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RESEARCH ARTICLE

A STUDY ON OPTIMIZATION OF PROCESS VARIABLES FOR OSMOTIC DEHYDRATION OF APPLE SLICES BY THE APPLICATION OF RESPONSE SURFACE METHODOLOGY.

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Abstract

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The Apple fruit (*Malus domestica*) is round in shape with firm juicy flesh and greenish, red or yellow in color when ripe. Nutritionally it is a good source of carbohydrate, dietary fiber and also contains vitamins, minerals and a variety of phytochemicals. The Response surface methodology (RSM) was used to optimize the influence of osmotic agents i.e. sucrose, glucose and glucose + sucrose(50:50), sugar concentrations (50˚, 55˚, 60˚, 65˚ and 70˚B), fruit to solution ratios (1:2,1:3,1:4,1:5,1:6) and process time varied from 60 to 180 min on osmotic dehydration of apple(golden delious) slices. In all the osmotic dehydration processes of apple slices, an increase in concentration, time duration up to certain duration and fruit to solution ratio resulted in higher water loss and solute gain. The osmotic agents, range of time, concentration and fruit to solution ratio are glucose + sucrose (50:50), 105-Min, 60˚B, and 1:4 (w/w), respectively for maximum water loss and solute gain was optimized.

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Introduction:-

The Apple fruit (*Malus domestica*) is round in shape with firm juicy flesh and greenish, red or yellow in color when ripe. Nutritionally it is a good source of carbohydrate, dietary fiber and also contains vitamins and minerals. Apples contain a variety of phytochemicals, including quercetin, catechin, phloridzin and chlorogenic acid, all of which are regarded as strong antioxidants (Jeanelle Boyer&Rui Hai Liu., 2004). Due to the nutritional content of the apple, it is an important part of human diet, it is often eaten raw, but can be used to make dessert, alcoholic and nonalcoholic beverages and more food products (Ewekeye T.S etal.2016)

Due to the improper post-harvest management there is wastage of fruits and vegetable. Post-harvest losses of fruits in general account for 20 - 25% of total fruit production in developed countries and even more in developing countries The huge wastage can be trimmed down by developing food processing industry and intensification of post-harvest infrastructure and filling the gap in the supply chain Fruit and vegetable processing is one major sector offering a large potential for exports. This industry is characterized by very high post-harvest losses. The losses range from 14-36% in the case of fruits and 10-25% in the case of vegetables (Lalsiemlienpulamte., 2008).

Thermal processing is generally used in the food industry for their efficacy and product safety. Excessive heat treatment may, however, cause undesirable changes in the food that deteriorate the sensory and nutritional

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characteristics of the final product. As consumers increasingly perceive fresh food as healthier than heat treated food, the industry is now seeking alternative non-thermal technologies to maintain most of the fresh attributes, safety and storage stability of food (Lado Beatrice & Yousef Ahmed.,2002). Water activity (aw) is the most important factor that affects the stability of dehydrated and dry products during storage. It is determinant for microbial growth and Influences degradation chemical reactions, non-enzymatic browning, enzymatic and physical nature (Maltini et al., 2003).

Osmotic dehydration (OD) is one of the food preservation methods where no heat treatment is involved, which is becoming an attractive complementary processing step in the chain of integrated food processing (Rastogi et al., 2002). OD involves immersing foods (fruit, vegetables, fish and meat) in a hypertonic (Osmotic) solution i.e. concentrated sugar, salt, alcohols or soluble starch solutions, which partially dehydrates the food (Mújica-Paz et al., 2003) This process also known as dewatering and impregnation soaking process (DIS process), can be used as a pretreatment before any complementary processing, and may lead to energy savings and quality improvement. This process improves and/or preserves nutritional properties, functional properties, reduces the time needed for dehydration and it is simple, and equipment and operation costs are low. (Moreno et al., 2000; Moreira et al., 2003)

As a pretreatment osmotic dehydration effectively improved the nutritional, sensorial and functional properties of the products.When compared to other dehydration methods, the unique aspect of this process, is the 'direct formulation' achievable through the selective incorporation of solutes, without modifying the food integrity. By balancing the two main osmotic effects, water loss and soluble solids uptake, the functional properties of fruit and vegetables could be adapted to many different food systems. (Danila Torreggiani 1993).

