# Optimization of some process variables in mass transfer kinetics of osmotic dehydration of pineapple slices

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**Abstract:** Response Surface Methodology was used with four factors on three levels to find out the optimum osmotic concentration, temperature, slice thickness and KMS concentration for better OD of pineapple slices. The slices of various thickness (3, 6, 9 mm) were dipped into various sugar concentration (50°B, 60°B, 70°B) with temperature (35°C, 45°C, 55°C) for six hours. KMS (0.025%, 0.05%, 0.075%) was added into the sucrose solution. Computer generated response surfaces, contour plot and ANOVA revealed that WL and SG increased linearly with the increase in sugar concentrations and temperatures of the solution. Optimization of parameters revealed that slice thickness and KMS percentage had least effect on mass transfer of slices. Optimum osmotic dehydration corresponded to 58-63°Bx sugar concentration, 55°C temperature, 6 mm slice thickness and 0.05 -0.065% KMS concentration. Regression analysis showed that the process variables have significant effect on osmosis.

Keywords: pineapple, osmotic dehydration, RSM, solid gain, water loss, weight reduction

## Introduction

Pineapple (Ananas comosus) is one of the most common tropical and subtropical fruit, consumed largely because of its attractive flavour and refreshing sugar-acid balance and a very rich source of vitamin C and organic acids (Bartolomew et al., 1995). Pineapple is one of the most important fruit crops of north eastern India especially in Arunachal Pradesh of India. The crop is being grown in 7900 ha with a production of 37000 tonnes per year (Anon, 2008). Consumer demand has increased for processed products that keep more of their original characteristics. Drying is one of the most common methods of food preservation for a long time. Innovation of novel technologies and developments are constantly taking place in the existing methods of drying (Sagar and Suresh Kumar, 2009). Low cost technologies for producing locally and globally consumable commodities need to be developed to encourage fruit and vegetable processing at home scale, cottage and small scale levels. Among drying and dehydration, osmotic dehydration gained attention recently due to its potential application in the food processing industry (Rastogi et al. ,2002; Suresh Kumar et al., 2008). Osmotic dehydration is widely used for the partial removal of water from plant tissues by immersion in a hypertonic solution. osmotic solution into the tissue. Since the membrane responsible for osmotic transport is not preferably selective, other solutes present in the cells can also be leached into the osmotic solution (Giangiacomo et al., 1987; Torregianni, 1993; Rastogi and Raghavrao, 1995; Alvarez., 1995; Rastogi et al., 2002). The rate of diffusion of water from any material made up of such tissues depends upon factors such as temperature and concentration of the osmotic solution (Marcotte and Le Maguer, 1992; Roult-Wack et al., 1992), temperature, the size and geometry of material (Suresh Kumar et al. 2006) the solution to material ratio (Lerici et al., 1985), massmetry of the material and the level of agitation of the solution. Water is diffused through the membrane from the dilute to the concentrated solution until equilibrium is reached. The solute is unable to diffuse through the membrane in the reverse direction or can do so only very slowly so that the net result of this process is a transfer of water to a concentrated solution. Quality improvement is related not only to the water removal without thermal stress but also to the impregnated solutes. With the correct choice of process variables, water removal and impregnation, it is possible to enhance natural flavour and colour retention in fruit products (Marcotte and Le Maguer, 1992). Response

The diffusion of water is accompanied by the simultaneous counter diffusion of solutes from the

surface methodology (RSM) is the useful technique for complex process variables. This technique can be used in engineering process, industrial research and biological investigation with emphasis on optimizing the process system. RSM is mainly used to reduce the number of experimental runs needed to provide statistically significant information (Ravindra and Chattopadhay, 2000). In recognition of the above needs and in order to explore the possibility of preparation of osmo- dehdrated products of pineapple slices the present investigation was carried out to standardize the relation between different process variables (concentration, temperature, slice thickness and KMS concentration) on osmotic dehydration and to determine a set of optimum processing conditions suitable for better mass transfer kinetics of OD pineapple slices.

### **Materials and Methods**

Raw material and preparation: The fruits of pineapple (cv. Kew) were obtained from the Agriculture research farm, Gori, Basar, Arunachal Pradesh. Well matured, firm and ripe fruits were selected and the outer skin was peeled manually by using knife. The slices were then made to various thicknesses viz., 3, 6 and 9 mm. The core of the slices was also removed by using the pineapple core remover.

