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STATISTICAL OPTIMIZATION OF PROCESS VARIABLES FOR OSMOTIC DEHYDRATION OF OKRA (*Abelmoschus esculentus*) IN SUCROSE SOLUTION

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Abstract

The objective of this study was designed to elucidate the effects of temperature, solute concentration, and size diameter and process time on the osmotic dehydration of okras in sucrose solution. Response Surface Methodology (RSM) with Central Composite Rotable Design (CCRD) was used with five levels and four factors (temperature, sucrose concentration, size diameter and process time) as independent variables, while water loss and solute gain as dependent (response) variables. The osmotic dehydration data was well fitted to a second-order quadratic polynomial regression model with high correlation coefficient ($R^2 >$ 0.90) using the Statistica program (v. 6.08). The quadratic regression models for the water loss and solute gain yielded significant ($p < 0.05$) and predictive results. The osmotic dehydration process was optimized for water loss and solutes gain. The predicted optimum conditions to achieve 39. 78 percent water loss and 10.16 percent solute gain were found to be: solute concentration, 49.28(% w/w); solution temperature, 40.79°C; sample size diameter, 15mm and process time, 4.49hr. At this predicted optimum point, the observed water loss and solute gain were found to be 38.87 and 10.65(g/100g initial sample), respectively.

Keywords: okra, optimization, osmotic dehydration, process variables, response surface, sucrose

1. Introduction

In most part of the world, okra (*Hibiscus/Abelmoschus esculentus*) also called 'lady finger' is considered as an important vegetable crop, for its economical and nutritive values. It is one of the main vegetable crops cultivated in tropical countries and warmer parts of temperate countries. Okra is grown for its fibrous pods containing seeds [1]. It is a source of protein, vitamins C and A, iron, calcium, dietary fiber and low saturated fat [2]. Freshly harvested okra has very high moisture content (88-90% wet basis) with safe moisture content for storage (10% wet basis) [3]. Due to its high moisture content, its shelf life does not increase [4], because it is subjected to rapid deterioration, resulting in chemical, physical and biological changes. Because of its sensitivity to storage, most fresh okras are preserved in some form. One of the most widely used methods of food preservation is drying which also extends the shelf-life of food. The removal of water from solid foods is a form of food preservation, inhibiting the growth of microorganisms, besides preventing a large part of biochemical reactions, which occur while the moisture is present [5]. The major aim of drying agricultural products is the reduction of the moisture content to a level which allows safe storage over an extended period [6].Moreover, drying of food materials leads to new more easily handled consumed products [7].

Osmotic dehydration (OD) is a technique that involves product immersion in a hypertonic aqueous solution leading to loss of water through the cell membranes of the product and subsequent flow along the inter-cellular space before diffusing into the solution [8]. The removal of water out of the tissue is completed by a counter-current diffusion of the osmotic agent from the solution toward the tissue. The driving force for water removal is the difference of chemical potential between the solution and the intracellular fluid. If the membrane is perfectly semi-permeable (i.e., water-permeable, solute-repellant) solute is unable to diffuse through the membrane into the cells [9]. However, due to absence of semi permeable membrane in food, there is always some solute diffusion into the food and leaching out of the foods own solute. Thus, mass transport in osmotic dehydration is actually a combination of simultaneous water and solutes transfer processes [10].These two simultaneous transports bring about depressing effect on the water activity (aw) of the samples [11]. As a result, osmotic dehydration can be applied either as an autonomous process or as a pre-treatment in alternative process, such as freezing, freeze drying, vacuum drying and air drying. In recent years, osmotic dehydration has received considerable attention due to the consumer demand of minimally processed products.

The use of osmotic dehydration process in the food industry besides reducing the drying time has several advantages such as final product quality improvement in terms of color, flavor, and texture; energy efficiency, product stability and retention of nutrients during storage [10, 12].More water than solute usually transfers due to deferential permeability of cellular membranes [13]. This technique also allows the incorporation of certain solutes, without modifying the integrity of the product. The osmotic dehydration process can be characterized by equilibrium and dynamic periods. In the dynamic period, the mass transfer rates are increased or decreased until equilibrium is reached. Equilibrium is the end of osmotic process, i.e. the net rate of mass transport is zero.

The performance of osmotic dehydration has been widely investigated and extensive results are presented in the literature for both fruits and vegetables, amongst other products. According to the findings, the mass transfer and final OD product quality depends on several factors, such as: tissue properties [14]; ripeness level in the case of fruits [15]; process temperature [16]; type of solute employed [17]; syrup concentration [5, 18]; format and dimension of the fruit pieces [19]; process time [20] and syrup: fruit mass ratio [19]. A few studies have also reported on the influence of solution agitation [21] and the application of vacuum [22].

More also, a number of researchers have conducted studies on the optimization of osmotic dehydration of fruits and vegetables using response surface methodology [23–25]. Only limited efforts have so far been made to study the air-drying of okra [1, 2, 26]. However, no effort has been made to study the osmotic dehydration of okra. Various osmotic agents such as sucrose, glucose, fructose, corn syrup and sodium chloride have been used individually or in some combinations for osmotic dehydration. The objective of this work was to study the osmotic dehydration of okra as a function of temperature, sucrose concentration, size of the sample (diameter) and processing time through response surface methodology (RSM) in order to identify process conditions for a high water loss at minimal solute uptakes (as an extensive sugar uptake is undesirable and the product can no longer be marketed as 'natural') and to optimize the osmotic dehydration as a pre-treatment to further processing.

2. Materials and Methods

2.1. Sample

Fresh okras were obtained from a local market, sorted visually for size, maturity level and physical damage. The okras were washed with fresh water to remove any impurities adhered to the surface of the fruit. The product was stored under refrigeration until used. Commercial sucrose was purchased from local market.

2.2. Methods

2.2.1. Experimental procedure

The okras were cut into slices or diskof different sizes (diameter) with length to diameter ratio of 1:1 and only the middle parts were used. No blanching was conducted prior to osmosis as it has been reported to be detrimental to osmotic dehydration processes as a result of the loss of semi-permeability of the cell membranes [27] and reduction of b -carotene [28]. The osmotic solution was prepared by dissolving the required quantity of sucrose in distilled water (w/w), under the conditions given by the central composite experimental design (Table 1). For each experiment, known weights of okra disks (approximately 10 g) were put in stainless steel containers (of 500ml capacity each) containing calculated volumes of osmotic solutions of different concentrations (45–65% w/w)and placed inside a temperature-agitation controlled shaker (Tecnal, TE421)preset at the specified temperature (40–60°C).Gentle agitation of 80 rpm was applied for a good mixing of the medium. The mass ratio of osmotic medium to okra samples was 10:1 to avoid significant dilution of the medium and subsequent decrease of the driving force during osmotic process.

