Osmotic Dehydration of Beetroot in Salt Solution: Optimization of Parameters through Statistical Experimental Design

P. Manivannan* and M. Rajasimman

Abstract—Response surface methodology was used for quantitative investigation of water and solids transfer during osmotic dehydration of beetroot in aqueous solution of salt. Effects of temperature (25 – 45°C), processing time (30–150 min), salt concentration (5–25%, w/w) and solution to sample ratio (5:1 – 25:1) on osmotic dehydration of beetroot were estimated. Quadratic regression equations describing the effects of these factors on the water loss and solids gain were developed. It was found that effects of temperature and salt concentrations were more significant on the water loss than the effects of processing time and solution to sample ratio. As for solids gain processing time and salt concentration were the most significant factors. The osmotic dehydration process was optimized for water loss, solute gain, and weight reduction. The optimum conditions were found to be: temperature – 35oC, processing time – 90 min, salt concentration – 14.31% and solution to sample ratio 8.5:1. At these optimum values, water loss, solid gain and weight reduction were found to be 30.86 (g/100 g initial sample), 9.43 (g/100 g initial sample) and 21.43 (g/100 g initial sample) respectively.

Keywords—Optimization, Osmotic dehydration, Beetroot, salt solution, response surface methodology
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I. INTRODUCTION

BETA vulgaris, commonly known as beetroot or beet which is the common American English term for the vegetable, is a flowering plant species in the family Chenopodiaceae. Several cultivars are valued around the world as edible root vegetables, fodder (mangel) and sugar-producing beet (wikipedia). Beetroot can be peeled, steamed, and then eaten warm with butter as a delicacy; cooked, pickled, and then eaten cold as a condiment; or peeled, shredded raw, and then eaten as a salad. Pickled beets are a traditional food of the American South. It is also common in Australia and New Zealand for pickled beetroot to be consumed on a burger. One increasingly popular preparation involves tossing peeled and diced beets with a small amount of oil and seasoning, then roasting in the oven until tender. Garden beet juice is a popular health food. Betanins, obtained from the roots, are used industrially as red food colourants, e.g. to improve the colour of tomato paste, sauces, desserts, jams and jellies, ice cream, sweets and cereals. Red beet also makes a rich, red, Burgundy style wine. The Romans used beetroot as a treatment for fevers and constipation, amongst other ailments. *Beta vulgaris* roots contain significant amounts of vitamin C, whilst the leaves are an excellent source of vitamin A. They are also high in folate, soluble and insoluble dietary fibre and antioxidants. It is among the sweetest of vegetables, containing more sugar even than carrots or sweet corn. The content of sugar in beetroot is no more than 10%.

Osmotic dehydration is a water removal process involving soaking foods, mostly fruits and vegetables, in a hypertonic solution such as concentrated sugar syrup. Two major simultaneous counter-current flows occur during osmotic dehydration: and important water flow out of the food into the solution and a simultaneous transfer of solute from the solution into the food [1]. Osmotic dehydration is used as a pretreatment for many processes used to improve nutritional, sensorial and functional properties of food without changing its integrity [2]. It generally precedes process such as freezing, freeze-drying, vacuum drying or air-drying. It also increases sugar to acid ratio, and improves texture and stability of pigments during dehydration and storage [3]. It is effective around ambient temperatures, so heat damage to texture, colour and flavour can be minimized [2]. The other major application is to reduce the water activity of food materials so that microbial growth will be inhibited. Since most food materials contain large amount of water, they are cost intensive to ship, pack and store [4]. Osmotic dehydration is acknowledged to be an energy efficient method of partial dehydration, since there is no need for a phase change. There are numerous studies on osmotic dehydration of vegetables [5-10].

