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OPTIMIZATION OF OSMOTICALLY DEHYDRATED GINGER CANDY USING RESPONSE SURFACE METHODOLOGY

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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 ginger slices to develop a product called "ginger candy" having medicinal properties. The ginger slices were first osmotically pre-treated and then convectively dried at constant air temperature of 60 °C for achieving a safe moisture level of 12% (wet basis). The Box- Behnken Design (BBD) in Response surface methodology (RSM) was used to investigate the effect of sugar concentration (40-60 °Bx), solution temperature (40-50 °C) and immersion time (30-60 min) on the water loss, sugar gain, overall acceptability and water activity. The optimized conditions of 51.88 oBx sugar concentrations, 40 oC temperature and immersion time of 30 min removed about 28.53 % of water with a minimum sugar gain of 4.75%, 0.33 water activity and overall acceptability of 70.62 % for ginger slices. Analysis of variance (ANOVA) revealed that the process variable, such as temperature and concentration, have remarkable effect on responses. For the proposed model, the comparison of the empirical model with experimental results was evaluated using correlation coefficient (R2) which was found to be 0.994, 0.995, 0.992 and 0.972 for water loss, sugar gain, water activity and overall acceptability, respectively.

Keywords- Ginger candy, Modelling, Osmotic dehydration, Response Surface Methodology (RSM)

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Introduction

Osmoticdehydration (OD) is a dehydration process of foods that involves soaking a food in hypertonic sugar and/or salt solution to reduce the moisture content of foods before actual drying process [8]. The process of osmotic treatment before convective drying is advantageous as far as the quality of the given food product is concerned. The research studies show that osmotic dehydration improves the product quality in terms of colour, flavour and texture [10,17,19,22]. In osmotic dehydration process, heat damage to colour and flavour are minimised, as products are not subject to a high temperature over an extended period of time.

India is the largest ginger producing country in the world. The area under cultivation in India is 105.5 thousand ha. Its production in India is 517.8 thousand tonnes and in Maharashtra, it is 254 tonnes [3]. Ginger (*Zingiberofficinale*) has inflammatory properties and it can be used as an Ayurvedic medicine. Generally, ginger is available in rhizomes form and juice. It is not consumed directly as a medicine due to its spicy taste. It has various medicinal uses, e.g., it promotes digestion, improves blood, lower blood glucose, relieves cold etc. It deteriorates rapidly due to high percentage of moisture content. Approximately, 25-30% of total produce is lost every year in handling, storage and transportation. Osmotic dehydration is gaining popularity as a complementary processing step in the chain of integrated food processing due to low energy consumption and quality related advantages. Therefore, it becomes important to determine the optimum processing conditions that yield maximum water loss and minimum sugar gain during osmotic dehydration.

Response Surface Methodology (RSM) is an important tool in the process optimization and product quality improvement. It is a collection of experimental design and optimization techniques that enables the researcher to determine the relationship between the response and the independent variables. It is typically used for mapping a response surface over a particular region of interest, optimizing the response, and 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 ginger. Hence, the present study on osmotic dehydration of ginger slices was carried out to determine the effect of process parameters on sugar gain, water loss, water activity and overall acceptability and optimize these parameters using RSM.

Materials and Methods

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

Sample Preparation and Experimental Method

The fresh ginger was cleaned and washed under tap water. Thereafter, it was peeled and cut into slices of 3 mm thickness by using a slicer. Osmotic dehydration was done in sugar solution with different concentrations such as 40, 50 and 60 °B. The concentration of the solution was measured by using hand refractometer at room temperature. The sample/solution ratio was constant 1:4 (w/w). The ginger slices were weighed and submerged in the sugar solution at 40, 45 and 50 ^{0}C. The temperature was maintained constant using hot water temperature bath. The samples were removed from the solution at different time intervals (30, 60 and 90 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 [5,21].The detail of treatment combinations is given in [Table-1]. The partially osmotically dried ginger was further dehydrated in a tray dryer at 60°C temperature to a safe moisture level of 12% (wet basis). The moisture content was measured by using hot air oven at 105 ± 2 °C for 24 h [4].