After the predetermined process time, samples were removed from the solution, drained and the excess of solution at the surface was removed with absorbent paper for subsequent mass determination. Water loss (WL) represented by % (g water/100g initial wet okra) and solute gain (SG) represented by % (g solids/100g initial wet okra) were determined by gravimetric method, according to the following relations [19]:

$$SG = \frac{m_f m_o}{M_o} \times 100 \quad (1)$$

$$WL = \frac{(M_o - m_o) - (M_f - m_f)}{M_o} \times 100 \quad (2)$$

Where: M_o is initial weight of fresh okra before osmotic treatment, g; M_f, final weight of okra after time t of osmotic treatment, g; m_o, dry weight of fresh okra, g; m_f, final dry weight of okra after time t of osmotic treatment, g.

Table 1: Experimental range and levels of the variables.

Dependent variable	-2	-1	0	+1	+2
Solute Concentration (X_1), w/w	35	45	55	65	75
Solution Temperature (X_2), oC	30	40	50	60	70
Size Diameter (X_3), mm	1	5	10	15	20
Process time (X_4)(hr)	0.67	2	4	6	8

2.2.2. Experimental design

For the optimization of osmotic dehydration process, the experiments were conducted according to a central composite rotatable design (CCRD) [29] with three variables at five levels each. The design was generated by Design Expert statistical software (version 6.08, Stat-Ease, Inc., Minneapolis, MN). For rotatable designs, the variances and covariance of the estimated coefficients in the fitted model remain unchanged when the design points are rotated about its centers. The independent variables were solution temperature, sucrose concentration, sample size (diameter) and process time. Each independent variable was coded at five levels between -1 and +1 (Table 1), including the centre point (0) and two axial points (-2 and +2) [29], and eleven triplicate combinations were performed, including seven replications of the centre point and a total of 31 experimental runs were generated (Table 2).The critical ranges of selected parameters were determined by preliminary experiments. The low and high levels in the actual (uncoded) form were taken as 45–65 (w/w), 40–60℃, 5–15mm and 2–6hrs for osmotic solution concentration, temperature, and sample size diameter and process time, respectively.

Data from the central composite experimental design were subjected to the following second-order polynomial regression analysis using least square regression methodology to obtain the parameters of the mathematical models.

$$\begin{aligned} Y =& b_0 + b_1A + b_2B + b_3C + b_4D + b_{11}A^2 + b_{22}B^2 \\ &+ b_{33}C^2 + b_{44}D^2 + b_{12}AB + b_{13}AC + b_{14}AD \\ &+ b_{23}BC + b_{24}BD + b_{34}CD \end{aligned} \tag{3}$$

Where b's are constant regression coefficients; Y is the response (i.e. WL or SG, %); A, B, C and D are sucrose concentration (% w/w), temperature (℃), size diameter (mm) and process time (hr), respectively. Statistical significance of the terms in the regression equations was examined. Response surface plots were generated with the same software.

3. Results and Discussion

3.1. Osmotic dehydration of Okra: water loss and solute gain

The results of the osmotic dehydration of okra with respect to water loss and solute gain are presented in Fig. 1. At sucrose: okra mass ratio of 10:1, each pair of experimental runs, run number 1 and 2; 3 and 4; 5 and 6; 7 and 8; 9 and 10; 11 and 12; 13 and 14; 15 and 16; 17 and 18 had the same process conditions with different solute concentration; results shows that increase in solute concentration increased the amount of water loss and solute gain by the sliced okra. This observation is due to the fact that an increase in solute concentration accentuates the osmotic pressure gradient between the fruit and the solution, hence establishing an enhanced driving force for mass transfer[25, 30].Similar results have been reported for other fruits and vegetables [24, 25, 30–33]. Low osmotic solution concentration implies a lower process driving force and subsequently longer treatment time. On the other hand; run numbers 18 and 25 through similar osmotic process condition but with higher solute concentration were tested and the results shows that extra higher solute concentration can decrease the amount of water loss and sugar gain. This behaviour may probably be due to high viscosity of the concentrated sucrose concentration and formation of a superficial layer of solutes on the product (case hardening effect), which makes circulation and mass transfer difficult [16, 24, 30]. As reported by Raoult-Wack [14], such barriers impair both the gain and loss of solutes by the product. Giraldo et al. [34] studied the mass transfer during osmotic dehydration of mango. The processes were carried out at 30℃, using 35° Brix, 45° Brix, 55° Brix and 65° Brix sucrose. They reported that water transfer rate increased when the concentration of sucrose increased up to 45° Brix, whereas, this effect did not appear between 55° Brix and 65° Brix. In general, highly concentrated osmotic solutions have shown an enhancement in water loss and solutes uptake during osmotic process.

Effect of different solution temperature were investigated at the same process condition of solute concentration, size diameter and process time for each pair of experimental runs (run numbers 1 and 3; 2 and 4; 5 and 7; 6 and 8; 9 and 11; 10 and 12; 13 and 15; 14 and 16; 19 and 20) and the findings demonstrated that increase in solution temperature resulted in increased water loss and solute gain. This result is in accordance with those of Germer et al. [24], Pereira et al. [30], Abud-Archila et al. [31], Ispir and Togrul [32] and Mundada et al. [35] who correspondingly studied the osmotic dehydration of apple, pineapple, mango, tropical fruit, water melon, yam bean, apricot, peaches and pomegranate arils, respectively. A rise in temperature usually accelerates and increases

Table 2: Experimental runs generated by Central Composite Rotable Design (CCRD) for osmotic dehydration of okra.