RSM is a collection of statistical techniques for designing experiments, building models, evaluating the effects of factors and searching for the optimum conditions. It is widely used for multivariable optimization studies in several biotechnological processes such as optimization of media, process conditions, catalyzed reaction conditions, oxidation,

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production, fermentation, biosorption of metals etc., [11-16]. It has also been used to determine the optimal values for process parameters in various processes [17-19]. In RSM, several factors are simultaneously varied. The multivariate approach reduces the number of experiments, improves statistical interpretation possibilities, and evaluates the relative significance of several affecting factors even in the presence of complex interactions. It is employed for multiple regression analysis using quantitative data obtained from properly designed experiments to solve multivariable equations simultaneously. There are several work has been carried out on optimization of vegetables by RSM method [20-24]. However, no information is available on the statistical modeling of beetroot drying by osmotic dehydration. Hence this study focuses on the modeling of the water loss, solid gain and weight reduction as a function of the process variables and to find the optimum operating conditions that maximize water loss and weight reduction and minimize the solid gain using response surface methodology.

II. MATERIALS AND METHODS

A. Materials

Fresh beetroots purchased locally, were thoroughly washed with water to remove adhering soil and other debris. Then, they were cut into circular pieces of 30 mm diameter and 3 mm thickness. The average moisture content of the beetroot was found to be 89% on a wet basis. Salt, the osmotic agent, was purchased from a local supermarket. The osmotic solution is prepared by mixing the salt with proper amount of pure water.

B. Experimental Design and Statistical Analysis

Response surface methodology (RSM) was used to estimate the main effects of osmotic dehydration process on water loss (WL) and solid gain (SG) in beetroot slices. A central composite design was used with temperature (25 – 45°C), processing time (30 -150 minutes), salt concentrations (5 - 25% w/w) and solution to sample to ratio (5:1 – 25:1) being the independent process variables (Table I). For the generated 30 experiments, RSM was applied to the experimental data using design expert 7.1.5.

C. Experimental Procedure

The osmotic dehydration was conducted in a 500 ml Erlenmeyer flask, which is placed in a thermostatically controlled water bath shaker. Beetroot was cut into small pieces and weighed and then placed into dehydrating vessel containing salt solution of varying concentrations (5–25%). The vessel was placed into the water bath at a constant temperature. The solution to sample ratio is varied between 5:1–25:1. At each sampling time (0.5–2.5 h), the beetroot slices were taken out and then gently blotted with adsorbent paper and weighed. The effect of temperature was also investigated and the experiments were conducted between temperatures of 25 - 45°C. The average moisture and dry

TABLE I
THE LEVELS OF DIFFERENT PROCESS VARIABLES IN CODED AND UN-CODED FORM FOR THE OSMOTIC DEHYDRATION OF BEETROOT

Independent variable	Range and levels				
	-2	-1	0	+1	+2
Temperature (A, °C)	25	30	35	40	45
Processing time (B, min)	30	60	90	120	150
Salt Concentration (C, w/w)	5%	10%	15%	20%	25%
Solution to sample ratio (D, %w/w)	5:1	10:1	15:1	20:1	25:1

matter content of the samples were determined by tray drying at 70°C. In each of the experiments fresh osmotic syrup was used. All the experiments were done in triplicate and the average value was taken for calculations. Agitation was necessary to improve mass transfer, maintain uniform concentration, temperature profile and prevent the formation of a dilute solution film around the samples. For each experiment the agitation speed of 200 rpm was used and maintained constant.

In order to follow adequately the osmotic dehydration kinetics, individual analysis for each sample were carried out and from these weight reduction (WR), solid gain (SG) and water loss (WL) data were obtained, according to the expressions

$$WR = ((M_o - M)/M_o) \qquad (1)$$

$$WL = WR + SG \qquad (2)$$

$$SG = ((m - m_o)/M_o) \qquad (3)$$

Where M_0 - initial mass of sample (g), M - mass of sample after dehydration (g), m_0 - initial mass of the solids in sample (g), m - mass of the solids in sample after dehydration (g).

III. RESULTS AND DISCUSSION

A. Fitting Models

Experiments were performed according to the CCD experimental design given in Table II in order to search for the optimum combination of parameters for the osmotic dehydration of beetroot. A Model F-value of 33.53, 57.47 and 45.75 for WL, SG and WR implies respectively that the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. The Lack of Fit F-value of 25.58, 6.66 and 18.30 for WL, SG and WR implies the Lack of Fit is significant. There is only a 0.01% chance that a "Lack of Fit F-value" this large could occur due to noise.