## Osmotic treatment

The weighed amount of pineapple fruits were suspended in sugar solution containing three levels of potassium meta bisulphite (KMS) viz., 0.025%, 0.05% and 0.075% and 0.1% citric acid in the wide mouthed container. Osmotic concentration of sugar was maintained viz., 50, 60 and 70 °B with the temperature constant viz., 35, 45 and 55°C. For each treatment, the temperature of the solution was maintained at pre-set value. The ratio of the fruits and osmotic solution was maintained as 1:4 in order to ensure proper soaking of the samples without any agitation. Samples were withdrawn after 6 hrs, drained quickly and wiped gently with tissue paper, and analyzed for different process variables like solid gain, water loss, water reduction and sensory attributes.

Response surface methodology (RSM) was adopted in the experimental design and analysis (Myers and Montgomery 1995). Four factors with three levels Box- Behnken factorial design was applied to run RSM with 27 experimental runs. The independent uncoded variables and their coded values are presented in Table 1. The material was weighed before and after the process to calculate the percentage of weight reduction (WR), water loss (WL) and solid gain (SG), as per the following formulae:

$$WR (\%) = \frac{Wi - Wf}{Wi} \times 100 \dots ... 1$$
$$SG (\%) = \frac{WSf - WSi}{Wi} \times 100 \dots ... 2$$
$$WL (\%) = SG + WR \dots ... 3$$

where *Wi*= initial weight of the sample (g), *Wf*= final weight of the sample (g), *WSi*= initial total solids content (%), *WSf*= final total solids content (%). Total solids content was determined by drying the sample to a constant weight.

## Sensory analysis

Descriptive sensory evaluation was carried out to determine the effect of osmo-drying on the quality attributes of osmo-dehydrated pineapple slices. A 6-member sensory panel was used to evaluate the various descriptors for colour, texture and taste of osmotic dehydrated fruits. Attributes were scored for degree of liking on 9-point hedonic scale of 1 to 9 (1=dislike extremely, 9=like extremely) and the average value was recorded. Score of 5.5 and above were considered acceptable.

## Statistical analysis

The RSM was applied to the experimental data using a commercial statistical package (Statistica Version 6.0, USA). A second order polynomial action was fitted to the data to obtain regression equation.

$$Yk = \beta k 0 + \sum_{i=1}^{4} \beta kiXi + \sum \beta kiiXii + \sum_{i=1}^{3} \sum_{j=i+1}^{4} \beta kijXiXj \dots 4$$

where, Yk; response dependant variable (Y<sub>1</sub>; WL, Y<sub>2</sub>; SG, Y<sub>3</sub>; WR and Y<sub>4</sub>; Sensory score), Xi; response independent variables and  $\beta_{ko}$ ,  $\beta_{ki}$ ,  $\beta_{kij}$  are constant regression coefficients. The statistical significance of the terms in the regression equation was examined by analysis of variance (ANOVA). Response surface and contour plots were generated and the optimization of process variables were carried out by identifying the desirability of process variables with observed and predicted values (Dhingra and Paul, 2005). The models generated were used to fit the different processing variables in optimization of mass transfer process of OD pineapple slices.

### **Results and Discussion**

The second order polynomial equation to the

Process variables	Uncoded symbol	Coded symbol		
		-1	0	1
Sugar concentration ( <sup>0</sup> Bx)	$\mathbf{X}_{1}$	50	60	70
Temperature ( <sup>0</sup> C)	$X_2$	35	45	55
Slice thickness (mm)	$X_{3}$	3	6	9
Pottasium Meta bisulphite (KMS %)	$X_4$	0.025	0.050	0.075

Table 1. Coded and uncoded values of different process variables and their values

Table 2. Box- Behnken factorial design matrix and observed values of process variables\*