Run	**Type**	**Concentration** (w/w)		**Temperature** (°C)		**Size** (mm)		**Process Time** (hr)	
		Actual	Coded	Actual	Coded	Actual	Coded	Actual	Coded
1	Fact	45.00	(-1)	40.00	(-1)	5.00	(-1)	2.00	(-1)
2	Fact	65.00	(+1)	40.00	(-1)	5.00	(-1)	2.00	(-1)
3	Fact	45.00	(-1)	60.00	(+1)	5.00	(-1)	2.00	(-1)
4	Fact	65.00	(+1)	60.00	(+1)	5.00	(-1)	2.00	(-1)
5	Fact	45.00	(-1)	40.00	(-1)	15.00	(+1)	2.00	(-1)
6	Fact	65.00	(+1)	40.00	(-1)	15.00	(+1)	2.00	(-1)
7	Fact	45.00	(-1)	60.00	(+1)	15.00	(+1)	2.00	(-1)
8	Fact	65.00	(+1)	60.00	(+1)	15.00	(+1)	2.00	(-1)
9	Fact	45.00	(-1)	40.00	(-1)	5.00	(-1)	6.00	(+1)
10	Fact	65.00	(+1)	40.00	(-1)	5.00	(-1)	6.00	(+1)
11	Fact	45.00	(-1)	60.00	(+1)	5.00	(-1)	6.00	(+1)
12	Fact	65.00	(+1)	60.00	(+1)	5.00	(-1)	6.00	(+1)
13	Fact	45.00	(-1)	40.00	(-1)	15.00	(+1)	6.00	(+1)
14	Fact	65.00	(+1)	40.00	(-1)	15.00	(+1)	6.00	(+1)
15	Fact	45.00	(-1)	60.00	(+1)	15.00	(+1)	6.00	(+1)
16	Fact	65.00	(+1)	60.00	(+1)	15.00	(+1)	6.00	(+1)
17	Axial	35.00	(-2)	50.00	(0)	10.00	(0)	4.00	(0)
18	Axial	75.00	(+2)	50.00	(0)	10.00	(0)	4.00	(0)
19	Axial	55.00	(0)	30.00	(-2)	10.00	(0)	4.00	(0)
20	Axial	55.00	(0)	70.00	(+2)	10.00	(0)	4.00	(0)
21	Axial	55.00	(0)	50.00	(0)	1.00	(-2)	4.00	(0)
22	Axial	55.00	(0)	50.00	(0)	20.00	(+2)	4.00	(0)
23	Axial	55.00	(0)	50.00	(0)	10.00	(0)	0.67	(-2)
24	Axial	55.00	(0)	50.00	(0)	10.00	(0)	8.00	(+2)
25	Center	55.00	(0)	50.00	(0)	10.00	(0)	4.00	(0)
26	Center	55.00	(0)	50.00	(0)	10.00	(0)	4.00	(0)
27	Center	55.00	(0)	50.00	(0)	10.00	(0)	4.00	(0)
28	Center	55.00	(0)	50.00	(0)	10.00	(0)	4.00	(0)
29	Center	55.00	(0)	50.00	(0)	10.00	(0)	4.00	(0)
30	Center	55.00	(0)	50.00	(0)	10.00	(0)	4.00	(0)
31	Center	55.00	(0)	50.00	(0)	10.00	(0)	4.00	(0)

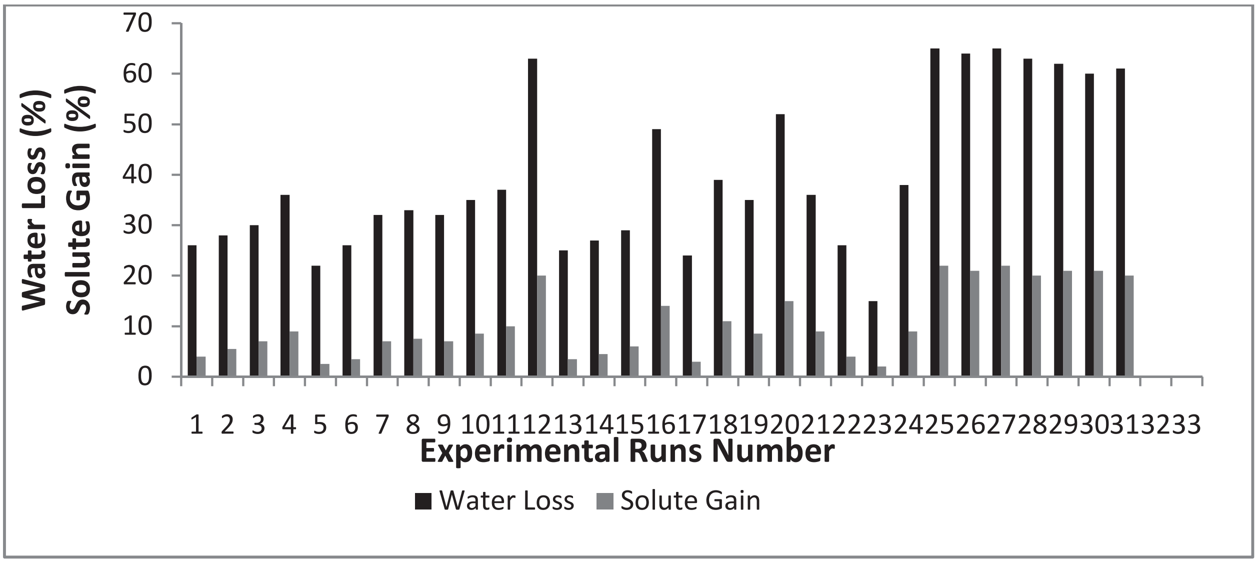


Figure 1: Osmotic dehydration of okra.

mass transfer. Higher temperatures promote faster water loss and gradual increase in solids absorption 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 [30, 36]. Thus, high temperature would release trapped air from the okra tissue resulting in more effective removal of water by osmotic pressure. Relatively run numbers 1 and 5; 2 and 6; 9 and 13; 10 and 14had the same process operating condition with different size diameter; finding shows that increase in size diameter led to decreased amount of water loss and solute gain. A similar observation has been reported for osmotic drying of apple slices of different sizes [37]. Antonio et al. [38] has reported that fruit and vegetables which are sliced provided higher water loss and solute gain than those with cubic geometry.