Weight reduction, sugar gain and water loss

The weight reduction, sugar gain and water loss in ginger slices during osmotic process was determined by using equation given below [18].

$$\text{Weight reduction (WR), \%} = \frac{W_2 - W_1}{W_1} \times 100 \quad \text{[Eq-1]}$$

$$\text{Sugar Gain (SG), \%} = \frac{[W_3 - W_1 \times \{(100 - m_1)/100\}]}{W_1} \times 100 \quad \text{[Eq-2]}$$

Water Loss (WL), % = Water Removed (WR) + Sugar Gain (SG) [Eq-3]

Where, W_1, W_2 and W_3 are initial, final and oven dried weight of sample after osmosis (g) and m_1 is initial moisture content of sample before osmosis, % (wet basis).

Water activity

The water activity of experimental samples was determined by using water activity meter (Aqua lab powkit model).

Sensory evaluation

The dried ginger obtained from different combination were served for sensory evaluation by a panel of 10 semi trained judges according to a method of [2] on 9 point hedonic scale. The average score of 10 judges for different quality characteristics *viz.* colour, texture, taste were taken and the overall acceptability was calculated.

Design of experiment

Response Surface Methodology was used to design experiment, model and optimize selected response variables. The statistical software package (Design-Expert ver 8.0.7, Stat-Ease Inc., Minneapolis, USA trial version) was used for regression analysis of experimental data and to plot response surface. The Box-Behnken Design (BBD) of three variables and the three levels including 17 trials formed by 5 central points were used [7] for designing the experiments [Table-1].

Table-1 *Details of different treatments for osmotic dehydration.*

Parameters	Notations	Process variables (Coded)		
		-1	0	+1
Concentration of syrup, °B	A	40	45	50
Temperature of solution, °C	B	40	50	60
Time of immersion, min.	C	30	60	90

The generalized second-order polynomial model used in the response surface analysis was as follow:

$$\eta = \beta_o + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^{k} \beta_{ii} x_i x_j \quad \text{[Eq-4]}$$

where η is the dependent variable (response variable) to be modelled, xi and xj are the independent variables (factors), β0, βi, βii, and βij are the regression coefficients for intercept, linear, quadratic, and interaction terms, respectively. Experimental data were fitted to the second order polynomial model and the regression coefficients were obtained.

Optimization

During optimization of industrial processes, usually several response variables describing the quality characteristics and performance measurements of the systems, have to be maximized while some response variables need to be minimized. In many cases, responses are competing, i.e., improving one response may have an opposite effect on another one, which further complicates the situation. Several approaches have been used to tackle this problem. 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. The advantages of using desirability functions include the following: (1) responses that have different scaling can be compared, (2) the transformation of different responses to one measurement is simple and quick, and (3) both qualitative and quantitative responses can be used [19]. 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 the “desired” limits. The method finds operating conditions (x2) that provide the “most desirable” response values. In the present study, desirability functions were developed for maximizing water loss, overall acceptability and minimizing sugar gain and water activity.

Result and Discussions

Experimental data for response surface method

The experimental data for water loss, sugar gain, water activity and overall acceptability are presented in [Table-2]. The second order polynomial response surface model was fitted to each response variable. Regression analysis and ANOVA were calculated for fitting the model and the significant of the model term was examined statistically. The computed regression coefficients of the second order polynomial models for the response variables along with the corresponding R^2 and coefficient of variance (CV) are given in [Table-3]. As a general rule, the coefficient of variation (CV) should not be greater than 10% [20] and in the current study, the coefficients of variation were found to be in range of 1.55 to 2.62 % for all the responses [Table-3]. A relatively lower value of the coefficient of variation indicates better precision and reliability of the experiments carried out. It has been observed that sugar concentration and solution temperature exerted pronounced effect (higher coefficient values). Analysis of variance shows that the models are highly significant ($p \leq 0.001$) for all the responses [Table-3]. The coefficient of determination (R^2) values of all responses are quite high (>0.97), indicating a high proportion of variability was explained by the data and the RSM models were adequate [Table-3] [12,13].