The Fisher F-test with a very low probability value ($P_{model} > F = 0.0001$) demonstrates a very high significance for the regression model. The goodness of fit of the model is checked by the determination coefficient (R^2). The coefficient of determination (R^2) was calculated to be 0.9690, 0.9817 and 0.9771 for WL, SG and WR respectively. This implies that more than 95% of experimental data was compatible with the data predicted by the model (Table II) and only less than 5% of the total variations are not explained by the model. The R^2 value is always between 0 and 1, and a value >0.75 indicates aptness of the model. For a good statistical model, R^2 value

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should be close to 1.0. The adjusted R^2 value corrects the R^2 value for the sample size and for the number of terms in the model. The value of the Adj R^2 (0.9401 for WL, 0.9646 for SG, 0.9558 for WR) is also high to advocate for a high significance of the model. If there are many terms in the model and the sample size is not very large, the adjusted R^2 may be noticeably smaller than the R^2. Here in this case the adjusted R^2 value is lesser than the R^2. The Pred R^2 for WL - 0.8217, SG -0.8946 and WR - 0.8682 are in reasonable agreement with the Adj R^2. The value of CV is also low as 4.02, 7.08 and 4.77 indicate that the deviations between experimental and predicted values are low. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this work the ratio is found to be >19, which indicates an adequate signal.

The experimental results are analyzed through RSM to obtain in empirical model for the best response. The results of theoretically predicted response are shown in Table II. The mathematical expression of relationship to the response with variables are shown below

$$Y_1 = 30.22 - 3.93\,A - 1.13\,B + 3.13\,C - 0.81\,D + 1.13\,AB - 0.59\,AC + 1.00\,AD + 0.44\,BC - 0.26\,BD + 0.24\,CD + 1.32\,A^2 + 0.014\,B^2 + 1.51\,C^2 + 7.604\text{E-}003\,D^2 \qquad (4)$$

$$Y_2 = 9.57 + 0.45\,A + 0.77\,B + 3.64\,C + 0.31\,D - 0.39\,AB - 0.16\,AC - 0.11\,AD + 0.32\,BC - 0.52\,BD + 0.47\,CD + 0.098\,A^2 - 0.38\,B^2 - 0.024\,C^2 + 0.10\,D^2 \qquad (5)$$

$$Y_3 = 20.65 - 4.42\,A - 2.15\,B - 0.57\,C - 1.11\,D + 1.61\,AB - 0.50\,AC + 1.14\,AD + 0.19\,BC + 0.24\,BD - 0.22\,CD + 1.13\,A^2 + 0.75\,B^2 + 1.44\,C^2 - 0.18\,D^2. \qquad (6)$$

where Y_1, Y_2, Y_3 are water loss (%), solid gain (%) and weight reduction (%) respectively, and A, B, C, and D are the coded values of the test variables, temperature (^oC), processing time (min), salt concentration (%w/w), and solution to sample ratio (%w/w) respectively.

The results of multiple linear regressions conducted for the second order response surface model are given in Table III. The significance of each coefficient was determined by Student's t-test and p-values, which are listed in Table III - V. The larger the magnitude of the t-value and smaller the p-value, the more significant is the corresponding coefficient. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case, A, B, C, D, AB, AD, A^2, C^2 and A, B, C, D, AB, AD, A^2, B^2, C^2 are significant model terms for WL and WR respectively. Values greater than 0.10 indicate the model terms are not significant. This implies that the linear and square effects of temperature, processing time, salt concentration and solution to sample ratio are more significant than the other factors. Table III also indicates that the model terms A, B, C, D, AB, BD, CD, B^2 are very significant influence on SG.

