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Optimization of osmotic dehydration process of pointed gourd by response surface methodology

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Abstract
Response Surface Methodology was used for quantitative investigation of effect of salt concentration (5, 10, 15, 20 and 25 %), solution temperature (30, 35, 40, 45 and 50 ºC) and immersion time (30, 60, 90, 120 and 150 min.) on the water loss, solute gain, moisture content of osmo-dehydrated sample, sensory and nutritional properties of pointed gourd samples. Quadratic regression equations describing the effects of these factors on the water loss, solute gain and moisture content of osmo-dehydrated sample were developed. Osmotic dehydration treatment facilitated best results in sensory quality of the final product for the optimized parameters of salt concentration- 10.27 %, solution temperature- 45 ºC, and immersion time- 120 minutes.

Keywords: optimization; osmotic dehydration; pointed gourd; response surface methodology

Introduction
Pointed gourd (*Trichosanthes dioica*) is one of the important cucurbitaceous vegetables due to its high nutritional and medicinal values. It is extensively grown in river beds in the state of Bihar, Uttar Pradesh, West Bengal and Assam in India. It is known as "king of gourds" because of its high nutrient content and medicinal value (Saha *et al.* 2004) [6]. It is very beneficial to improve gastric health. Under ordinary storage conditions, pointed gourd has a very short shelf- life of 3-4 days. It is highly perishable due to its high water content. Depletion of chlorophyll takes place very fast and it results in yellowing of skin and pulp (Kumar *et al*. 2012) [3]. The pointed gourd is a seasonal vegetable and is available during summer till monsoon season. Preserving food products to extend its shelf-life helps the food to be available in the off-season at any place.

Sharma and Shrivastava (2017) [7] carried out solar cabinet drying (54±5ºC) of blanched (hot water for 4 min. at 100ºC), raw and treated (0.5% KMS) pointed gourd samples, and found that KMS treated solar cabinet dried pointed gourd were good and acceptable after rehydration. Osmotic dehydration is one of the easiest and cheapest methods for the preservation of high moisture vegetables. It is a solid-liquid contact process where the removal of water from a solid food is done by immersion in a concentrated aqueous solution. The driving force for water removal is the osmotic pressure difference between the food material and the surrounding medium. The osmotic dehydration process is carried out at constant concentration of solution and temperature which leads to moisture loss and solute gain by the food sample as a function of time.

Osmotic dehydration is one of most important complimentary treatment and food preservation technique in the processing of dehydrated foods, since it presents some benefits such as reducing the damage of heat to the flavor, colour, inhibiting the browning of enzymes and decrease the energy costs (Khan, 2012) [2]. Thus, in order to increase shelf-life, reducing post-harvest losses and making it available throughout the year, osmotic dehydration of fresh pointed gourd was carried. The selection of the osmotic solution, composition and the process conditions aims at maximizing the water removal and minimizing the solute gain. Osmotic dehydration technique has the potential to create rural industries, increase employment opportunities in rural areas. Osmo dehydration does not require any sophisticated equipments (Nazaneen *et al*., 2017) [4]. Response surface methodology (RSM) is widely used for analysis in food industries. In RSM, the variations of several factors are simultaneously studied. The objectives of present study are to optimize the effects of salt concentration, solution temperature and immersion time on water loss, solute gain and moisture content of osmo-dehydrated sample.

Materials and Methods

Preparation of Samples

The fresh and good quality (tender) *Swarnarekha* variety pointed gourds were procured from local market of Pusa. They were properly washed in running water and wiped with blotting paper. Pointed gourds were cut into discs of size approximately (30 mm diameter and 4 mm thickness), before cutting the ends.

Experimental design and statistical analysis

Response surface methodology (RSM) was used to analyze the effects of osmotic dehydration process on water loss (WL), solute gain (SG) and moisture content (MC) of osmo-dehydrated sample. Central Composite Rotatable Design (CCRD) was applied to reduce the number of total experiments, which came to 20 for 3 factor 5 level design having salt concentration (5,10, 15, 20 and 25), solution temperature (30, 35, 40, 45 and 50 ^oC) and immersion time (30, 60, 90, 120 and 150 mins.). The sample to solution ratio was kept constant (1:5). Out of total 20 experiments, the best combination was selected on the basis of optimization by response surface methodology (Patil *et al*. 2014) [5].