Keeping all these point in view the aim of the present study is to investigate the optimization of the processing variables to maximize the overall acceptability of the product by using RSM (Response Surface Methodology).

Material & Methods:-

The apple of golden delicious variety used in present investigation was obtained from the local market and selected for the experiments according to a similar size and ripeness. Their initial moisture content, M_0, was $84 \pm 0.5\%$, expressed on dry basis.

Cutting the apple slices:-

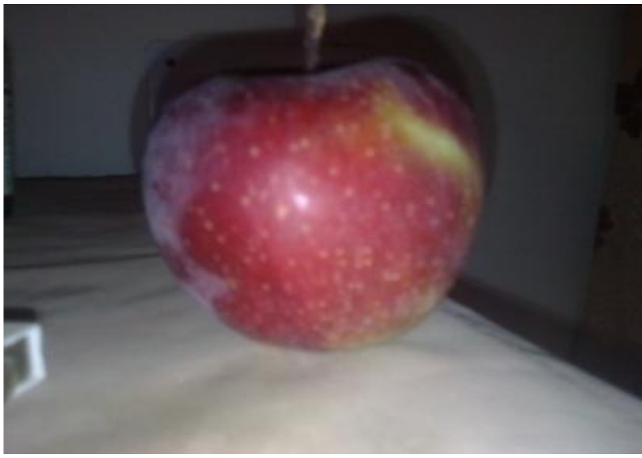
Apple was washed, skinned, trimmed and then with the help of sharp knife (stainless steel) they are cut into thin circular slices of 0.5 -0.7 cm thickness (approximately).

Chemicals and Reagents:-

All the reagents and chemicals used for the study were of Analar grade and procured from M/s Sigma Chemicals, Corporation, USA and M/s BDH Company.

Osmotic Concentration of Apple slices:-

Osmotic Concentration of apple slices was done in the solutions prepared by using sucrose , glucose and glucose +sucrose (50:50) as osmotic agents to make Osmotic solution of 50◦, 55◦, 60◦, 65◦ and 70◦brix respectively. Thereafter apple slices were dipped into the Osmotic solution made from sucrose, glucose and glucose +sucrose (50:50) of 50◦, 55◦, 60◦, 65◦ and 70◦brix respectively. The ratio of the sample (apple slice) to the osmotic solution was 1:2, 1:3, 1:4 .1:5 and 1:6 respectively, the process time from 0 to 180 min.



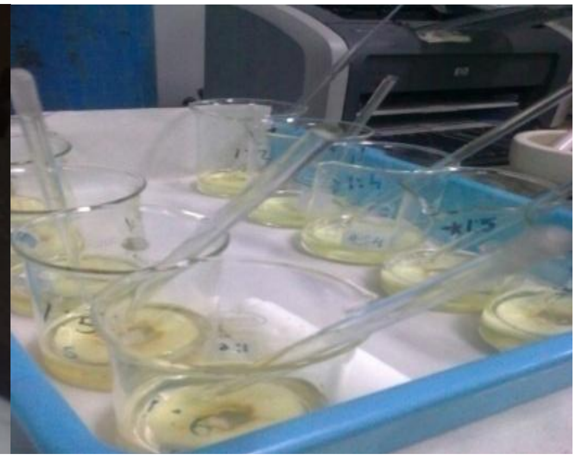


Figure 1:-Apple of variety Red Delicious which is used for osmotic dehydration.
Figure 2:- Process showing osmotic dehydration of apple slices.