TABLE II
EXPERIMENTAL CONDITIONS AND OBSERVED RESPONSE VALUES OF CCD

No	A	B	C	D	Experimental, %			Predicted, %		
					WL	SG	WR	WL	SG	WR
1	1	-1	1	1	27.69	5.46	22.23	32.94	9.46	23.5
2	-1	1	-1	-1	15.9	1.96	13.94	21.78	5.14	16.66
3	0	0	0	-2	22.01	2.54	19.47	26.4	5.36	21.08
4	0	0	0	0	21.52	4.37	17.15	27.36	7.3	20.08
5	0	0	2	0	30.2	14	16.2	32.62	14.28	18.38
6	0	2	0	0	24.16	10.8	13.4	26.28	11.18	15.12
7	0	-2	0	0	25.57	2.5	23.07	28.44	3.42	25.04
8	-2	0	0	0	16.4	5.5	10.97	23.5	8.02	15.5
9	0	0	0	2	22.93	4.68	18.25	28.32	9.24	19.08
10	-1	-1	1	-1	16.7	2.7	14	28.12	8.24	19.92
11	-1	-1	-1	-1	22.49	0.2	22.29	22.86	1.26	21.62
12	0	0	0	0	21.52	4.37	17.15	27.36	7.3	20.08
13	1	-1	1	-1	24.46	5.68	18.78	31.98	7.52	24.5
14	1	1	-1	-1	18.67	0.03	18.64	25.64	4.42	21.24
15	-1	1	1	1	21.65	13.3	8.34	28	14.06	13.96
16	0	0	0	0	21.52	4.37	17.15	27.36	7.3	20.08
17	1	1	1	-1	31.2	14.8	16.38	30.9	11.4	19.54
18	1	1	-1	1	17.26	2.28	14.98	26.6	6.36	20.24
19	0	0	-2	0	19.22	0.23	18.99	22.1	0.32	21.78
20	0	0	0	0	21.52	4.37	17.15	27.36	7.3	20.08
21	0	0	0	0	21.52	4.37	17.15	27.36	7.3	20.08
22	1	-1	-1	1	20.47	0.3	20.17	27.68	2.48	25.2
23	0	0	0	0	21.52	4.37	17.15	27.36	7.3	20.08
24	1	-1	-1	-1	19.93	0.49	19.44	26.72	0.54	26.2
25	-1	1	-1	1	13.4	4.4	9	22.74	7.08	15.66
26	1	1	1	1	31.05	13.7	17.39	31.86	13.34	18.54
27	2	0	0	0	23.95	4.04	19.91	31.22	6.58	24.66
28	-1	-1	1	1	22	7.85	14.15	29.08	10.18	18.92
29	-1	-1	-1	1	26.34	7.82	18.52	23.82	3.2	20.62
30	-1	1	1	-1	20.91	10.1	10.79	27.04	12.12	14.96

B. Response Surfaces and Contour Plots

Response surface plots as a function of two factors at a time, maintaining all other factors at fixed levels are more helpful in understanding both the main and the interaction effects of these two factors. These plots can be easily obtained by calculating from the model, the values taken by one factor where the second varies with constraint of a given Y value. The response surface curves were plotted to understand the interaction of the variables and to determine the optimum level of each variable for maximum response. The response surface curves for WL, SG and WR are shown in Figures (1-18). The nature of the response surface curves shows the interaction between the variables. The elliptical shape of the curve indicates good interaction of the two variables and circular shape indicates no interaction between the variables. From figures it was observed that the elliptical nature of the contour in 3D-response surface graphs depict the mutual interactions of all the variables. There was a relative significant interaction between every two variables, and there was a maximum predicted yield as indicated by the surface confined in the smallest ellipse in the contour diagrams.
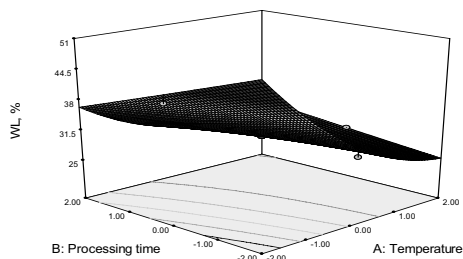
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Fig. 1 3D plot of the combined effect of the temperature and processing time on water loss