Osmotic dehydration

The osmotic dehydration (OD) was conducted in 250 ml glass beakers. In first stage the salt solutions of desired concentrations were prepared by dissolving required amount of salt in distilled water. Pointed gourd discs sample weighing 20g each were placed into the glass beakers containing salt solutions of varying concentrations with solution to product ratio 5:1. The beakers were placed into thermostatically controlled water bath at a particular constant temperature for a particular time interval. At each sampling time, pointed gourd discs were taken out and gently blotted with adsorbent paper and weighed.

Measurement of water loss, solute gain and moisture content of osmo-dehydrated sample

The mass transfer parameters i.e. water loss (WL), solute gain (SG) and moisture content of osmo-dehydrated sample was obtained according to the expressions (Panagiotou *et al*.) [5].

$$\text{Water Loss (WL, \%)} = \frac{(M_o - m_o) - (M - m)}{M_o} \times 100 \qquad (1)$$

$$\text{Solute Gain (SG, \%)} = \frac{m - m_o}{M_o} \times 100 \qquad (2)$$

$$\text{Moisture Content (MC, \%)} = \frac{M_o - M}{M_o} \times 100 \qquad (3)$$

Where,
M_o – Initial weight of fresh sample prior to OD (g);
M – Weight of sample after OD (g);
m – Dry weight of sample after OD (g);
m_o – Dry weight of fresh sample before OD (g).

Results and Discussion

Experiments were carried out using the CCR design given in the table. Water loss (WL), salt gain (SG) and moisture content (MC) of osmo-dehydrated sample were three major dependent quality attributes which were found to be dependent on three independent variables SC, ST and IT. The calculated data of different dependent variables was used to develop multiple regression polynomial equation using a computer program (design expert 11). The evaluation and analysis of coefficients were done statistically.

A polynomial equation was fitted to the data in order to get the regression equation. The statistical significance of the terms in the regression equation was examined by analysis of variance (ANOVA). The generation of response surface plots was done with the same software and optimization of process variables was carried out. A wide variation in all the responses was observed for different experimental conditions i.e. 35.66 to 45.91% for WL, 3.2 to 14.95% for SG and 66.32 to 81.97% for MC of OD sample. The effect of simultaneous variation of two significant independent variables on a particular dependent variable, keeping the remaining two non-significant/ least significant independent variable constants was found out. The model F-Value of 3.23, 43.29 and 12.44 for WL, SG and MC of OD sample respectively implies the model is significant. Responses at different processing conditions during quality parameter analysis are shown in Table 4.1.

The results of the experiments were analyzed through RSM to obtain empirical model for the best response. The mathematical expression showing relationship to response with variables are shown below:

$$\text{WL (\%)} = 40.42 - 1.12\ SC + 0.95\ ST - 0.19\ IT - 0.08\ SC \times ST + 0.66\ SC \times IT - 2.62\ ST \times IT - 0.17\ SC^2 - 0.55\ ST^2 - 0.49\ IT^2\ (R^2=0.74) \qquad (4)$$

$$\text{SG (\%)} = 8.80 + 1.72\ SC + 0.68\ ST + 0.90\ IT + 0.53\ SC \times ST - 0.06\ SC \times IT + 0.57\ ST \times IT - 0.42\ SC^2 - 0.06\ ST^2 - 0.14\ IT^2\ (R^2=0.97) \qquad (5)$$

$$\text{MC of OD sample (\%)} = 74.98 - 1.53\ SC - 1.06\ ST - 0.97\ IT - 0.52\ SC \times ST - 0.08\ SC \times IT - 1.51\ ST \times IT + 0.58\ SC^2 + 0.27\ ST^2 + 0.33\ IT^2\ (R^2= 0.91) \qquad (6)$$