Treatment	X <sub>1</sub> (Conc)	X <sub>2</sub> (Temp)	X <sub>3</sub> (Thickness)	X <sub>4</sub> (KMS)	Y <sub>1</sub> (WL)	Y <sub>2</sub> (SG)	Y <sub>3</sub> (WR)	Y <sub>4</sub> (Sensory score)
1	1	0	-1	-1	38.3	13.1	25.2	7.7
2	0	1	0	1	41.8	14.6	27.2	8.1
3	-1	-1	0	1	26.7	9.1	17.6	5.8
4	0	1	-1	-1	40.2	14.1	26.1	8
5	1	0	0	1	38.4	13.3	25.1	7.7
6	-1	-1	1	0	31.3	10.5	20.8	6.2
7	1	0	1	0	37.6	13	24.6	7.5
8	-1	-1	-1	-1	26.7	8.8	17.9	5.6
9	0	1	1	0	42.6	14.7	27.9	8.3
10	-1	0	0	0	33.8	11.2	22.6	6.9
11	-1	0	1	-1	33.7	10.3	23.4	6.4
12	1	1	-1	1	39.4	13.8	25.6	7.9
13	0	-1	1	-1	34.5	12.3	22.2	7.3
14	0	-1	0	0	35.1	11.9	23.2	7.8
15	0	-1	-1	1	30.6	9.8	20.8	6.1
16	1	1	0	0	43.2	14.9	28.3	8.6
17	-1	0	-1	1	32.7	10.9	21.8	6.6
18	1	1	1	-1	42.1	14.7	27.4	8.2
19	0	0	1	1	37.9	12.7	25.2	7.4
20	0	0	0	-1	37.4	12.9	24.5	7.5
20	-1	1	-1	0	35.6	11.9	23.7	7.2
21	0	0	-1	0	38.6	13.6	25	7.7
22	1	-1	1	1	35.1	11	24.1	6.8
23	1	-1	0	-1	34.8	11.7	23.1	7
24	1	-1	-1	0	33.7	10.6	23.1	6.6
26	-1	1	1	1	29.5	10.4	19.1	6
20	-1	1	0	-1	31.8	10.1	21.7	6.4

\*The experimental runs were performed in random order

Effect	WL	SG	WR	Sensory Score
Intercept $\beta_{k0}$	39.83	13.74	26.08	8.10
$\beta_{k1}$	3.38**	1.27**	2.11**	0.61*
$\beta_{k2}$	3.21**	1.30**	1.90**	0.53**
$\beta_{k3}$	0.47**	0.16*	0.31*	0.04
$\beta_{k4}$	-1.74*	-0.13*	-0.28*	-0.09
$\beta_{k11}$	-2.94	-1.32	-1.61	-0.63
$\beta_{k22}$	1.22*	0.39*	0.83	0.16
$\beta_{k33}$	-0.33	-0.17	-0.15	-0.23
$\beta_{k44}$	-1.74	-0.61	-1.13	-0.39
$\beta_{k12}$	0.67*	0.53*	0.17*	0.18
$\beta_{k13}$	0.35	0.20	0.14	0.11
$\beta_{k14}$	-0.15*	-0.33*	0.18*	-0.10
$\beta_{k23}$	0.95	0.46	0.48	0.24
$\beta_{k24}$	0.10	0.28	0.18	0.07
$\beta_{k34}$	-0.27	-0.01	-0.26	-0.03

Table 3. Regression co-efficient of different responses

# Table 4. ANOVA for the response variables

Source	Df	Sum of Squares			
		WL	SG	WR	Sensory Score
Model	14	495.1**	81.8**	181.4**	16.8*
Linear	4	397.4**	60.4**	147.8*	11.8*
Quadratic	4	79.9**	13.8**	27.7*	3.7
Cross product	6	16.8*	7.6	6.1*	1.3
Residual					
Lack of fit	10	31.3	2.8	19.0	0.96
Pure error	2	6.7	0.18	4.1	0.03
Total error	12	38.01	3.01	23.1	0.99
r <sup>2</sup> %		92.8	96.4	88.3	94.2