Estimation of dry matter and Moisture content of apple slices (AOAC, 1995):-

The samples were oven dried at 103±2°C for16 hrs in uncovered pre-weighed Petri dishes (AOAC, 1995). After drying, Petri dishes were covered with lid and cooled in desiccators containing silica gel for 1 hour before weighing. Moisture content of the whole sample was calculated by;

Solid content/gm sample before osmotic Concentration = Z

Weight of the fruit before osmotic Concentration =M_o (g)

Weight of the fruit after osmotic Concentration for fruit at any specified time (t) =M (g)

Dry matter of fruit after osmotic Concentration for any specified time (t) = m_t (g)

Dry matter (m_o) = Z*M_o

Moisture content (dry basis) = $\frac{M - m_t}{m_t}$

Calculation of Water Loss and Solute Gain during osmotic Concentration:-

Weight reduction (WR) = $M_o - M(g)$

Solid gain = $m_t - m_o$

Solid gain/100 gm fruit sample= $\frac{SG(g) \times 100}{M_o}$

Water loss (WL,g) = $WR + SG(g)$

Water loss/100 gm fruit sample= $\frac{WL(g) \times 100}{M_o}$

Table 1:-Variables in RSM Design.

Variables	**Levels used**	
Independent variable	Low	High
Time(X_4)	30	180
Concentration(X_2)	50	70
Glucose : Sucrose (X_1)	0	100
Fruit to solution ratio(X_3)	2	6
Dependent variable		
Water loss (Y_1)		
Solute gain(Y_2)		
Water loss (WL) Y1		
Solid gain (SG) Y2		

Results and Discussion:-

Optimization of osmotic dehydration process by using Response Surface Methodology:-

Response surface methodology (RSM) was used to estimate the main effects of osmotic dehydration process on water loss (WL) and solid gain (SG) in apple slices. A face centered design was used with glucose: sucrose (%), solution concentration (50-70°B), Fruit to solution ratio (1:2 to 1:6) and immersion time (30–180 minutes) being the independent process variables. The RSM was applied to the experimental data using a commercial statistical package, Design-Expert version 6.01 (Stat ease Inc., Minneapolis, USA). The following polynomial model was fitted to the data:

$$Y= bo + b_1X_1 +b_2X_2 + b_3X_3+ b_4X_4 + b_{11}X_1^2 + b_{22}X_2^{2} + b_{33}X_3^{2}+ b_{44}X_4^2+ b_{12}X_1X_2 + b_{13}X_1X_3+ b_{14}X_1X_4 + b_{23}X_2X_3+ b_{24}X_2X_4+ b_{34}X_3X_4 \quad (A)$$

Where b_n were constant regression coefficients; Y was the response (*i.e.* WL or SG, g/100g); X_1, X_2, X_3 and X_4 were glucose: sucrose (%), sugar solution concentration (^{0}B), Fruit to solution ratio (w/w) and time (min), respectively.

Table 2:- Experimental design for all responses for optimization of process of osmotic dehydration of apple slices.

S.No	Glucose: Sucrose(X_1)	Brix *(X2)*	F:S ratio *(X3)*(X_3)	Time *(X4)*	Water Loss (Y1)	Solid Gain (Y2)
1	0	50	2	30	21.18	2.94
2	100	50	2	30	23.32	3.16
3	0	70	2	30	21.21	3.30
4	100	70	2	30	21.46	4.08
5	0	50	6	30	20.57	3.34
6	100	50	6	30	22.48	4.44
7	0	70	6	30	22.08	3.30
8	100	70	6	30	25.77	5.23
9	0	50	2	180	37.08	9.10
10	100	50	2	180	50.23	13.58
11	0	70	2	180	44.22	12.41
12	100	70	2	180	54.19	15.60
13	0	50	6	180	40.97	10.89
14	100	50	6	180	51.03	14.08
15	0	70	6	180	45.02	12.86
16	100	70	6	180	55.04	16.07
17	0	60	4	105	37.33	8.95
18	100	60	4	105	43.55	11.09
19	50	50	4	105	37.77	9.16
20	50	70	4	105	39.25	10.74
21	50	60	2	105	37.34	9.91
22	50	60	6	105	38.82	10.37
23	50	60	4	30	22.02	4.56
24	50	60	4	180	47.64	13.28
25	50	60	4	105	37.89	10.54
26	50	60	4	105	38.06	11.35
27	50	60	4	105	39.78	12.05
28	50	60	4	105	38.78	11.89
29	50	60	4	105	36.55	10.56
30	50	60	4	105	36.94	9.23

Diagnostics checking of fitted Model:-

Regression analysis for different models indicated that the fitted quadratic models accounted for more than 95% of the variation in the experimental data, were found to be more significant. Multiple regression equation was generated relating water loss and solid gain to coded levels of the variables.

