables further reduced sugar gain.

To visualize the combined effect of two variables on the sugar gain, the response surface and contour plots (Fig 2 A, B and C) were generated for the fitted model as a function of two variables while keeping third variable at its central value.

The sugar gain increased rapidly in the early stages of osmosis after which the rate of sugar gain from sugar syrup to aloe vera gel sample slowed down with time.

The sugar gain was found to increase with temperature. As it was explained for water loss, temperature has an effect on the cell membrane permeability that could allow solute to enter by loosing its selectivity. Decrease of solution viscosity at higher temperature may influence sugar gain due to fact that lower viscosity decreases the resistance to diffusion of solutes into the sample (food product) tissue. Increased concentration of the sugar syrup also led to increase in sugar gain probably due to an increase of osmotic pressure gradient and consequent loss of functionality of cell plasmatic membrane that allows solute to entre.

It was observed from these figures (Fig 1 & 2) that the moisture loss as well as the solid gain increased non linearly with time at all concentrations. Both moisture loss and solid gain were faster in the initial period of osmosis and then the rate decreased. This was because osmotic driving potential for moisture as well solid transfer will keep on decreasing with time as the moisture keeps moving from sample to solution and the solids from solution to sample. Further progressive solid 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 (Hawkes and Flink, 1978). 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.

Optimization of osmotic dehydration of aloe vera gel

Numerical and graphical multiresponse optimization technique was adapted to determine the workable optimum conditions for the osmotic dehydration of aloe vera gel. To perform this operation, Design expert programme version 8.0.4.1 of the STAT-EASE software (Statease Inc, Minneapolis, USA, Trial version), was used for simultaneous optimization of the multiple responses.

The constraints were set such that the selected variables (T, C and θ) 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 4.59 percent as most important quality (sweetness attribute). The desired goals for each factor and response are shown in Table 6. 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 7 shows the software generated optimum conditions of independent variables with the predicted values of responses.

The optimum values of process variables obtained by numerical optimization:

Temperature of syrup, ^{o}C : 31.44 ≈ 31

Concentration of syrup, ^{o}Brix : 25.35 ≈ 25

Duration of osmosis, min : 128.73 ≈ 129

The superimposed contours of all responses for C and T (Fig 3 A) and C and θ (Fig 3 B) and their intersection zone for maximum water loss and targeted sugar gain (4.59%) indicated the ranges of variables which could be considered as the optimum range for best product quality. The ranges of optimum values of process variables obtained from the superimposed contours, are as follows,

Temperature of syrup (T) : 30-38 ^{o}C

Concentration of syrup (C) : 18-29 ^{o}Brix

Duration of osmosis (θ) : 77-150 min

Verification of the model for osmotic dehydration of aloe vera gel

Osmotic dehydration experiments were conducted at the optimum process conditions (x_1 = 31 ^{o}C, x_2 = 25 ^{o}Brix and x_3 = 29 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 8. 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 3.76 percent and 7.85 percent respectively. Therefore, it could be concluded from above discussion that model Eqns. (9) and (11) are quite adequate to assess the behavior of the osmotic dehydration.

Response Surface Method was used to determine the optimum operating condition that yield maximum water loss and optimum sugar gain (4.59 % w/w). Analysis of variance has shown that the effects of all the process variables including syrup temperature syrup concentrations and duration of osmosis were statistically significant. Second order polynomial models were obtained for predicting water loss and solid gain. The optimal conditions for maximum water loss and optimum solid gain correspond to syrup temperature of 31.44 ^{o}C, syrup concentration of 25.35 ^{o}Brix and duration of osmosis of 128.73 min in order to obtain water loss of 39.69 % and sugar gain of 4.45 %.

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