\*significant at 5% level \*\* significant at 1% level

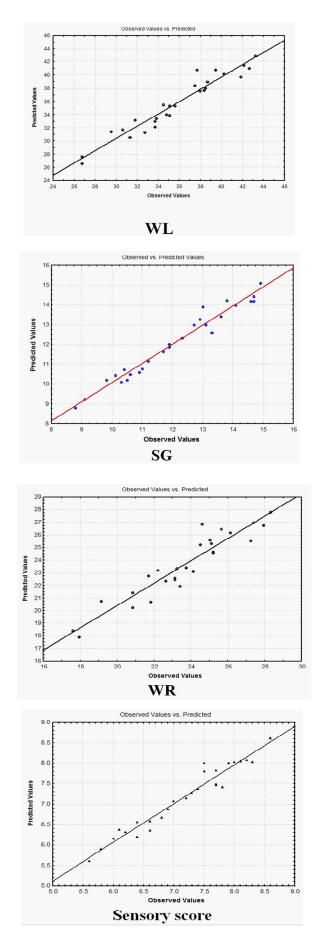


Figure 1. The predicted and observed values for different process variables

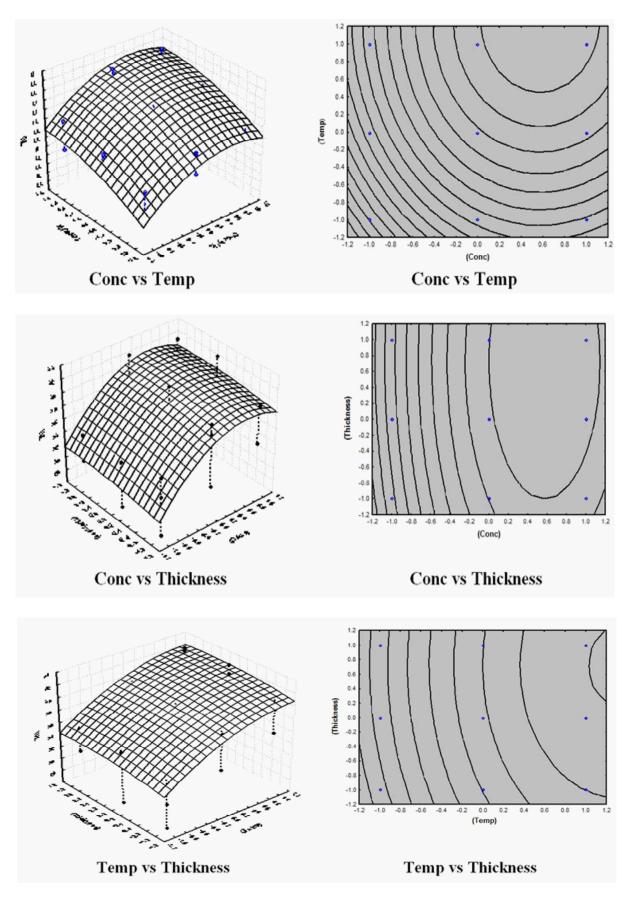


Figure 2. Response surface and contour plots showing the effect of processing variables on WL

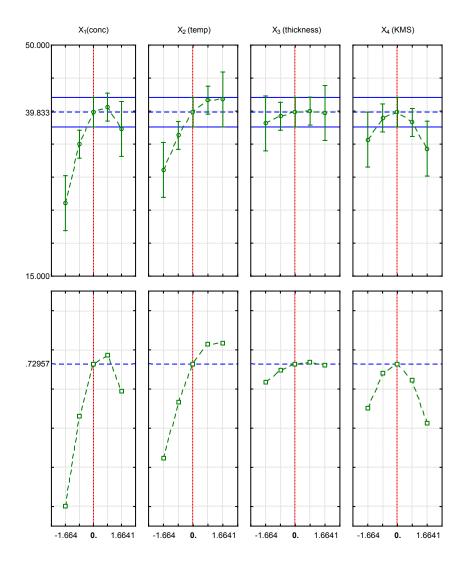


Figure 3. Profiles for water loss on predicted values and desirability

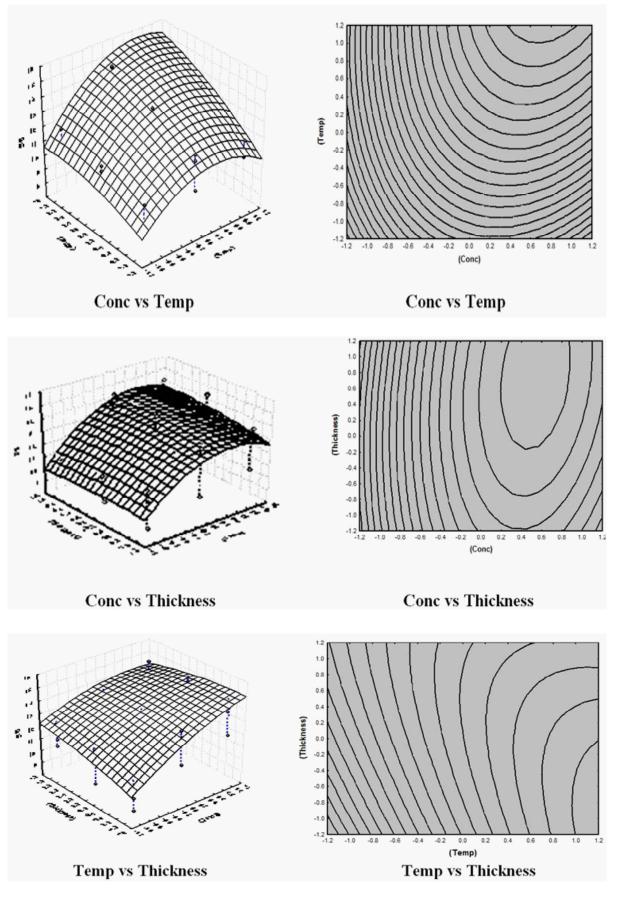


Figure 4. Response surface and contour plots showing the effect of processing variables on SG