[Table-3] shows the model F-value of 178.37, 244.79, 135.75 and 37.97 for water loss (WL), sugar gain (SG), water activity (WA) and Overall acceptability (OA) respectively, which implies that the model is significant. Values of "Prob> F" less than 0.0500 indicate that the model terms are significant. The lack of fit, which measures the fitness of model, did not result in a significant F value in case of all responses, indicating that these models are sufficiently accurate for predicting those responses.

Water loss and sugar gain

The observations for water loss and sugar gain with different combinations of the process parameters are presented in [Table-2]. The regression equation describing the effect of the process variables on water loss and sugar gain of osmo dehydrated ginger slices in terms of actual level of the variables is given below:

WL=32.78+5.72 A+1.9B+2.61C-1.20AB-0.57AC+0.095BC-2.66A2-0.40B2-0.71C2 [Eq-5]

where, A = (concentration-50)/10, B = (Temperature-45)/5 and C = (time of immersion –60)/30, respectively in real terms of syrup temperature, concentration and duration of osmosis. The magnitude of P and F values in [Table-3] indicate the maximum positive contribution of osmotic solution temperature, solution concentration and duration on the water loss during osmotic dehydration. It implies that water loss increases with increase in solution temperature and concentration. The increased sugar gain with increase in syrup temperature might be due to the collapse of the cell membrane at higher temperatures. Similar results have also been reported by [9,11,15]. The linear terms of concentration, temperature and time have positive effect on water loss. The quadratic terms of duration have negative effect on water loss. Further, the interactions of ‘temperature and concentration’, ‘concentration and time of immersion’ have positive effect, whereas the interactions of time and temperature’ have negative effect on water loss [Figure-1].

SG=5.78+1.40A+0.48B+0.68C-0.100AB+0.095AC+0.11BC-0.41A2-0.051B2.18C2 [Eq-6]

The linear terms of concentration, temperature and time have positive effect on water loss. The quadratic terms of temperature have negative effect on sugar gain, whereas the interactions terms have negative effect on sugar gain [Figure-2].

Table-2 *Experimental data for the three-factor three level response surface analyses.*

Run	Process Variables (Actual)			Responses			
	Syrup Concentration, °B	Temperature of solution, °C	Time of immersion, min	Water Loss, %	Solid Gain, %	Water activity	Overall Acceptability, %
1	40	50	45	27.03	4.41	0.45	62.36
2	50	45	45	32.78	5.78	0.35	70.39
3	50	45	45	32.78	5.78	0.35	70.35
4	60	50	45	36.88	7.22	0.21	79.35
5	60	40	45	34.81	6.42	0.26	72.36
6	50	50	60	35.77	6.76	0.32	72.36
7	50	45	45	32.79	5.77	0.37	70.31
8	40	40	45	20.17	3.21	0.48	68.34
9	50	45	45	32.78	5.78	0.35	70.39
10	50	45	45	32.78	5.78	0.35	70.39
11	50	45	45	32.78	5.78	0.35	70.39
12	50	45	45	32.78	5.78	0.35	70.39
13	50	40	60	32.32	5.64	0.39	72.36
14	40	45	30	20.7	3.29	0.43	59.68
15	40	45	60	27.5	4.5	0.43	60.38
16	60	45	30	32.45	5.68	0.23	75.36
17	50	50	30	30.82	5.24	0.36	71.34