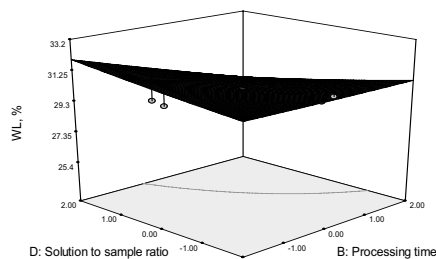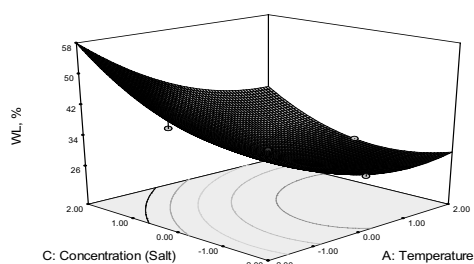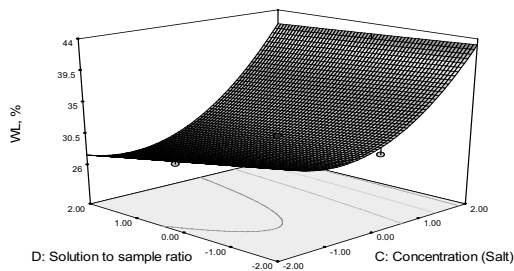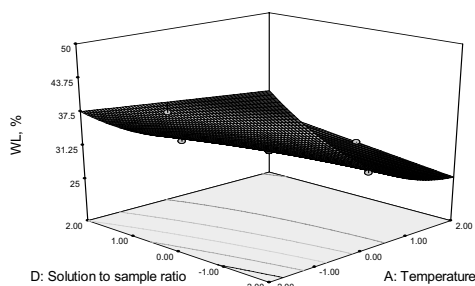Fig. 2 3D plot of the combined effect of the temperature and salt concentration on water loss

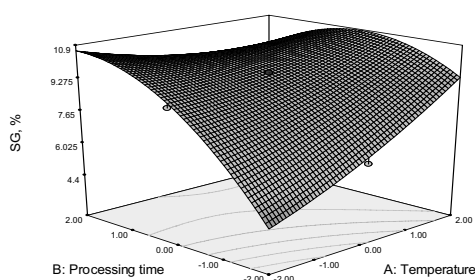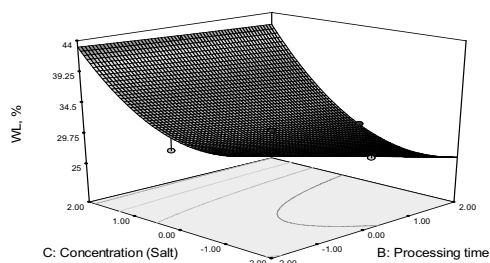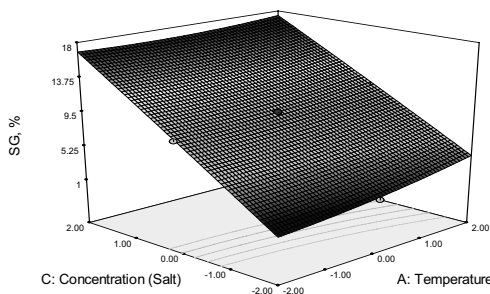Fig. 3 3D plot of the combined effect of the temperature and solution to sample ratio on water loss

Fig. 4 3D plot of the combined effect of the processing time and salt concentration on water loss

Fig. 5 3D plot of the combined effect of the processing time and solution to sample ratio on water loss

Fig. 6 3D plot of the combined effect of the salt concentration and solution to sample ratio on water loss

Fig. 7 3D plot of the combined effect of the temperature and processing time on solid gain

Fig. 8 3D plot of the combined effect of the temperature and salt concentration on solid gain

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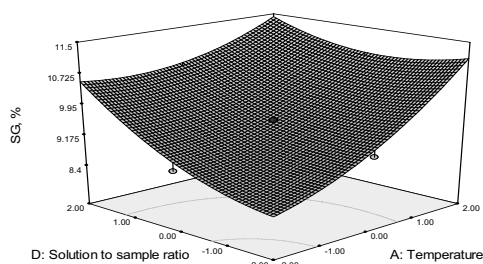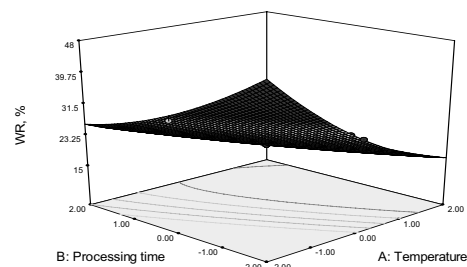International Journal of Chemical and Molecular Engineering
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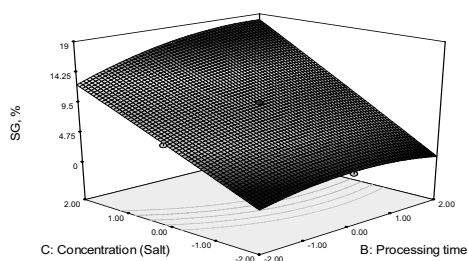Fig. 9 3D plot of the combined effect of the temperature and solution to sample ratio on solid gain