The full second-order multiple regression models were regressed for all the responses at different processing conditions and the regression coefficients along with the coefficient of determination (R^2) were calculated. The sign and magnitude of coefficients indicate the effect of the variable on the response. The negative sign of the coefficients means the decrease in response when the level of the variable is increased while positive sign indicates an increase in the response. Significant interaction suggests that the level of one of the interactive variable can be increased while the other decreased for a constant value of response and non-significant interaction is not considered. A second order polynomial of the following form was fitted to the data of all the responses.

$$Y = a_0 + \sum_{i=1}^{n=3} a_i X_j + \sum_{i=1}^{n=3}\sum_{i=1}^{n=3} a_{ij} X_i X_j$$

Where, x_i and x_j are in coded form and Y is the response of effect of variable.

Optimization of osmotic dehydration

In order to optimize the process conditions during osmotic dehydration, the following considerations were taken: (1) maximization of WL and (2) minimization of SG and MC of osmo-dehydrated sample. The response surface curves plotted are shown in fig. (1-3). The elliptical nature of the contour in 3D-response surface graphs represents the mutual interaction between one dependent variable vs. two independent variables.

Table 2: Central composite rotatable design matrix with calculated values of response (dependent) variables.

Treatment	Independent Variables			Dependent Variables		
	SC (%)	ST (ºC)	IT (min.)	WL (%)	SG (%)	MC of OD sample (%)
1.	10	35	60	44.89	6.75	75.66
2.	20	35	60	45.91	14.95	66.32
3.	10	45	60	39.38	3.20	81.97
4.	20	45	60	35.66	8.55	76.88
5.	10	35	120	39.18	10.5	73.63
6.	20	35	120	39.06	14.05	70.19
7.	10	45	120	39.72	4.20	80.61
8.	20	45	120	37.25	8.50	76.41
9.	6.59	40	90	42.02	6.50	77.04
10.	23.41	40	90	38.82	10.45	73.82
11.	15	31.59	90	38.15	7.95	76.71
12.	15	48.41	90	37.55	8.01	76.86
13.	15	40	39.54	37.41	6.95	78.06
14.	15	40	140.46	38.7	8.7	75.68
15.	15	40	90	37.20	6.05	79.21
16.	15	40	90	35.68	6.5	79.10
17.	15	40	90	37.91	7.09	77.85
18.	15	40	90	39.46	6.60	77.80
19.	15	40	90	40.03	7.15	76.98
20.	15	40	90	39.70	6.90	77.38

Table 3: ANOVA for effect of independent parameters on percent water Loss

Source	Degree of freedom (df)	Sum of Squares	Mean Square	F-value
Model	9	96.02	10.67	3.23*
A-SC	1	17.00	17.00	5.15*
B-ST	1	12.54	12.54	3.80NS
C-IT	1	0.5094	0.5094	0.1544NS
AB	1	0.0573	0.0573	0.0174NS
AC	1	3.53	3.53	1.07NS
BC	1	55.08	55.08	16.69**
A^2	1	0.4394	0.4394	0.1332NS
B^2	1	4.43	4.43	1.34NS
C^2	1	3.48	3.48	1.05NS
Residual	10	33.00	3.30	
Lack of Fit	5	18.61	3.72	1.29NS
Pure Error	5	14.39	2.88	
Cor Total	19	129.02		

**highly significant at 1% level, *significant at 5% level, NS non-significant

Table 4: ANOVA for effect of independent parameters on percent solute Gain.

Source	Degree of freedom	Sum of Squares	Mean Square	F-value
Model	9	65.81	7.31	43.29**
A-SC	1	40.32	40.32	238.70**
B-ST	1	6.48	6.48	38.36**
C-IT	1	11.28	11.28	66.80**
AB	1	2.31	2.31	13.68**
AC	1	0.0313	0.0313	0.1850NS
BC	1	2.64	2.64	15.66**
A^2	1	2.57	2.57	15.20**
B^2	1	0.0676	0.0676	0.4002NS
C^2	1	0.3158	0.3158	1.87NS
Residual	10	1.69	0.1689	
Lack of fit	5	1.02	0.2044	1.53NS
Pure Error	5	0.6671	0.1334	
Cor Total	19	67.49		

**highly significant at 1% level, *significant at 5% level, NS non-significant

Table 5: ANOVA for effect of independent parameters on percent moisture content of osmo-dehydrated Sample.