Table-3 *ANOVA for different models.*

Variables/ Factors	DF	F Values							
		WL, %		SG, %		WA, %		OA, %	
		F Values	p-value Prob> F	F Values	p-value Prob> F	F Values	p-value Prob> F	F Values	p-value Prob> F
Model	9	178.37	< 0.0001	244.79	< 0.0001	135.75	< 0.0001	37.97	< 0.0001
A- Concentration	1	1062.42	< 0.0001	1538.37	< 0.0001	1146.82	< 0.0001	271.76	< 0.0001
B- Temperature	1	121.27	< 0.0001	178.66	< 0.0001	25.61	0.0005	0.95	0.3536
C-Duration	1	221.07	< 0.0001	360.22	< 0.0001	0.00	1.0000	1.46	0.2540
AB	1	23.31	0.0007	3.94	0.0753	1.21	0.2967	35.61	0.0001
AC	1	5.19	0.0459	3.55	0.0887	0.30	0.5941	0.61	0.4522
BC	1	0.15	0.7097	4.55	0.0587	24.55	0.0006	0.83	0.3837
A2	1	131.15	< 0.0001	76.82	< 0.0001	10.48	0.0089	13.12	0.0047
B2	1	3.00	0.1139	1.15	0.3080	7.01	0.0244	16.57	0.0022
C2	1	9.48	0.0117	14.69	0.0033	7.01	0.0244	4.59	0.0578
Residual	10								
Lack of Fit	3	0.93	0.504	0.32	0.813	3.17	0.095	2.7	0.18
Pure Error	7								
Core Total	19								
R2		0.994		0.995		0.992		0.972	
Adj R2		0.988		0.991		0.985		0.946	
C.V, %		1.59		1.83		2.62		1.55	
SD		0.50		0.10		0.01		1.09	

Similar results have been reported by [1] [14]. The increase in water loss and sugar gain with increase in syrup concentration due to increased osmotic pressure in the syrup increased the driving force available for water transport. This trend is in agreement with green beans [6], banana slices [16], and papaya [11].

Water activity

The observations for water activity with different combinations of the process parameters are presented in [Table-2]. The regression equation describing the effect of the process variables on water activity of osmo dehydrated ginger slices in terms of actual level of the variables are given as:

WA = 0.35-0.11A-0.016B+0.00C-5.000E-003AB-2.500E-003AC-0.022BC-0.014A2 +0.011B2-0.011C2 [Eq 7]

The linear terms of concentration and temperature have positive effect on water loss. The quadratic terms have positive effect on water activity, whereas the interactions terms 'temperature and time' have positive effect on water activity [Figure-3]

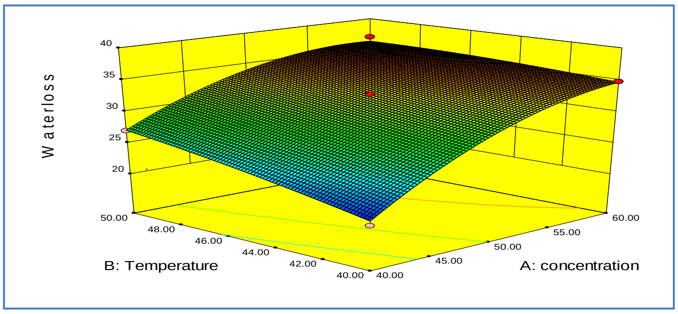

Fig-1 3D plot of combine effect of the temperature and concentration on water loss of ginger.

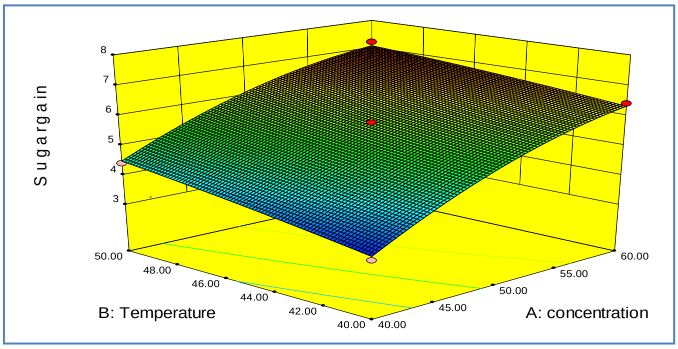

Fig-2 3D plot of combine effect of the temperature and concentration on sugar gain of ginger.