Models were developed as follows:-

$WL = +38.44 +3.19 * X_1 +1.31 * X_2 +0.64 * X_3 +12.52 * X_4 +1.57 * X_1^2 -0.36 * X_2^2 \ 0.8 * X_3^2 -4.05 * X_4^2 -0.21* X_{12} +0.011* X_{13} +2.20 * X_{14} +0.22 * X_{23} +1.01 * X_{24} +0.16 * X_{34}$

$SG = +10.63 +1.12 * X_1 +0.72 * X_2 +0.36* X_3 +4.64 * X_4 -0.31 * X_1^2 -0.37 * X_2^2 -0.19 * X_3^2 -1.40 * X_4^2 +7.714E-003* X_{12} +0.047* X_{13} +0.63 * X_{14} -0.12* X_{23} +0.45 * X_{24} +0.023* X_{34}$

The experimental values for water loss and solid gain under different treatment conditions are presented in Table 2. Regression equations describing the effect of osmotic dehydration variables on the water loss (WL) and solid gain (SG) of apple slices are given in Table 3 & 4. During the experiment high correlation coefficients (i.e. R^2) were obtained for both responses indicating good fit of experimental data to Equation. The ANOVA also showed that lack of fit was not significant for both responses at P=5% level

Table 3:- Regression summery and ANOVA table for water loss for coded Values of process variables

Source	df	Mean Square	Sum of Squares	F- value	p-level	
Model	14	231.89	3246.40	154.65	< 0.0001	Significant
X_1	1	183.15	183.15	122.15	< 0.0001	
X_2	1	30.95	30.95	20.64	0.0004	
X_3	1	7.43	7.43	4.96	0.0417	
X_4	1	2820.98	2820.98	1881.40	< 0.0001	
X_1^2	1	6.35	6.35	4.24	0.0574	
X_2^2	1	0.35	0.35	0.23	0.6384	
X_3^2	1	1.64	1.64	1.10	0.3119	
X_4^2	1	42.43	42.43	28.30	< 0.0001	
X_{12}	1	0.69	0.69	0.46	0.5079	
X_{13}	1	0.00	0.00	0.00	0.9722	
X_{14}	1	77.43	77.43	51.64	< 0.0001	
X_{23}	1	0.81	0.81	0.54	0.4744	
X_{24}	1	16.38	16.38	10.92	0.0048	
X_{34}	1	0.43	0.43	0.28	0.6022	
Residual	15	1.50	22.49			
Lack of Fit	10	1.55	15.47	1.10	0.4868	Not significant
Pure Error	5	1.40	7.02			
R^2		0.9931				
Adj R^2		0.9867				

All main effects linear and quadratic were calculated for each model. The regression coefficients are shown in Table 3 & 4, as well as the coefficient of determination obtained for both models. The coefficient of determination for water loss and solid gain (R^2= 0.9931 and R^2= 0.9820, respectively) are quite high for response surfaces.

Table 4:-Regression summery and ANOVA table for solid gain for coded values of process variables.