Fig. 10 3D plot of the combined effect of the processing time and salt concentration on solid gain

Fig. 11 3D plot of the combined effect of the processing time and solution to sample ratio on solid gain

Fig. 12 3D plot of the combined effect of the salt concentration and solution to sample ratio on solid gain

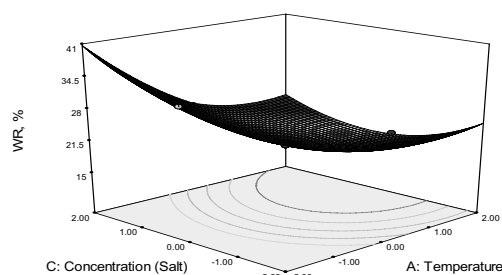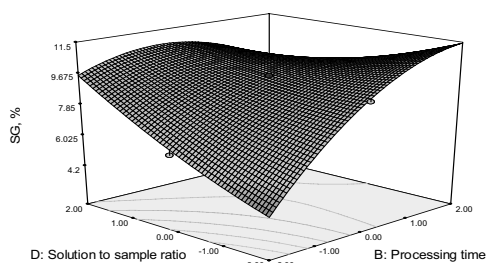Fig. 13 3D plot of the combined effect of the temperature and processing time on weight reduction

Fig. 14 3D plot of the combined effect of the temperature and salt concentration on weight reduction
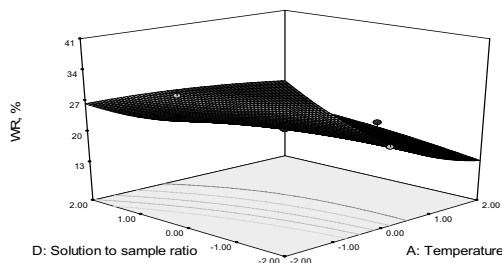

Fig. 15 3D plot of the combined effect of the temperature and solution to sample ratio on weight reduction
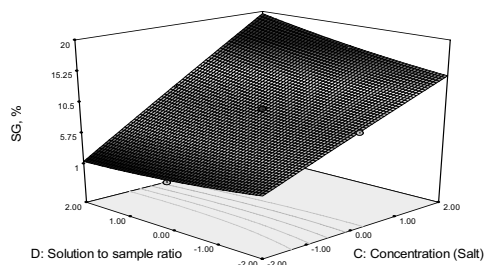

Fig. 16 3D plot of the combined effect of the processing time and salt concentration on weight reduction

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Fig. 17 3D plot of the combined effect of the processing time and solution to sample ratio on weight reduction

