RESPONSE SURFACE ANALYSIS AND OPTIMIZATION OF OSMOTIC DEHYDRATION OF *Musa acuminata* SLICES

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ABSTRACT

The mass transfer characters were quantitatively investigated during osmotic dehydration of *Musa acuminata* slices using response surface methodology with sugar concentration of 30-50 ºBrix, temperature of 30-50°C, osmotic dehydration time of 30-180 minutes and sample to solution ratio of 1:5-1:15 g/ml as the independent process variables. Quadratic regression equations were obtained to describe the effects of independent process variables on the weight reduction (WR), solid gain (SG) and water loss (WL). From the results, it was found that, all the independent variables had significant effect on mass transfer characteristics during osmotic dehydration of banana slices. The optimal condition for osmotic dehydration of *M.acuminata* slices was found to be: osmotic solution concentration of 43ºBrix, sample to solution ratio of 1:10 g/ml, osmotic solution temperature of 50°C and osmotic dehydration time of 164 minutes respectively.

Keywords: *M. acuminata* slice, Osmotic dehydration, Mass transfer, Optimization

INTRODUCTION

The fruits and vegetables are inevitable part of the diet of humankind as they are considered to be the prime source of carbohydrates, indigestible minerals, essential amounts of vitamins (especially vitamins A and C) and calories which provide the nutritional values for body building and wellness of the human beings (**Salunke *et al.*, 1991**). As most of the fruits contain moisture content more than 75 per cent, they are highly prone to the attack of molds and yeasts thereby causing spoilage to this perishable commodity up to 30 per cent of total production (**Janisiewicz *et al.*, 1999**). The application of appropriate methods of processing and preservation could definitely reduce the spoilage of fruits and vegetables. Among the various processing and preservation techniques employed to enhance the shelf life of fruits and vegetables, osmotic dehydration (OD) is one of the simple, low cost and energy efficient method based on the removal of natural water content from fruits and vegetables by immersing them in n hypertonic osmotic solutions prepared with sugars, salts, alcohols and starch solutions (**Erle & Schubert, 2001; Azuara & Beristain, 2002**). The OD process kinetics is influenced by the nature of the osmotic solute and it has a great impact on the nutritional and sensorial properties of the final product (**Forni, 1997; Ferrando, 2001**).
Musa acuminata is one of the largely consumed fruit around the world and India is the major contributor (9%) of the total production. The fruit has high nutritional value, rich in starch and sugars. Apart from these, it also contains good amount of health benefitting anti-oxidants, vitamins A, C and minerals such as phosphorus, potassium, sodium, calcium and magnesium (**Borges, 1997**). The addition of *M.acuminata* in our daily diet provides an array of health benefits such as lowering of blood pressure, reduce the development of childhood leukemia, combat the formation of free radicals known to cause cancer, supporting heart health, curing diabetes and so on. Owing to the wide range of beneficial properties of *M.acuminata*, it is necessary to preserve this fruit from the losses due to perishability and to add value to it in order to make it shelf stable. But, very few research works have been performed by researchers in the osmotic dehydration of *M.acuminata* using sugar solution as an osmotic agent. Hence, in this present work, response surface methodology (RSM) was used to determine and optimize the effect of process variables (Osmotic sugar concentration, osmotic solution temperature, immersion time and sample to solution ratio) on the mass transfer characters (weight reduction, solid gain and water loss) during osmotic dehydration of *M.acuminata* slices.

MATERIALS AND METHODS

Materials

Fresh *M.acuminata* (local variety) of uniform size and ripeness were obtained from the local market in Coimbatore, TamilNadu, India. The samples were sorted out visually for homogeneous maturity without having any physical damages, washed in running tap water and surface of the samples were wiped with tissue paper in order to eliminate the surplus water adhering over the surface. After that, the peel was removed manually and the pulp was cut into slices of uniform thickness (2.5± 0.2cm diameter and 5mm thickness) and stored at 5°C. The initial moisture content of slices was determined by using hot air oven method (**AOAC, 2000**) and it was found to be 76.4% (wb).