Source	df	Mean Square	Sum of Squares	F- value	p-level	
Model	14	33.07	462.97	58.40	< 0.0001	Significant
X_1	1	22.76	22.76	40.20	< 0.0001	
X_2	1	9.27	9.27	16.36	0.0011	
X_3	1	2.34	2.34	4.13	0.0602	
X_4	1	387.43	387.43	684.15	< 0.0001	
X_1^2	1	0.25	0.25	0.44	0.5193	
X_2^2	1	0.36	0.36	0.64	0.4371	
X_3^2	1	0.09	0.09	0.16	0.6970	
X_4^2	1	5.11	5.11	9.02	0.0089	
X_{12}	1	0.00	0.00	0.00	0.9678	
X_{13}	1	0.04	0.04	0.06	0.8062	

X_{14}	1	6.29	6.29	11.11	0.0045	
X_{23}	1	0.23	0.23	0.40	0.5365	
X_{24}	1	3.30	3.30	5.82	0.0291	
X_{34}	1	0.01	0.01	0.01	0.9044	
Residual	15	0.57	8.49			
Lack of Fit	10	0.30	2.97	0.27	0.9636	Not significant
Pure Error	5	1.11	5.53			
R^2		0.9820				
Adj R^2		0.9652				

Analysis of variance:-

To evaluate the goodness of the model, F test were conducted. The F-values for water loss and solid gain were 73.75 and 35.61 respectively. On this basis, it can be concluded that the selected models adequately represent the data for water loss and solid gain of osmotic dehydration of apple slices.

The computer generated 3D surfaces were generated using regression equations, as shown in figure 3 to 8. The figure 3 - 5 shows the variation of water loss as a function of glucose: sucrose ratio and time, and as a function of immersion time and fruit to solution ratio. The water loss increased gradually with the sucrose solution over the entire osmotic dehydration process (Fig 5).

Conditions for optimum responses:-

Models were useful in indicating the direction in which to change variables in order to maximize water loss and solid gain. Therefore the multiple regression equation was solved for the maximum water loss and solid gain. The coded values for the optimum responses were first decoded into actual values as per the equations in Table 4 and then these values (b1….b3) were transformed into actual variables (A...C) by solving the algebraic equation as described in experimental design. The response surfaces are obtained by selecting two variables and the third variable has the value that lead to the optimum response in the equations y_1 and y_2. The surfaces are presented in Figs 3 - 7.

Diagnostic checking of fitted model and surface plots for water loss:-

The effect of various process parameters on water loss are indicated in Figs 3-5. The water loss varied from 20.57 to 55.04 g/100g with change in process parameters. Glucose: sucrose and immersion time has most significant effects in apple slice. Fig 3 shows that water loss increases with increase in brix and glucose to sucrose ratio, while Fig 4 shows that water loss increases with increase in immersion time and glucose to sucrose ratio. Water loss slightly increase with increase in fruit to solution ratio then starts decreasing (Fig 5).

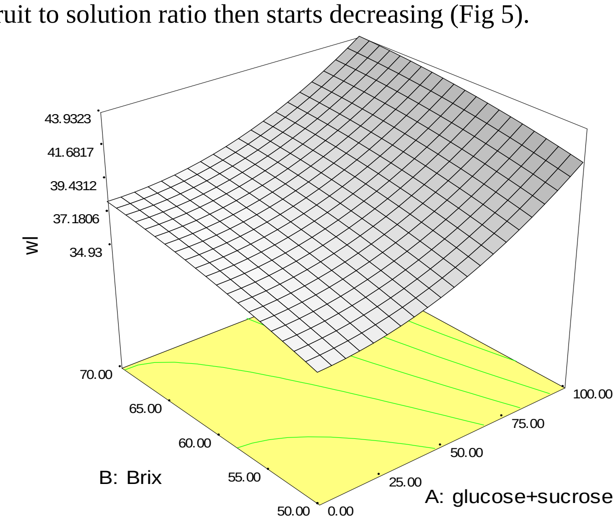


Figure 3:-Water loss during osmotic dehydration of apple slices as function of brix and glucose: sucrose ratio at fruit to solution ratio (1:4) and time (105 min).