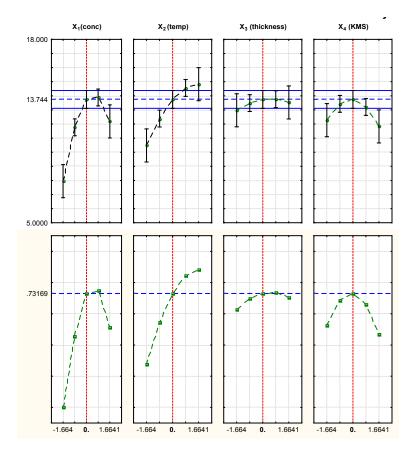
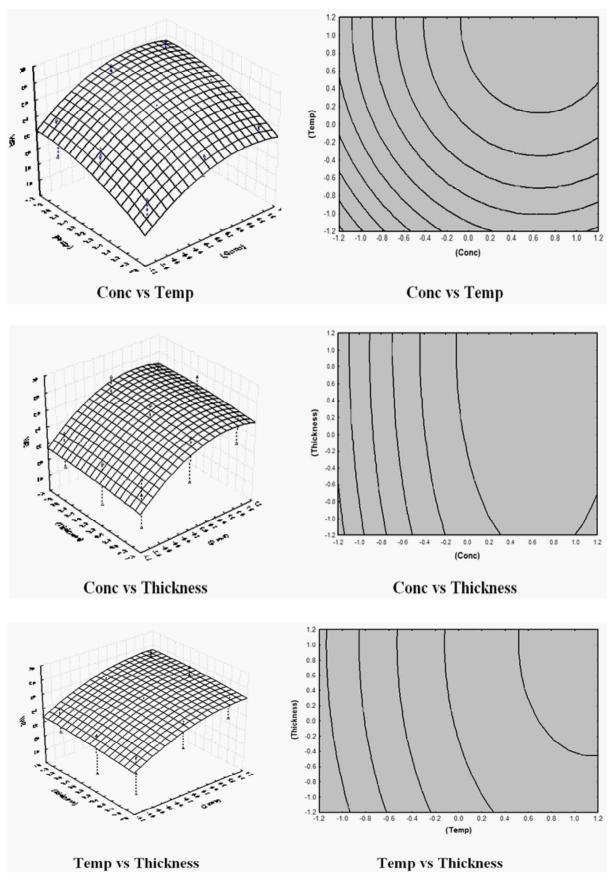
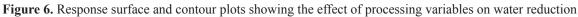