Furthermore, runs number 1 and 9; 2 and 10; 3 and 11; 4 and 12; 5 and 13; 6 and 14; 7 and 15; 8 and 16 had the same process conditions for osmotic dehydration but carried out at different process time; the results revealed that the amount of water loss and solute gain increased with increase in process time. This is in agreement with the observation of earlier workers who studied the osmotic dehydration of pineapple, cashew apple and melon [33, 37, 38]. In the most intense processing condition (65% w/w, 60°C, and 5mm for 6 hours), water loss and solute gain attained 63 and 20 (g/100 g of initial fresh fruit), respectively. Longer process time has been found to increase shrinkage, sweetness and overall acceptability of the final product [25]. In general, the experimental values for water loss and solutes gain under different treatment conditions (Fig. 1) showed that the water removal was always higher than the osmotic agent uptake, in agreement with the results of other workers [36, 38].

3.2. *Second order polynomial regression model and statistical analysis*

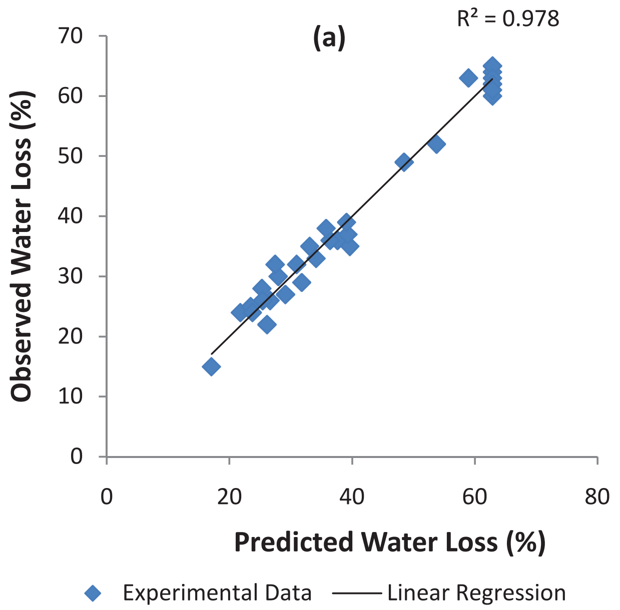
The experimental data were fitted to a second order polynomial regression model (Equation 3) containing 4 linear, 4 quadratic and 5 interaction terms [39] using the same experimental design software to derive the equation for water loss and sugar gain, respectively. The significance of each coefficient in the equation was determined by Student t-test and P-values. F-test indicated that all the factors and interactions considered in the experimental design are statistically significant ($P < 0.05$) at the 95 per cent confidence level. The regression equation obtained after analysis of variance gives the level of water loss (equation 4) and solute gain (equation 5) as a function of the different osmotic dehydration process variables: solute concentration, temperature, size diameter and process time (immersion time). All terms regardless of their significance are included in the following equations:

$$\begin{aligned} Y_{WL} = {} & 62.86 + 3.83A + 5.17B - 2.75C + 4.67D \\ & - 7.86A^2 - 4.86B^2 - 7.99C^2 - 9.11D^2 + 2.75AB \\ & - 0.75AC + 2.50AD + 0.00BC + 1.75BD - 1.75CD \end{aligned} \tag{4}$$

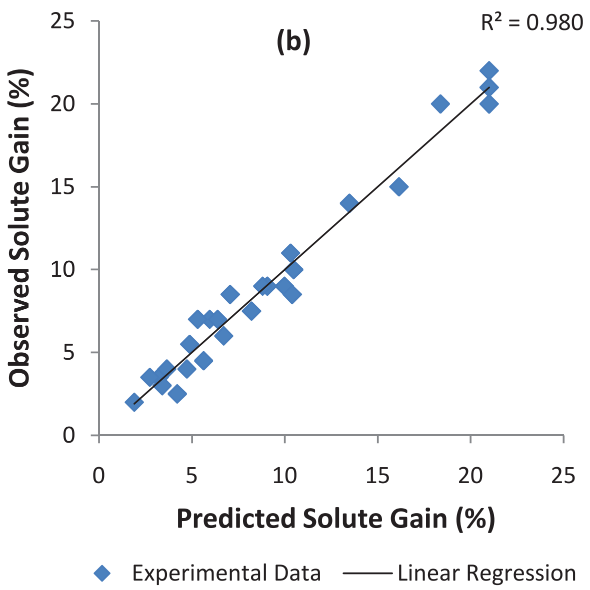
$$\begin{aligned} Y_{SG} = {} & 21 + 1.73A + 2.27B - 1.35C + 1.73D - 3.54A^2 \\ & - 2.35B^2 - 3.66C^2 - 3.91D^2 + 0.97AB - 0.28AC \\ & + 0.97AD - 0.031BC + 0.72BD - 0.78CD \end{aligned} \tag{5}$$

Where A is solute concentration, B is temperature; C is size diameter and D is process time.

To test the fit of the model, the regression equation and determination coefficient (R^2) were evaluated (Table 3). The model F-value of 52.14 and 58.82 implies the model is significant for WL and SG, respectively. There is only a 0.01 per cent chance that a model F-value, this large could occur due to noise alone for WL and SG, respectively. The low probability value (< 0.0001) for both WL and SG indicates that the model is significant. The value of the determination coefficient ($R^2 = 0.9786$ for WL and 0.9809 for SG) being a measure of goodness of fit to the model indicates high degree of correlation between the observed value and predicted values (Fig. 2). The determination coefficient(R= 0.9892 for WL and 0.9904 for SG), suggests that more than 98.92 and 99.04 per cent of the variance is attributable to the variables and indicated a high significance of the model for WL and SG, respectively. Thus, 1.08 and 0.96 per cent of the total variance cannot be explained by the model for WL and SG, respectively. The fitted model is considered adequate if the F-test is significant ($P < 0.05$). The analysis of variances (ANOVA) quadratic regression model demonstrated that the model was highly significant for both WL and SG, as was evident from the very low probability ($P < 0.0001$) of the F -test and insignificant result from the Lack of Fit model (Table 3). The Lack of Fit test is performed by comparing the variability of the current model residuals to the variability between observations at replicate settings of the factors. The Lack of Fit is designed to determine whether the selected model is adequate to describe the observed data, or whether a more complicated model should be used. The Predicted R-Squared value of 0.8902 and 0.9026 for WL and SG, respectively, is correspondently in reasonable agreement with the Adjusted R-Squared value of 0.9598 and 0.9643. Adequate Precision measures the signal to noise ratio. A ratio > 4 is desirable. The ratio of 21.011 and 21.150 obtained for WL and SG, respectively, in this research indicates an adequate signal. This model can be used to navigate the design space. The coefficient of variation (CV) as the ratio of the



(a)



(b)

Figure 2: A plot of observed values versus predicted values for (a) water loss, and (b) solute gain.

standard error of estimate to the mean value of the observed response is a measure of reproducibility of the model, generally a model can be considered reasonably reproducible if its CV is not greater than 10 per cent. Hence, the low variation coefficient value (CV =7.87 for WL and 12.27 per cent for SG) obtained indicates a high precision and reliability of the experiments.