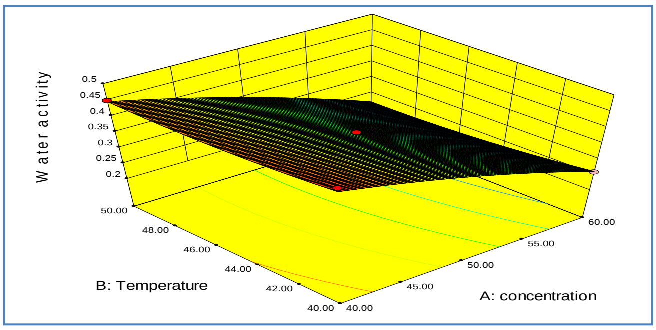

Fig-3 3D plot of combine effect of the temperature and concentration on water activity of ginger.

Sensory evaluation

The overall acceptability is influenced by the process concentration and temperature ($p<0.01$). As concentration increased from 40 °Bx to 60 °Bx, the taste of sample is sweet due to increase in sugar concentration. Meanwhile only immersion time was found to have a no linear and quadratic effect on the overall acceptability of osmo dried ginger slices.

The observations for overall acceptability with different combinations of the process parameters are presented in [Table-2]. The regression equation describing the effect of the process variables on overall acceptability of osmo dehydrated ginger slices in terms of actual level of the variables is given as:

OA = 70.38+6.33A+0.37B+0.46C+3.24AB-0.43AC-0.50BC-1.84A2+2.07B2-.09C2 [Eq. 8]

The linear term of concentration have positive effect on overall acceptability. The quadratic term concentration and temperature have positive effect on overall acceptability, whereas the interactions terms 'temperature and concentration' have positive effect on overall acceptability [Figure-4].

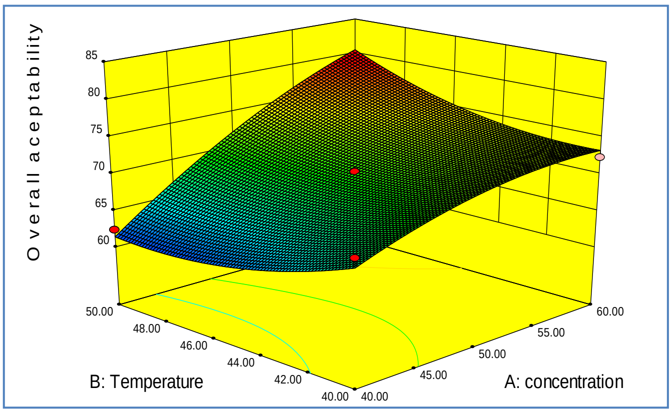

Fig-4 3D plot of combine effect of the temperature and concentration on overall acceptability of ginger.

Optimization of process parameters

Numerical optimization was carried out for the process parameters for osmo dehydrated ginger slices for obtaining the best product. The desired goals were chosen for each factor and response and different weights were assigned to each goal to adjust the shape of its particular desirability function [Table-4]. Solution no.1, having the maximum desirability value was selected as the optimum conditions of osmo dried ginger slices of 3 mm thickness [Table-4].

A graphical multi-response optimization technique was adopted to determine the optimum operating conditions for osmotic dehydration of ginger slices with the expected characteristics. The contour plots for all the responses were superimposed, and regions that best satisfy all the constraints were selected as optimum conditions [Figure-5]. The main criteria for process optimization was minimum sugar gain and water activity and maximum water loss and overall acceptability. The optimum region covered temperature range at 40 to 50 °C, immersion time for 30 min and 40-60 °Bx sugar solution. The preliminary trials were conducted and then finalized the limits of independent variables during the experimentation for optimization. It should be kept in mind that the optimum condition obtained in this study is valid within the limits of experimental factors used.