Figure 5. Profiles for solid gain on predicted values and desirability





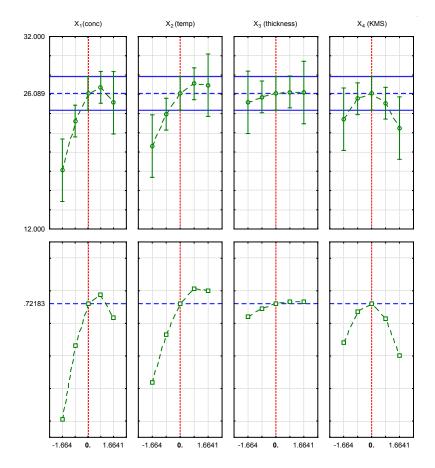


Figure 7. Profiles for water reduction on predicted values and desirability

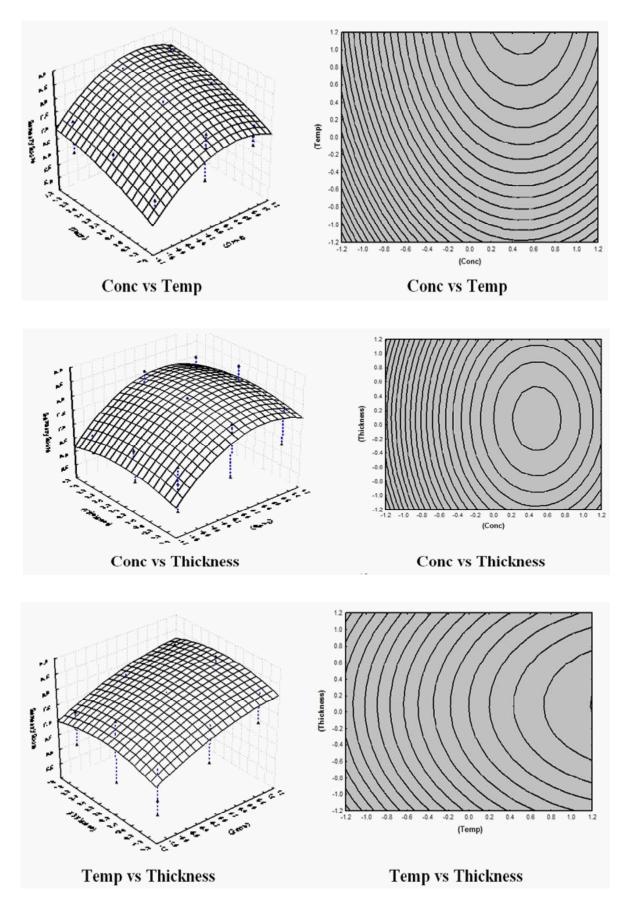


Figure 8. Response surface and contour plots showing the effect of processing variables on sensory score

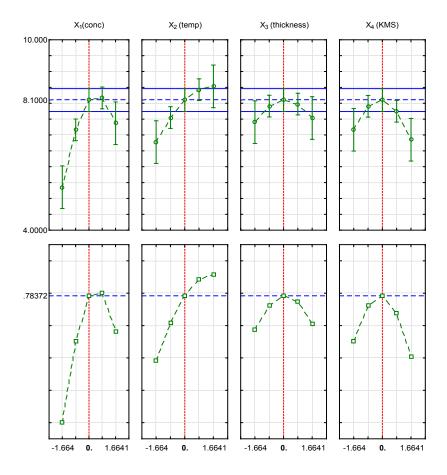


Figure 9. Profiles for sensory score on predicted values and desirability

different responses at different processing conditions was shown in Table 2. The regression coefficients obtained thereof are presented in Table 3. ANOVA was conducted to assess the significant effect of variables on the responses (Table 4). The models developed with uncoded dependent independent variables are as follows;

WL=  $39.83 + 3.38X_1 + 3.21X_2 + 0.47X_3 - 1.74X_4$ - $2.94X_1^2 - 1.22X_2^2 - 0.33X_3^2 - 1.74X_4^2 + 0.67X_1X_2$ + $0.35X_1X_3 - 0.15X_1X^4 - 0.95X_2X_3 + 0.10X_2X^4$ - $0.27X_3X_4$ SG=1374+127X + 130X + 016X - 013X - 132X^2

 $\begin{array}{l} SG{=}13.74{+}1.27X_{1}{+}1.30X_{2}{+}0.16X_{3}{-}0.13X_{4}{-}1.32X_{1}{}^{2}\\ {-}0.39X_{2}{}^{2}{-}0.17X_{3}{}^{2}{-}0.61X_{4}{}^{2}{+}0.50X_{1}X_{2}{+}0.20X_{1}X_{3}\\ {-}0.33X_{1}X_{4}{-}0.46X_{2}X_{3}{+}0.28X_{2}X_{4}{-}0.01X_{3}X_{4} \end{array}$ 

Sensory score=  $8.10 + 0.61X_1 + 0.53X_2 + 0.04X_3$ - $0.09X_4 - 0.63X_1^2 - 0.16X_2^2 - 0.23X_3^2 - 0.39X_4^2$ + $0.18X_1X_2 + 0.11X_1X_3 - 0.10X_1X_4 - 0.24X_2X_3$ + $0.07X_2X_4 - 0.03X_3X_4$ 

It was observed from Table 3 and 4 that the models developed for all the parameters other than sensory score to be very adequate, possessing no significant lack of fit. However the process variables did not have much influence on sensory attributes of pineapple slices. Nonetheless, results revealed that all the four process variables had a significant effect on overall effect on the three responses. Concentration, temperature had the most significant effect while the slice thickness and KMS concentration had the least significant effect. Computer generated response surfaces and contour plots were obtained using predictive models. Such three dimensional surfaces give accurate geometrical representation and provide useful information about the behaviour of the system within the experimental region. Predicted and observed values for water loss, solid gain, water reduction and sensory score of the fruits during osmosis process in sugar syrups are shown in Figure 1. These figures shows that the model is fit for process parameters besides showed the intrinsic properties of the fruits on structure, compactness of the tissue, the size of the contact surface between the fruit, water loss and sugar exchange.