Source	Degree of freedom	Sum of squares	Mean square	F-value
Model	9	87.23	9.69	12.44**
A-SC	1	32.00	32.00	41.06**
B-ST	1	15.31	15.31	19.64**
C-IT	1	12.85	12.85	16.49**
AB	1	2.20	2.20	2.82NS
AC	1	0.0605	0.0605	0.0777NS
BC	1	18.22	18.22	23.38**
A^2	1	4.90	4.90	6.28*
B^2	1	1.09	1.09	1.40NS
C^2	1	1.66	1.66	2.14NS
Residual	10	7.79	0.7793	
Lack of Fit	5	3.49	0.6984	0.8119NS
Pure Error	5	4.30	0.8602	
Cor Total	19	95.02		

**highly significant at 1% level, *significant at 5% level, NS non-significant

Water loss

Loss (45.91%) was observed at the combination of salt concentration (SC) – 20%, solution It is evident from Table. 2 that the overall variation in Water Loss was from 35.66 to 45.91%. The minimum value of water Loss (35.66%) was observed at the combination of salt Concentration (SC) -20%, solution temperature (ST) – 45ºC, immersion Time (IT) – 60 minutes. However, the maximum value of water temperature (ST) – 35ºC, immersion Time (IT) – 60 minutes. In above model, the high value of the coefficient of determination (R^2) obtained for the response variable (WL) accounted for and adequately explained 74.42% of the total variation. Data were statistically analyzed and ANOVA for effect of independent variables on water loss is presented in Table.3 which shows that SC and ST were significant at 5% level of significance. Table 5 also reveals that the interaction of ST and IT is highly significant at 1% level.

Fig. 1 shows the response surface plot of water loss (WL) vs. two independent variables SC and ST. Negative sign of coefficient values of linear term (SC) indicates that with increase in salt concentration there is decrease in the water loss. The positive sign of the coefficient values of linear term (ST) indicates that the water loss first increases and then becomes constant with increase in solution temperature. High temperature seem to accelerate 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 (Uddin *et al*. 2004) [9].

Solute gain

It is evident from Table 2 that the overall variation in solute gain was from 3.2 to 14.95%. The minimum value of solute gain (3.2%) was observed at the combination of salt concentration (SC)-10%, solution temperature (ST)-45ºC, immersion time (IT)-60 minutes. However, the maximum value of solute gain (14.95%) was observed at the combination of salt concentration (SC)-20%, solution temperature (ST)-35ºC, immersion time (IT)-60 minutes. The analysis of variance (ANOVA) for the effect of independent variables on solute gain is presented under Table 4, which shows that all the three independent variables were highly significant at 1% level of significance. The interactions of SC and ST as well as ST and IT are highly significant at 1% level of significance. The quadratic of SC is highly significant at

1% level of significance. Sridevi and Genitha (2012) [8] reported the maximum positive contribution of all the three variables namely temperature, processing time and sugar concentration on the solid gain. This trend of variation of solute gain with respect to IT and SG is in general agreement with the results of Chavan *et al.* (2010) [1] for osmotic dehydration of ripe banana slices. Fig. 2 shows the response surface plot of solute gain (SG) vs. two independent variables SC and ST. The plot reveals that solute gain increases with increase in solute concentration at all solution temperatures.

Effect on moisture content of osmo-dehydrated sample

It is evident from Table 2 that the overall variation in moisture content was from 66.32 to 81.97%. The minimum value of moisture content was observed at the combination of salt concentration (SC)-20%, solution temperature (ST)-35° C, and immersion time (IT) - 60 minutes. However, the maximum value of moisture content was observed at the combination of salt concentration (SC)-10%, solution temperature (ST)-45° C, and immersion time (IT)-60 minutes. The analysis of variance (ANOVA) for the effect of independent variable on moisture content (MC) of osmo-dehydrated (OD) sample is presented under Table 5.3, which shows that all the three independent variables were highly significant at 1% level of significance. The interaction of ST and IT is highly significant at 1% level of significance. The quadratic of SC is significant at 5% level of significance.