The coefficient of the model (parameter estimation) and the corresponding P-values are presented in Table 4.The significance of regression coefficients was considered, ignoring those with an insignificant effect on the response at a significance level of 95 per cent. The P-values of the regression coefficients suggest that among the independent test variables, linear, quadratic and interaction effects of solute concentration, temperature and process time are highly significant. The insignificant effects (factors and interactions) with P-values higher than 0.05were ignored. In this study, A, B, C, D, A^2, B^2, C^2, D^2, AB, AD, BD and CD are significant model terms for WL and SG, respectively. Thus, statistical analysis of all the experimental data showed that solute concentration, temperature, size diameter and process time had a significant effect on water loss and solute gain in this study. Moreover, it is observed that solute concentration; solution temperature and process time exerted more pronounced linear effect (higher coefficient values) on water loss and solute gain, respectively. That is, water loss and solute gain was mostly and positively influenced by temperature, followed by process time and solute concentration. While the size diameter negatively influenced rate of water loss and solute uptake. The strong influence of temperature on osmotic dehydration has been clearly shown before in the previous works of Lenart and Flink [40], Lazarides et al. [36] and Antonio et al. [9] using other products and geometry. A similar observation has been reported for sucrose concentration and solution temperature on water loss and solute gain for pumpkin osmotic dehydration [25]. The quadratic effect of the independent variables on the rate of water loss and solute uptake was negative.

3.3. Influence of factors interaction on water loss and solute gain

Table 4 showed that water loss and solute gain were influenced positively by interaction of solute concentration (A) and solution temperature (B); solute concentration (A) and process time (D); solution temperature (B) and process time (D). While WL and SG were negatively influenced by the interaction of size diameter (C) and process time (D). However, there was no interaction between solute concentration (A) and size diameter (C), and solution temperature (B) and size diameter (C) as to impact on the rate of water loss and solute uptake, respectively.

The graphical representation of the response shown in Figs. 3 - 6 helped to visualize the effect of solute concentration (A), temperature (B), size diameter (C) and process time (D) on water loss and solute gain, respectively. The effect of interaction of solute concentration (A) and temperature (B) on water loss and solute gain is illustrated in Fig. 3. Fig. 3a shows that

Table 3: Analysis of variance (ANOVA) for the quadratic response surface model fitting to the osmotic dehydration data of okra.

Source	**SS**	**DF**	**MS**	$\mathbf{F_{-value}}$	**Prob > F(** $\mathbf{P_{-value}}$ **)**
WL: Model Residual	7160.48	14	511.46	52.14	< 0.0001
Lack of Fit	134.08	10	13.41	3.52	0.0685
Pure Error	22.86	6	3.81		
Correlation Total	7317.42	30			R^2 = 0.9786; Adj R^2 = 0.9598. Predicted R^2 = 0.8902; Adequate Precision = 21.011
SG: Model Residual	1388.57	14	99.18	58.82	< 0.0001
Lack of Fit	22.98	10	2.30	2.35	0.0716
Pure Error	4.00	6	0.67		
Correlation Total	1415.55	30			R^2 = 0.9809; Adj R^2 = 0.9643. Predicted R^2 = 0.9026; Adequate Precision = 21.150

WL= water loss; SG = solute gain; SS = sum of square; DF = degree of freedom; MS = Mean square

Table 4: Coefficient of the model for osmotic dehydration of okra.

Factor	**Coefficient Estimate**	**Standard Error**	$\mathbf{F_{-value}}$	$\mathbf{P_{-value}}$	**Remarks**
WL: b0	62.86	1.18	52.14	< 0.0001	Significant
b1	3.83	0.64	35.95	< 0.0001	Significant
b2	5.17	0.64	65.32	< 0.0001	Significant
b3	-2.75	0.64	18.50	0.0005	Significant
b4	4.67	0.64	53.29	< 0.0001	Significant
b11	-7.86	0.59	180.11	< 0.0001	Significant
b22	-4.86	0.59	68.86	< 0.0001	Significant
b33	-7.99	0.59	185.89	< 0.0001	Significant
b44	-9.11	0.59	241.95	< 0.0001	Significant
b12	2.75	0.78	12.34	0.0029	Significant
b13	-0.75	0.78	0.92	0.3524	Not significant
b14	2.50	0.78	10.19	0.0057	Significant
b23	0.00	0.78	0.00	1.0000	Not significant
b24	1.75	0.78	5.00	0.0400	Significant
b34	-1.75	0.78	5.00	0.0400	Significant
SG: b0	21.00	0.49	58.82	< 0.0001	Significant
b1	1.73	0.27	42.56	< 0.0001	Significant
b2	2.27	0.27	73.40	< 0.0001	Significant
b3	-1.35	0.27	26.10	0.0001	Significant
b4	1.73	0.27	42.56	< 0.0001	Significant
b11	-3.54	0.24	212.09	< 0.0001	Significant
b22	-2.35	0.24	93.57	< 0.0001	Significant
b33	-3.66	0.24	227.35	< 0.0001	Significant
b44	-3.91	0.24	259.46	< 0.0001	Significant
b12	0.97	0.32	8.91	0.0088	Significant
b13	-0.28	0.32	0.75	0.3991	Not significant
b14	0.97	0.32	8.91	0.0088	Significant
b23	-0.031	0.32	0.0093	0.9245	Not significant
b24	0.72	0.32	4.90	0.0417	Significant
b34	-0.78	0.32	5.79	0.0285	Significant

maximum water loss occurred when osmotic treatment was conducted in the higher temperature and higher solute concentration. A similar effect was observed for solute gain (Fig. 3b), where it can be seen that increasing temperature and concentration of the osmotic solution also caused an increase in solute gain. Singh et al. [41] also reported a similar observation for solute gain in the osmotic dehydration of carrot; while they observed a negative interaction effect of concentration and temperature on water loss.