# Changes in water loss

Table 2 indicates that the amount of WL increased with the increase in concentration of sugar and temperature. Maximum WL was 43.2% while the minimum was 26.7%. The regression eo-efficeeient was 0.92 in case of water loss. Table 3 clearly indicates that the lack of fit was not significant confirming the

significance of the model at both 1 and 5%. i.e. the process variables in the model have significant effect on the response. It was further observed that the cross product was least significant for water loss (Table 4). The effect of process variables on WL is showed in Figure 2 in RSM and contour plots. It was lucid that they had profound effect on process parameters. As the slice thickness and the KMS concentration had very least effect on process parameters their interaction effect was not plotted through RSM and contour plots. The osmosis effect increased linearly with the increase of sugar concentration up to 60-63 % (Figure 2). Further increase of sugar concentration reduces the water loss that might have lead to the sugar gain by the fruits which was not desirable (Rahman and Lamb, 1990). This is attributed to the diffusion of water from dilute medium to concentrated solution (hypertonic solution) through a semi-permeable membrane until the concentration equilibrium was reached. The driving force in this process is the water activity gradient caused due to the osmotic pressure. For solute concentrations of above 60% and below 40%, there was impregnation and crystallization of sugar and poor moisture removal respectively. This strongly suggests that for optimal osmotic dehydration, the sugar concentrations should be in the range of 55- 63%. Increase in temperature up to a certain degree increases the osmosis. Further increase in temperature affects the semi-permeability of the cell walls and reduces the rate of osmosis. This may be due to reduction in viscosity of hypertonic solution and increase in diffusion coefficient of water increased at high temperature (Shahabuddin et al., 1990; Yao and Le Maguer, 1996; Park et al., 2002).

Increase in WL was very less in 50°B sugar concentration and 35°C temperatures. This may be due to the fact that the low concentration of sugar syrup may get diluted and reach the near saturation point quickly which would not help in removing more water during the osmosis process. It was concluded that the every 10°C increase in temperature or by increasing in Brix of sugar, there was an almost equal increase in the final water loss (Roult-wack, 1994). These variables can be manipulated only up to certain range above which they affect adversely the quality. It is also showed that the dehydration rate was better in concentrated syrup due to an increased osmotic pressure in the sugar syrup at higher concentrations, which increased the driving force available for water transport (Barat et al., 2001). The contour plots (Figure 2) on interactions showed that there was linear increase in water loss with increase in concentration and temperature however same kind of interaction was not found with thickness and concentration or

temperature. However the slice thickness above 6 mm thickness did not have any influence on the water removal in the same way the KMS concentration had very little influence on water loss from the pineapple slices (Figure 3). The coded value of 0.72 was best suitable for the process variables on WL.

# Changes in solid gain

The higher concentration raise the solid gain, maximum SG of 14.9% was recorded after 6 h in 70°B at 55°C while the minimum (8.8%) solid gain was recorded with minimum level of all the four process variables (Table 2) which was corresponding to treatment 16 and 8 respectively. Further the Table 3 illustrate that the lack of fit was not significant confirming the significance of the model. In linear terms sugar concentration, temperature and slice thickness were found to be significant in model terms except for KMS concentration. The regression coefficient  $r^2$  (0.96) value indicated that the model was most significant for solute intake into the slices. Figure 4 shows that the observed values are fitting well with the predicted value of the model. Most of the treatments were performed with optimum solid gain. However it was clear from the Table 4 that the cross product value was not significant for the model. Results obtained on apple cylinders treated with osmotic solutions suggested that volumetric shrinkage is essentially due to water removal/solid gain and offer a simple way to predict such changes during industrial processing (Sereno et al., 2001).

It was clear from the desirability profiles on solid gain (Figure 5) that with the increase in sugar concentration from 50 to 60°Bx the solid gain also increased. But above (0.73 coded value) certain coded value the solid gain gets decreased. The minimum solid gain was occurred with minimum concentration and temperature (Figure 4). This is attributed to longer time by osmosis to approach equilibrium between cellular fluids and osmotic solution along with volume and compactness of fruits solution (Barat et al., 2001). Tissue damage due to too high temperature causes a dramatic decrease in dehydration efficiency through increased solute and decreased moisture diffusivities.