Fig. 18 3D plot of the combined effect of the salt concentration and solution to sample ratio on weight reduction
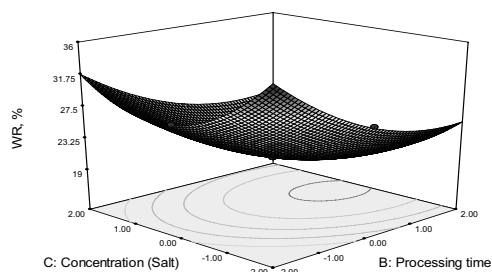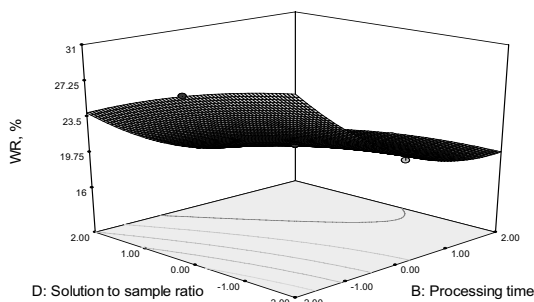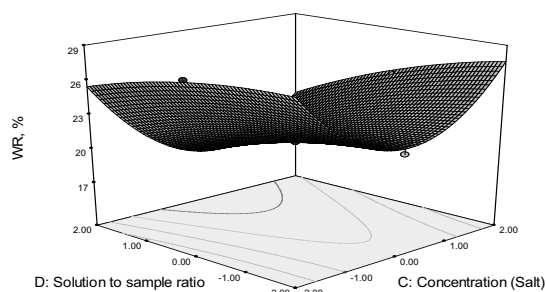
Water Loss

The magnitude of P and F values in Table III indicates the maximum positive contribution of salt concentration and negative contribution of temperature, process time and solution to sample ratio on the water loss during osmotic dehydration. It implies decreased water loss with increase in temperature, processing time and solution to sample ratio up to middle level (level 0) and this is clearly depicted in Figures (1–6). The quadratic terms of temperature, processing time and concentration have positive effect and solution to sample ratio have negative effect on water loss. Further, the interactions of A-B, A-D, B-C, C-D have positive effect, whereas the interactions of A-C, and B-D have negative effect on water loss.

Solid gain

The linear effects of all variables show positive effect on solid gain. It implies increased solid gain with increase of process variables (Figures7-12). The quadratic terms of processing time and concentration have negative effect on solid gain and temperature and solution to sample ratio has positive effect. The interactive effects of B-C and C-D have positive effect on solid gain, whereas the interactive effects of A-B, A-C, A-D and B-D have negative effect (Table IV).

TABLE III
ANALYSIS OF VARIANCE (ANOVA) FOR RESPONSE SURFACE QUADRATIC
MODEL FOR THE OSMOTIC DEHYDRATION OF BEETROOT-WATER LOSS

Source		Water Loss		
	Coefficient	Sum of Squares	F value	p-value
Constant	30.22	800.73	33.53	< .0001
A	-3.93	370.28	217.09	< .0001
B	-1.13	30.62	17.95	0.0007
C	3.13	234.44	137.45	< .0001
D	-0.81	15.73	9.22	0.0083
AB	1.13	20.5	12.02	0.0035
AC	-0.59	5.58	3.27	0.0905
AD	1	16.1	9.44	0.0077
BC	0.44	3.04	1.78	0.202
BD	-0.26	1.05	0.61	0.4459
CD	0.24	0.94	0.55	0.4702
A^2	1.32	47.62	27.92	< .0001
B^2	0.014	0.00527	0.00309	0.9564
C^2	1.51	62.45	36.61	<0.0001
D^2	0.0076	0.00159	0.00093	0.9761
Lack of fit		25.58		
R^2		0.969		
Adj R^2		0.9401		
Pred R^2		0.8217		
CV, %		4.02		
Std.Dev		1.31		
Adeq Precision		19.475		

Weight Reduction

The magnitude of P and F values in Table V gives the maximum negative contribution of temperature, processing time, salt concentration and solution to sample ratio on the water loss during osmotic dehydration. It implies decreased weight reduction with decrease of temperature, process time, salt concentration and solution to sample ratio. This is clearly shown in Figures 13-18. The quadratic terms of temperature, processing time and salt concentration have negative effect on weight reduction. Further, the interactions of A-B, A-D, B-C and B-D have a positive effect, whereas the interactions of A-C and C-D have negative effect on weight reduction.

The studies of the contour plot (Figures 1-18) also reveal the optimum region of the process conditions for the osmotic dehydration of beetroot using salt solution and are; temperature: 35 – 42, processing time: 90 - 120, salt concentration: 10 % - 15% and solution to sample ratio: 5:1 - 10:1.