Fig. 4 shows the 3D response surface plot of the interaction effect between solute concentration and process time (immersion time). This three dimensional plot explains that both solute concentration and process time has individual impact on water loss and solute gain as the individual coefficient of both solute concentration and process time is positive and their interaction effect is positive. However, the impact of process time is more than solute concentration as the individual coefficient value is higher for process time than for solute concentration. Maximum water loss was obtained at higher solute concentration and higher process time (Fig. 4a). A similar effect was observed for solute gain (Fig. 4b). Further increase in the amount of solute concentration ($> 65\%$ w/w), a significant decrease in water loss and solute gain occurred. This suggests that at a fixed solution temperature, the solute concentration can be decreased and that of process time has to be increased for higher water loss and minimum solute gain. Singh et al. [41] also observed a significant positive interaction effect of time and concentration on water loss and solute gain in the osmotic dehydration of carrot.

The interaction effect of changing solution temperature and process time on the rate of water loss and solute gain is shown in Fig. 5. This plot demonstrates that both solution temperature and process time have positive mutual impact on the rate of water loss and solute gain during the osmotic dehydration process. At a fixed solute concentration and size diameter, it is observed that maximum water loss (Fig. 5a) and solute gain (Fig. 5b) was obtained at a higher temperature and a higher process time.

Finally, Fig. 6 shows the 3D response surface plot of the effect of interaction between size diameter and process time. This plot demonstrates that both size diameter and process time has individual impact on water loss and solute gain as the individual coefficient of size diameter is negative and process time is positive; while their interaction effect is negative. At a fixed solute concentration and solution temperature, it is observed that maximum water loss (Fig. 6a) and solute gain (Fig. 6b) was attained at a smaller size diameter and higher process time. According to Barbosa-Córnvas and Vega Mercado [42], the migration of solute in osmotic dehydration depends on the selectivity and permeability of the foods, contact time and size of the material.

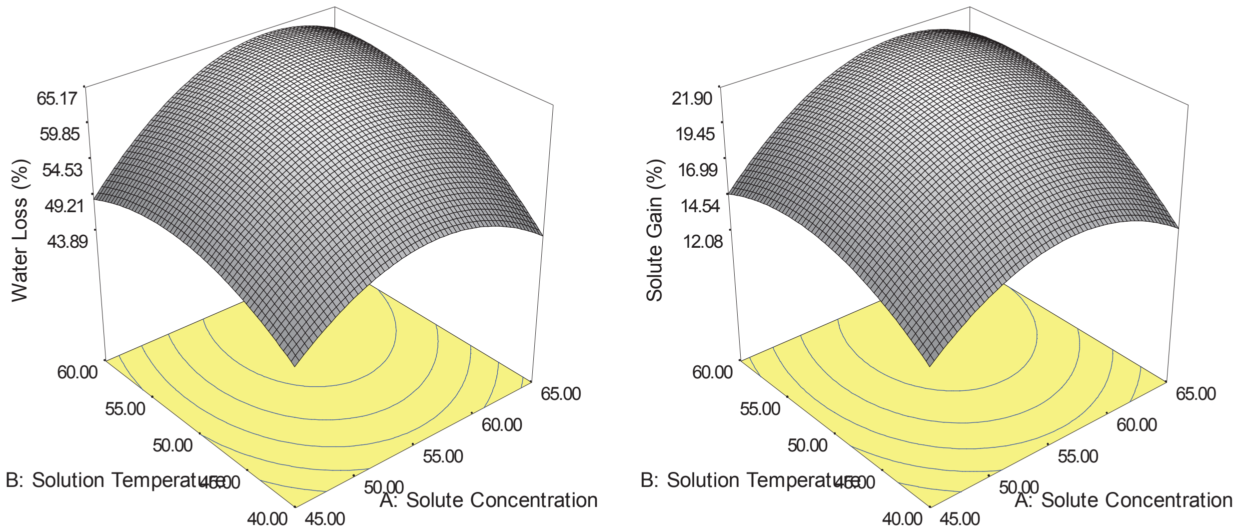
3.4. Factor plot

The factor effect function plot (Fig. 7) was used to assess the effect of each factor graphically. From the trace plot as shown in Fig. 7, it can be seen that each of the four variables used in the present study has its individual effect on water loss and solute gain in the osmotic dehydration of okra in sucrose solution. Gradual increase in solute concentration, solution temperature and process time from low level (coded value -1) to a higher level (coded value +1) resulted in the increase of both water loss and solute gain.

However, a gradual increase in size diameter of okra slices from low level to a higher level led to decrease in water loss and solute gain. Moreover, it is also to be noted from Fig. 6 that over the range of 40 to 60℃of solution temperature, the water loss and solute gain changed in a wide range. However, this was not the case for solute concentration and process time, respectively. This clearly indicates that keeping solute concentration and process time at the optimum level, a change in solution temperature will affect the water loss and solute gain process more severely than done otherwise.

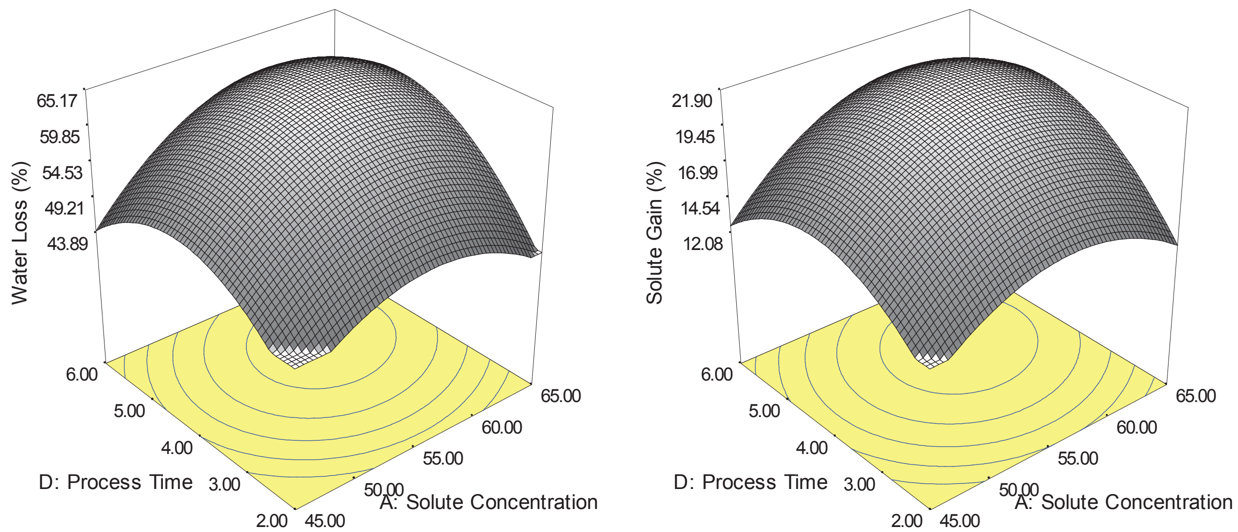
3.5. Optimized condition for osmotic dehydration of okra