Fig. 3 shows the response plot of moisture content (MC) of osmo-dehydrated sample vs. two independent variables SC and ST. The figure reveals that the moisture content of osmo-dehydrated sample first decrease and then become constant with increase in solute concentration at all solution temperatures. Also, moisture content of the sample decreases with increase in temperature of the solution at all solute concentrations.

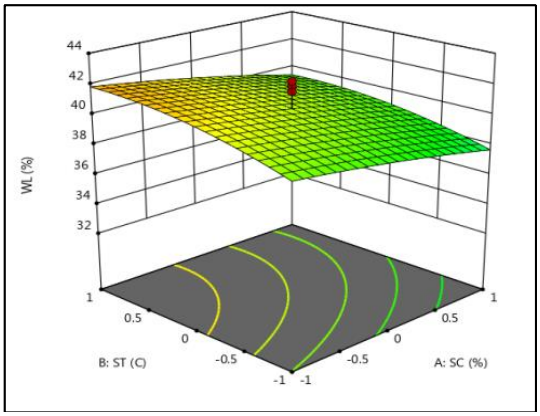

Fig 1: Response surface showing the effect of SC and ST on water loss

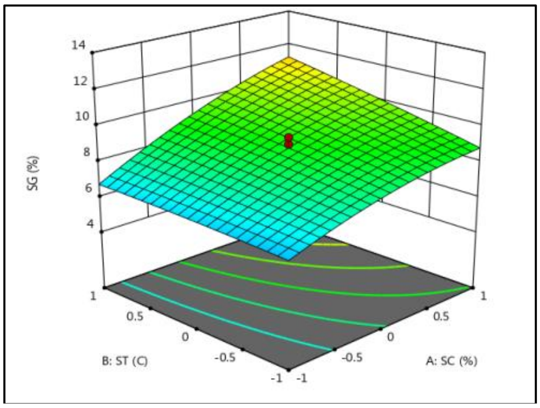

Fig 2: Response surface showing the effect of SC and ST on Solute Gain

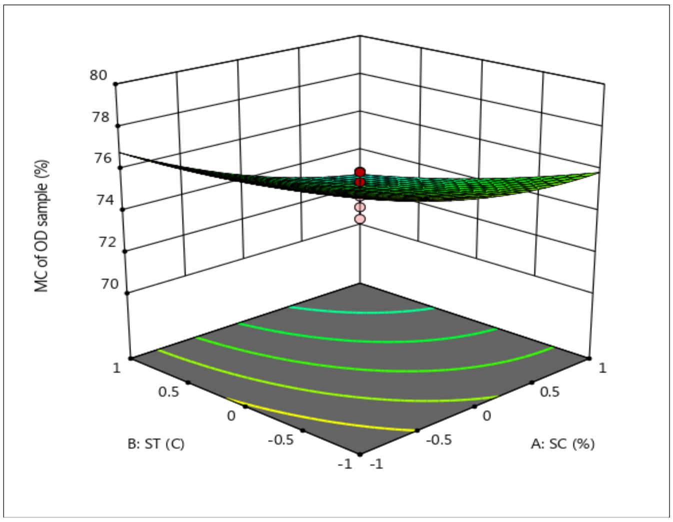

Fig 3: Response surface showing the effect of SC and ST on MC of OD sample

Conclusion

Response surface method was used to determine the optimum conditions that yield maximum water loss and minimum solute gain and moisture content of osmo-dehydrated sample. Analysis of variance has depicted that the effects of all process variables including solution temperature, salt concentration and immersion time were statistically significant. Second order polynomial models were obtained for predicting water loss, salt gain and moisture content of osmo-dehydrated sample.

The optimum solution from this package was emerged out as salt concentration (SC) - 10.275 %, solution temperature (ST) - 45° C and immersion time (IT) - 120 minutes in order to obtain optimized yield as WL- 43.113 %, SG- 8.304 and MC of osmo-dehydrated sample- 74.604.

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