## Changes in weight reduction

Weight reduction was increased with an increase in concentration and temperature of sugar syrup (Figure 6). However the KMS concentration had very little effect on water reduction as in the case for water loss and solid gain. The reason was that the viscosity of hypertonic solution was lowered and the diffusion coefficient of water increased at high temperature. The difference in WR attributed to osmosis, might be a cause of variation in WR rate among different concentration of sugars. Table 4 on ANOVA revealed that the cross product of process variables were least significant while the linear and quadratic value were significant. In addition the non significant value of lack of fit put the model as the perfect for process optimization for pineapple slices. The regression coefficient value was 0.91 which was highly significant for the model. Figure 1 on observed and predicted value further assures that the model was the fit for water reduction. Table 3 indicates that concentration and temperature influenced the water reduction more than slice thickness and KMS concentration. The negative value indicates that the KMS concentration had no effect on mass transfer kinetics. However it was very much essential for extending the storability of OD pineapple slices.

The Figure 6 on RSM and contour plot illustrates that WR got increased with increase in concentration and temperature with constant value of slice thickness (6mm) and KMS concentration (0.05%). Concentration and temperature versus thickness showed that the slice thickness did not have much influence on water reduction. Figure 7 on desirability clearly indicates that the concentration above 0.72 of coded value had the least effect on water reduction. However the temperature increases the WR as it level get increased. The KMS concentration above optimum (0.05%) did not have any effect on water reduction (Panagiatou et al., 1999).

## Changes in sensory score

Sensory score appear to be useful to select the best osmotic concentration and temperature as chemical constituents were very indistinctness. However it was evident form Table 3 and 4 that sensory attributes are least affected by different process variables. However treatment 16 scored highest value on sensory attributes (Table 2). But the high temperature and concentration negatively affect the colour of the slices. Best colour was obtained with 60°B with 55°C temperature (Figure 8). The contour plot clearly shows that none of the process variables distinctively affect the sensory attributes of pineapple slices. However the optimum process variables are required to get better produces with grater overall acceptability. This is due to prevention of enzymatic and oxidative browning as the fruit pieces were surrounded by sugar thus making it possible to retain good colour. Similar results were observed in dehydrated ripe mango slices (Suresh Kumar and Sagar, 2009). However, increase in high concentration and very high temperature hinders the solid gain and there by render poor colour score. Better flavour was also obtained with the same concentration and temperature. Very low score on texture was observed with very low concentration of sugar. Over all acceptability was also higher with treatment 16 than other combinations. Profiles on sensory attributes desirability (Figure 9) clearly indicate that the above 0.78 of coded value had the least effect on mass quality attributes. During the osmotic step, furanones, pyranones and to a lesser extent, esters remain in the fruit tissue, while alcohols and carbonyl compounds moves from the fruit to syrup, probably due to the solubility of these compounds in water (Rastogi et al., 2002). Furthermore, the concentration of strawberry slices, through osmotic dehydration, improves the volatile retention during air drying in such a way that previously osmo-dehydrated slices could be dried up to higher drying levels when compared to non treated fruit.

It was clear from the Table 2 and desirability figures that treatment 16 followed by treatment 18 performed well among the different response process combinations. The desirability figures showed that the coded value of 0.71 to 0.78 was best for WL, SG and WR. Therefore in order to optimize the process condition, the sugar concentration could be optimized with the range of 58-63°Bx, the temperature with 55°C, slice thickness equal to 6 mm and the KMS concentration to 0.05 -0.065% to obtain higher values for different process variables.

# Conclusion

A large number of food and biomaterials are dehydrated in a variety of units with diverse processing conditions. Energy consumption and quality of dried products are critical parameters in the selection of drying process. An optimum drying system for the preparation of quality dehydrated products is cost effective as it shortens the drying time and cause minimum damage to the product. Osmotic dehydration due to its energy and quality related advantages, is gaining popularity as a complimentary processing step, in the chain of integrated food processing. However, Osmotic treatments have been applied frequently as low cost processing method neglecting process optimization, but the current interest in this technique and the development of industrial applications on a large scale demand controlled processes. It is necessary to develop predictive models through understanding of mechanism of mass transport.

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