The optimum coded and uncoded value was obtained by solving equation 3 using numerical method. Numerical optimization based on desirability function was carried out. In order to provide an ideal case for osmotic dehydration, the goal for solute concentration, solution temperature, size diameter and process time was set in range based upon the requirements of the okra osmo-dehydration and water loss was set on maximize, while solute gain was set on minimize. The optimum coded and uncoded values of solute concentration, solution temperature, size diameter and process time were found to be 49.28 (% w/w), 40.79℃, 15 mm and 4.49 hr respectively, to attain a maximum water loss of 39.78 (g/100 g initial sample) and minimum solute gain of 10.16 (g/100g initial sample). Desirability was 0.542 for the experiment (Fig. 8). Nevertheless, series of experiment (validation experiment) were conducted to determine the optimum water loss and solute gain when the process variables were set at the favourable predicted optimum levels established above, through CCRD and RSM. At these optimum conditions, the water loss and solute gain was observed to be 38.87 and 10.65g/100g of initial sample, respectively. The results clearly indicated that no significant difference was observed.



(a) (b)

Figure 3: Response surface plot for (a) water loss (b) solute gain as a function solute concentration and solution temperature at 10 mm size diameter and 4 hr process time.



(a) (b)

Figure 4: Response surface plot for (a) water loss (b) solute as a function of solute concentration and process time at 50°Csolution temperature and 10 mm size diameter.

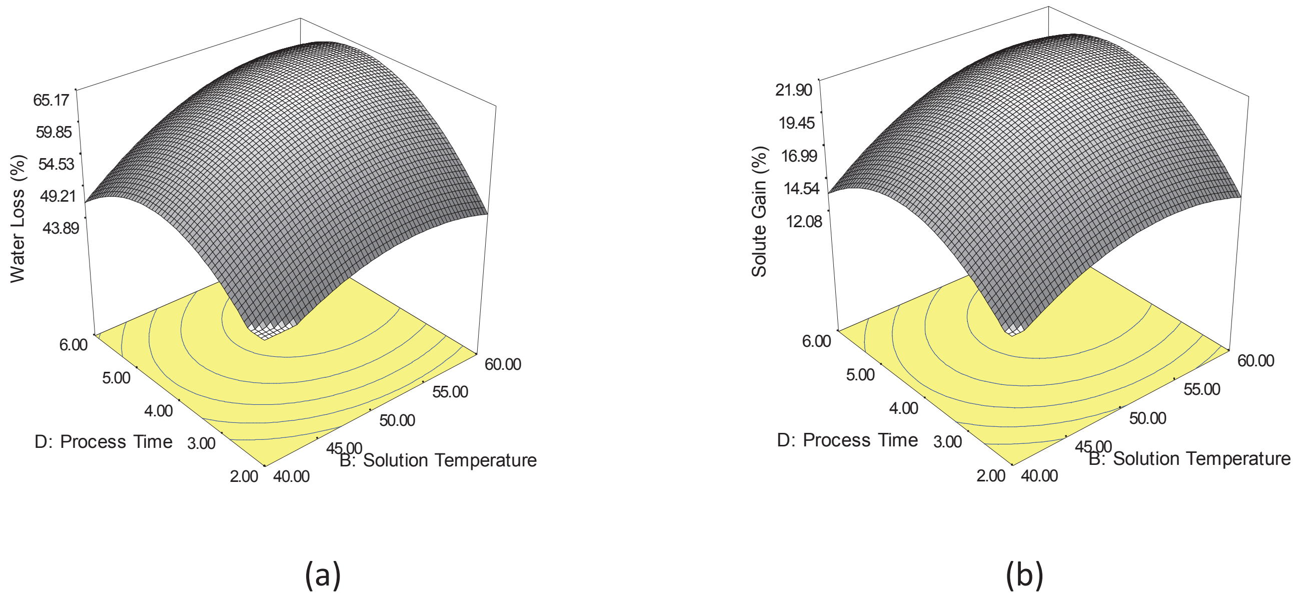


Figure 5: Response surface plot for (a) water loss and (b) solute gain as a function of solute temperature and process time at 55 (% w/w) solute concentrations and 10 mm size diameter.