Verification of the model for osmotic dehydration of ginger slices

Osmotic dehydration experiments were conducted at the optimum process conditions (A = 51.88 °Bx, B = 40 °C and C = 30 min) for testing the adequacy of model equations for predicting the response values.

Table-4 *Optimization criteria for different process variables and responses.*

Variables/Factors	Goal	Lower Limit	Upper Limit	Importance	Solution for optimum condition
A:Concentration	Minimize	40	60	3	51.88
B:Temperature	Minimize	40	50	3	40
C:Duration	Minimize	30	60	3	30
WL	Maximize	20.17	36.99	3	28.53
SG	Minimize	3.21	7.27	3	4.75
Water activity	Maximize	0.21	0.48	3	0.33
OA	Minimize	59.68	79.35	3	70.62

The observed experimental values (mean of 3 experiments) and the values predicted by the model equations are presented in [Table-5]. The experimental values were found to be very close to the predicted values for water loss, sugar gain, water activity and overall acceptability with the percent variation as 1.024, 0.435, 1.975 and 0.020%, respectively, which shows that the model is quite adequate to assess the behaviour of the osmotic dehydration of ginger slices.

Conclusions

RSM was used to determine the optimum operating conditions that yield maximum water loss and overall acceptability and minimum sugar gain and water activity for osmotic dehydration of ginger slices. The 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 sugar gain. The optimal conditions for maximum water loss and minimum solid gain correspond to solution temperature of 40^{o}C, processing time of 30 min, sugar concentration of 51.88 ^{o}Bxand solution to sample ratio of 4:1w/w for obtaining water loss of 28.53%, sugar gain of 4.75%, water activity of 0.33 and overall acceptability of 70.62%. The conformance of experimental results with the empirical model was evaluated using correlation coefficient (R^2), which was found to be 0.994, 0.0995, 0.0992 and 0.972 for water loss, sugar gain, water activity and overall acceptability, respectively for the proposed model.

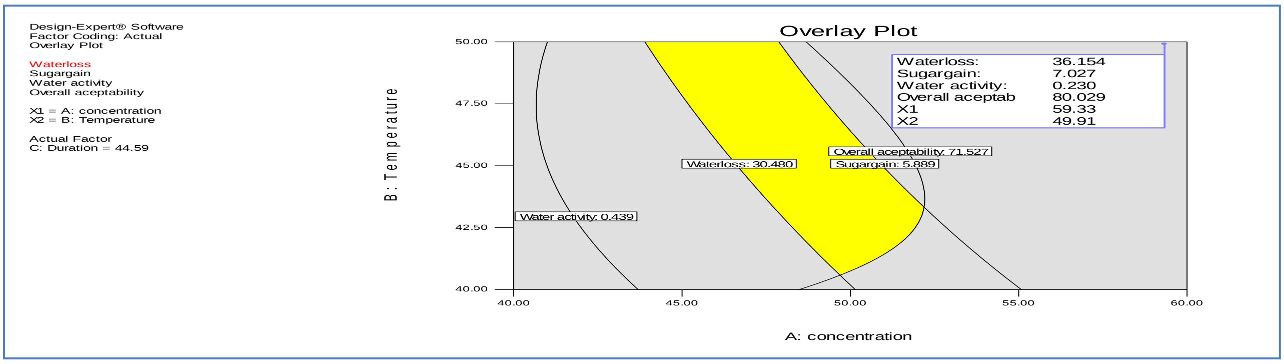

Fig-5 Overlay plot for all responses during osmotic dehydration of ginger slices

Table-5 *Comparison of experimental with predicted values*

Response	Prediction	Actual Value SD*	SE	%Variation	Mean difference
WL	24.562	23.988	0.088	1.024	0.574
SG	3.914	3.812	0.038	0.435	0.102
Water activity	0.342	0.336	0.002	0.020	0.006
OA	76.272	76.018	0.170	1.975	0.254

* Average of

Conflict of Interest: None declared

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