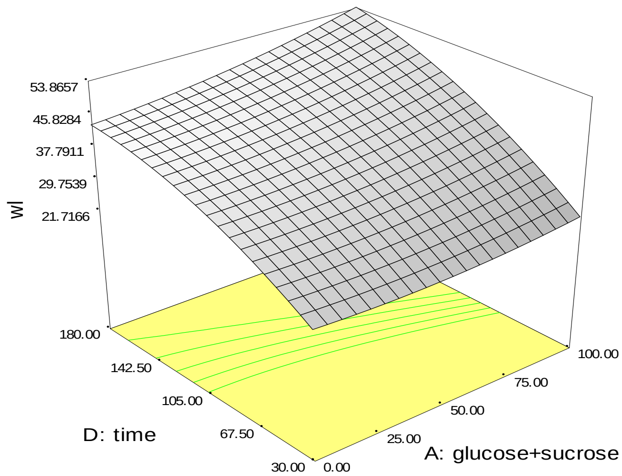


Figure 4:- Water loss during osmotic dehydration of apple slices as function ofimmersion time and glucose: sucrose at fruit to solution ratio (1:4) andsugar concentration (60˚B).

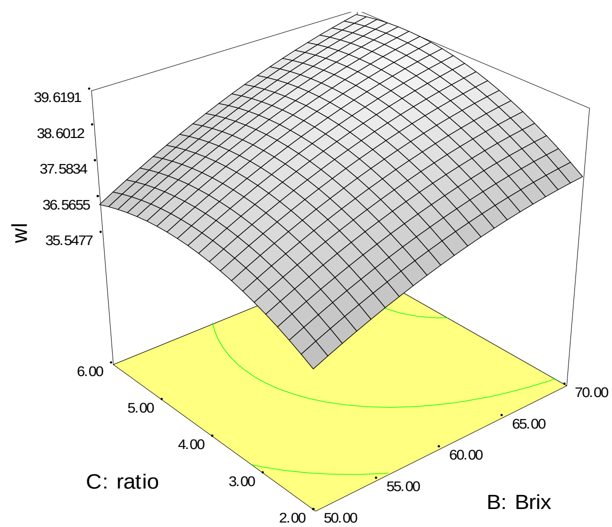


Figure 5:-Water loss during osmotic dehydration of apple slices as function offruit to solution ratio and sugar concentration at immersion time (105 min) and glucose: sucrose (50:50).

Diagnostic checking of fitted model and surface plots for solid gain:-

The effect of various process parameters on solid gain are indicated in fig 6-8. The solid gain varied from 2.93 to 16.06 g/100g with change in process parameters. Glucose: sucrose and immersion time has the most significant effect on solid gain in apple slice. Solid gain increases with increase in brix and glucose to sucrose ratio, time and glucose to sucrose ratio and fruit to solution ratio and brix as shown in Fig 6, 7 and 8.

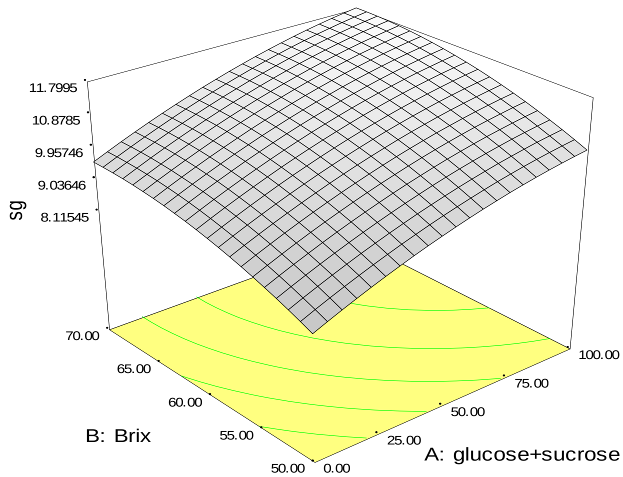


Figure 6:- Solid gain during osmotic dehydration of apple slices as function ofbrix and glucose: sucrose ratio at fruit to solution ratio (1:4) and time (105 min).