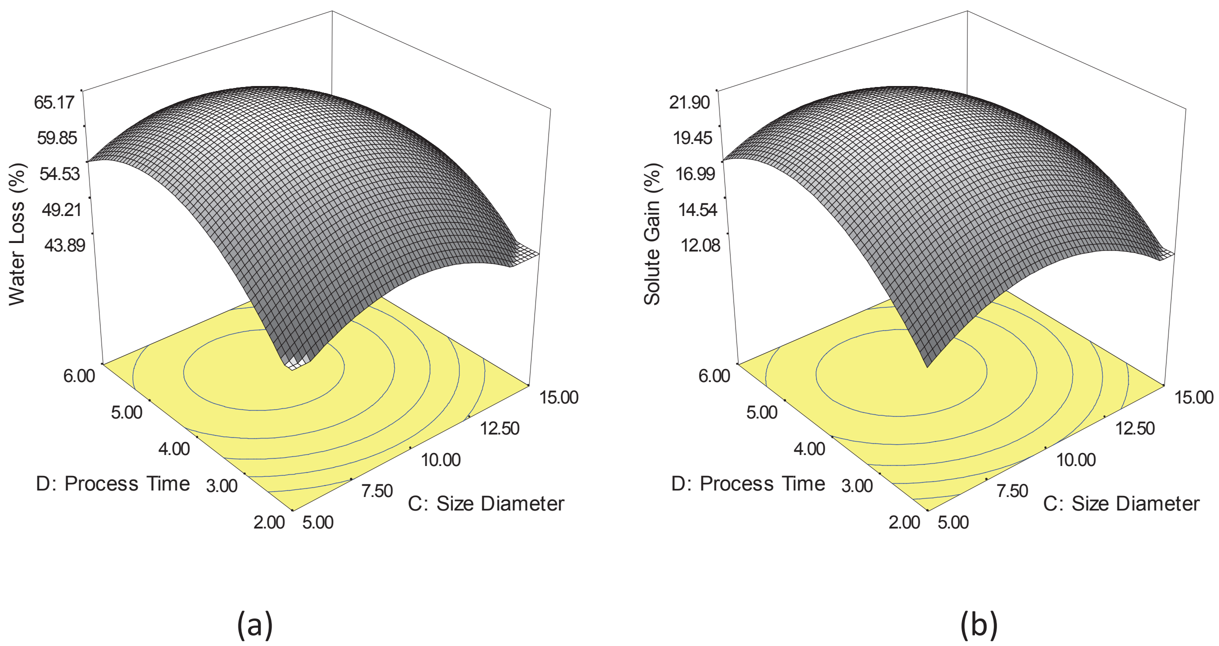


Figure 6: Response surface plot for (a) water loss and (b) solute gain as a function of size diameter and process time at 55 (% w/w) sucrose concentrations and 50°Csolution temperature.

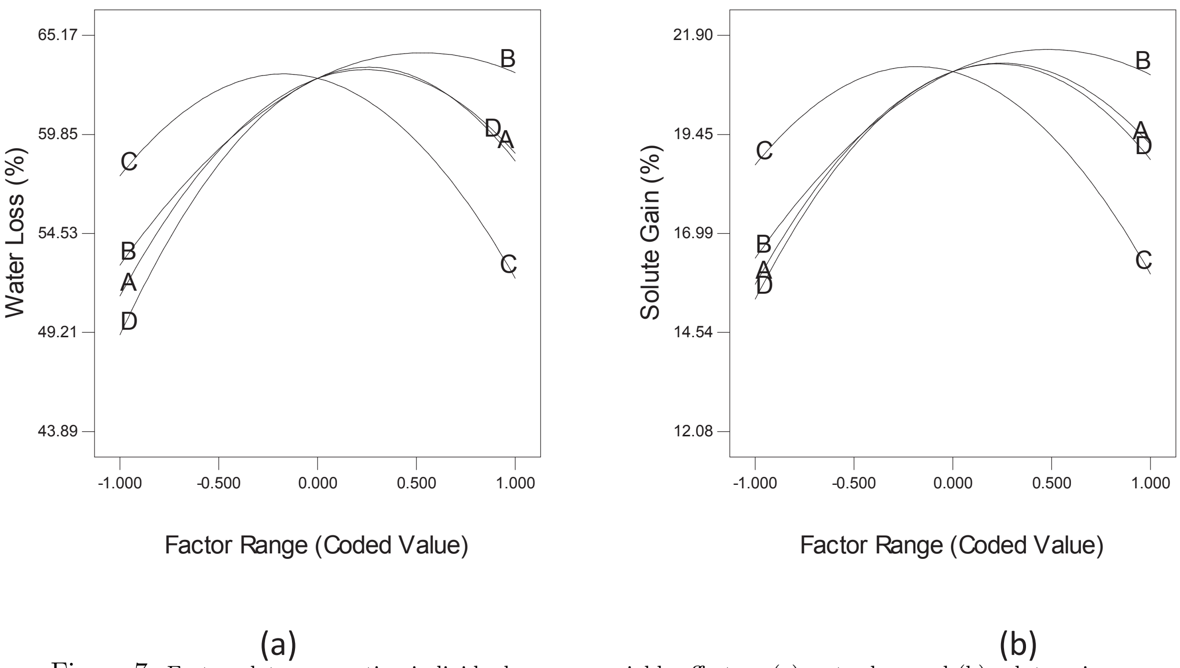


Figure 7: Factor plot representing individual process variable effect on (a) water loss and (b) solute gain.

4. Conclusions

This study demonstrated that the variables of temperature, solute concentration, and size diameter and process time had a strong influence on the rate of osmotic dehydration of okra slices using sucrose solution as an osmotic agent. The water loss and the solute gain during osmotic dehydration of okra slices were influenced positively by the temperature, solute concentration and process time and negatively by the sample diameter. The water loss and solute gain were influenced positively by interaction of the temperature and solute concentration, solute concentration and process time, temperature and process time and negatively by the interaction of solute concentration and size diameter, solution temperature and size diameter and size diameter and process time. The obtained quadratic regression models for the responses of water loss and solute gain were significant. Finally, the mathematical models obtained can also be employed to establish better process conditions. Considering an industrial process, it is understood that the best conditions are those combining the best water removal rates during the osmotic dehydration with the best sensory performances.

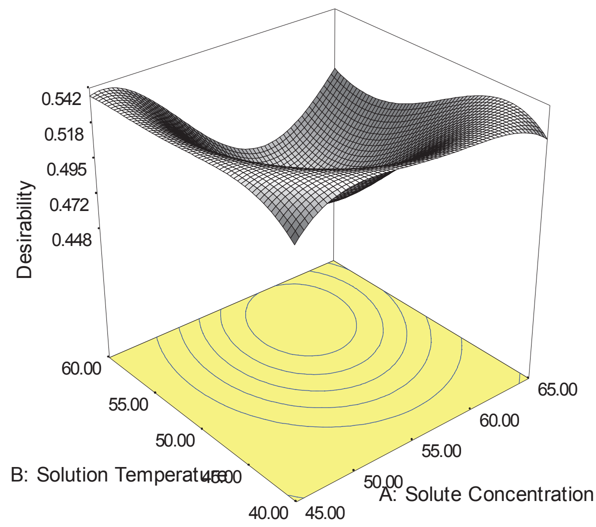


Figure 8: Desirability plot to optimize the osmotic dehydration process of okra.

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