C. Optimum Condition for Osmotic Dehydration

Optimum condition for osmotic dehydration of beetroot was determined to obtain maximum water loss and weight reduction and minimum solid gain. Second order polynomial models obtained in this study were utilized for each response in order to determine the specified optimum conditions. The sequential quadratic programming in MATLAB 7 is used to solve the

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TABLE IV
ANALYSIS OF VARIANCE (ANOVA) FOR RESPONSE SURFACE QUADRATIC
MODEL FOR THE OSMOTIC DEHYDRATION OF BEETROOT-SOLID GAIN

Source	Solid Gain			
	Coefficient	Sum of Squares	F value	p-value
Constant	9.57	357.05	57.47	< .0001
A	0.45	4.82	10.85	0.0049
B	0.77	14.21	32.03	<0.0001
C	3.64	318.06	716.73	<0.0001
D	0.31	2.25	5.07	0.0397
AB	-0.39	2.44	5.5	0.0332
AC	-0.16	0.41	0.93	0.3501
AD	-0.11	0.2	0.46	0.5073
BC	0.32	1.64	3.71	0.0734
BD	-0.52	4.32	9.73	0.007
CD	0.47	3.47	7.82	0.0136
A^2	0.098	0.26	0.59	0.4529
B^2	-0.38	4.03	9.08	0.0087
C^2	-0.024	0.016	0.037	0.85
D^2	0.1	0.28	0.64	0.4361
Lack of fit		6.66		
R^2		0.9817		
Adj R^2		0.9646		
Pred R^2		0.8946		
CV, %		7.08		
Std.Dev		0.67		
Adeq Precision		30.913		

TABLE V
ANALYSIS OF VARIANCE (ANOVA) FOR RESPONSE SURFACE QUADRATIC
MODEL FOR THE OSMOTIC DEHYDRATION OF BEETROOT- WEIGHT
REDUCTION

Source	Weight Reduction			
	Coefficient	Sum of Squares	F value	p-value
Constant	20.65	781.56	45.75	< .0001
A	-4.42	469.58	384.84	<0.0001
B	-2.15	110.77	90.78	<0.0001
C	-0.57	7.87	6.45	0.0227
D	-1.11	29.7	24.34	0.0002
AB	1.61	41.41	33.94	<0.0001
AC	-0.5	4.02	3.29	0.0896
AD	1.14	20.66	16.93	0.0009
BC	0.19	0.56	0.45	0.5103
BD	0.24	0.95	0.78	0.3913
CD	-0.22	0.77	0.63	0.4406
A^2	1.13	35.09	28.76	<0.0001
B^2	0.75	15.57	12.76	0.0028
C^2	1.44	57.26	46.92	<0.0001
D^2	-0.18	0.92	0.75	0.4
Lack of fit		18.3		
R^2		0.9771		
Adj R^2		0.9558		
Pred R^2		0.8682		
CV, %		4.77		
Std.Dev		1.1		
Adeq Precision		23.276		

second-degree polynomial regression equation 4, 5 and 6. The optimum values obtained by substituting the respective coded values of variables are: temperature – 35°C, processing time – 90 min, salt concentration – 14.31 w/w, solution to sample ratio – 8.5:1w/w. At this point, water loss, solid gain and weight reduction was calculated as 30.86 (g/100 g initial sample), 9.43 (g/100 g initial sample) and 21.43(g/100 g initial sample) respectively. These values are in accordance with an efficient osmotic dehydration process in which less than 10% solid gain are mostly aimed. The value of solid gain 9.43 (g/100 g fresh sample), which is hard to obtain for common osmotic dehydration processes, was achieved.

IV. CONCLUSION

In this study, RSM was used to determine the optimum operating conditions that yield maximum water loss and weight reduction and minimum solid gain in osmotic dehydration of beetroot. Analysis of variance has shown that the effects of all the process variables including temperature, time, salt concentration and solution to sample ratio were statistically significant. Second order polynomial models were obtained for predicting water loss, solid gain and weight reduction. The optimum conditions were found to be: temperature – 35°C, processing time – 90 min, salt concentration – 14.31% and solution to sample ratio 8.5:1. At these optimum values, water loss, solid gain and weight reduction were found to be 30.86 (g/100 g initial sample), 9.43 (g/100 g initial sample) and 21.43 (g/100 g initial sample) respectively.

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