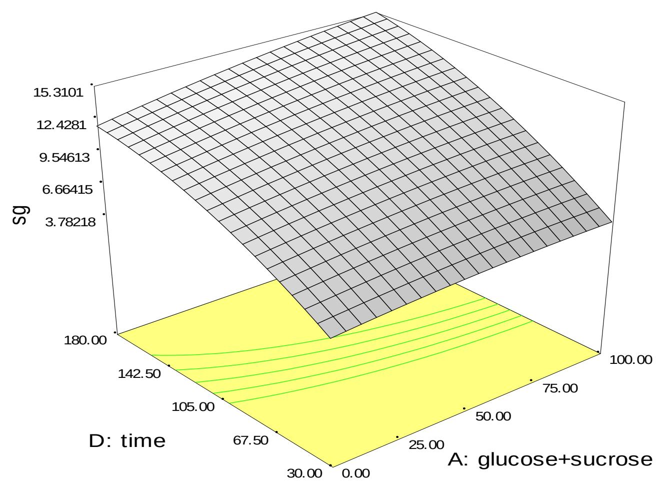


Figure 7:-Solid gain during osmotic dehydration of apple slices as function ofimmersion time and glucose: sucrose at fruit to solution ratio (1:4) andsugar concentration (60˚B).

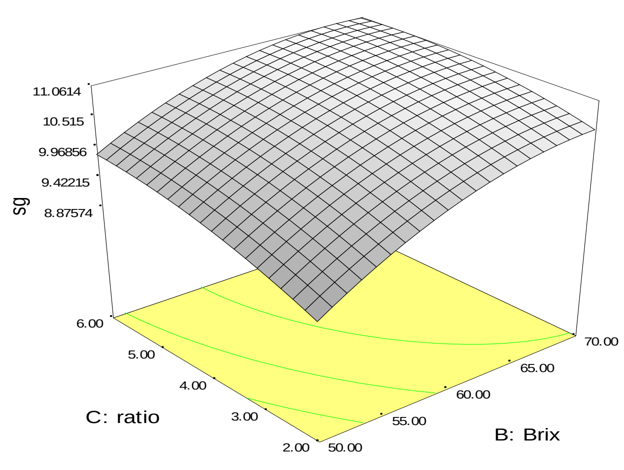


Figure 8:- Solid gain during osmotic dehydration of apple slices as function of fruit to solution ratio and sugar concentration at immersion time (105 min) and glucose: sucrose (50:50).

Optimization of the processing parameters to maximize overall acceptability of product:-
Design expert software was used to optimize the processing parameters like Glucose: sucrose, sugar concentration, fruit to solution ratio and time to maximize overall acceptability of product. The software uses second order model to optimize the responses. Table 5 showed constrains used for the optimization of processing parameters and Table 6 represents the optimized solution given by design expert.

Table 5:-Constraint selected in the range for optimization

Name	Goal	Operating conditions
Glucose: sucrose (%)	*Target*	0:100 < x_1< 100:0
Brix (^{0}B)	*Target*	*50 < x_2 < 70*
Fruit to solution ratio **Time (min)**	*Target* *target*	*1:2 < x_3 < 1:6* *30<x_4<180*
Water loss	*Maximum*	y_1
Solid gain	*Minimum*	y_2

Table 6:- Optimized level (in the range) and predicted optimum values.

Variables	Optimum Value	Responses	Predicted Value
Glucose: sucrose **Brix** **Fruit to solution ratio** **Time**	50:50 60 1:4 105	**Water loss** **Solid gain**	38.43 10.63

Conclusion:-
Response surface methodology was effective in identifying the optimum processing conditions for OD of apple slices using osmotic agent of glucose+sucrose (50:50), fruit to solution ratio 1:4 (w/w), concentration 60˚B, range of time 105-Min and these optimum condition reduced the original water content of the apple slices by about 38.43% and increased the solid gain by 10.63 %. Therefore, osmotic dehydration of apple slices could effectively be used as a pretreatment prior to conventional drying or freeze drying and helpful l to maintain the natural quality that is nutritional properties, sensory properties & functional properties of the product.

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