Preparation of osmotic solution

Food grade sugar (99.5% purity) was purchased from a super market located at Coimbatore, TamilNadu, India. Osmotic solutions of sugar at different concentrations (30, 40 and 50ºBrix) were prepared by dissolving the required quantity of sugar in distilled water and the prepared solution was kept at refrigerated condition prior to experiments. The sugar concentration was measured with a portable refractometer.

Osmotic Dehydration Experiment

Osmotic dehydration experiments were carried out as per the design of experiments with four different independent variables (sugar concentration, osmotic solution temperature, immersion time and sample to solution ratio). Known weight of *M.acuminata* slices were immersed in the Erlenmeyer flasks containing required quantity of osmotic solution and kept at temperature controlled chamber in order to maintain the different temperatures (30-50°C). The Erlenmeyer flasks were covered with a plastic cover during the experiments with the purpose of eliminating the evaporation of osmotic solution. The experiments were carried out at different osmotic solution concentrations (30-50ºBrix), different temperatures (30-50°C), sample to solution ratio (1:5-1:15 g/ml) and immersion time (30-180 min). At specified time interval, the *M.acuminata* slices were taken out from the hypertonic solution and weighed after removing the solution adhered on the surface of the slices using filter paper (Whatman No.1). Triplicate experiments were carried in the all the experimental condition, mean value was calculated and used to determine the weight reduction (WR), solid gain (SG) and water loss (WL).

Determination of Osmotic Dehydration Characteristics

Osmotic dehydration is a two way process where both the water loss and solid gain are acquired simultaneously from the sample as well as from the solution. The reduction in weight is attributed by the loss of water from the slices and increase in the weight of the slices due to solute gain from the solution. The solid gain is the net uptake of solids during osmotic dehydration by the slices on initial weight basis. The water loss (WL) is defined as the net weight loss of the fruit on initial weight basis. The assessment of mass transfer properties (weight reduction (WR), solid gain (SG) and water loss (WL)) between the osmotic solution and samples during this process were estimated by the formula described elsewhere (**Tiroutchelvame et al., 2015**).

Experimental design and statistical analysis

Response surface methodology (RSM) is one of the experiential statistical modeling methods employed to obtain experimental data from the appropriately planned experiments to resolve the multivariate equations concurrently. A Box Behnken Design (BBD) with three level four factors was used to plan the experiments and it is exhibited in Table 1. The process parameters opted for the optimization were, osmotic solution concentration(X_1), sample to solution ratio (X_2), osmotic temperature (X_3) and osmotic dehydration time (X_4). The BBD design comprises of 29 experiments and with three central points. Each process variable was regulated or coded at three levels (+1, 0 and -1) and coding of the variables was done according to formula described by **PrakashMaran et al., (2012)**. The experimental data was fitted to a second order polynomial equation

in order to recognize the appropriate model terms. The general form of the quadratic model can be described elsewhere (**PrakashMaran et al., 2013**). Multiple regression analysis method was employed on the experimental data in order to evaluate the significance of regression coefficients of process variables. Analysis of variance (ANOVA) tables were generated from the experimental data using Design Expert Statistical Software package 8.0.7.1 (Stat Ease Inc., Minneapolis, USA).

Microstructure analysis

The fresh and osmotically treated (optimal condition) *M.acuminata* slices were examined using scanning electron microscopy (SEM) in order to study the influence of osmotic dehydration process on the microstructure of the *M.acuminata* samples. The microstructure of the samples was viewed under a Jeol scanning electron microscope (JSM-6390) and the images were recorded.

RESULTS AND DISCUSSION

Model fitting and statistical analysis

A total number of 29 experiments were performed with different combinations of process variables in order to investigate and optimize the influence of independent variables (osmotic solution concentration, sample to solution ratio, osmotic temperature and osmotic time) on the responses (WR, SG and WL) and the results are shown in Table 1.

Table 1 Experimental design matrix with observed results

Run No	Concentration (ºBrix)	SS ratio (g/ml)	Temperature (°C)	Time (min)	WR (%)	SG (%)	WL (%)
1	40	1:15	40	180	21.92	10.71	26.57
2	40	1:5	50	105	26.05	14.86	34.85
3	50	1:10	50	105	35.11	20.91	49.96
4	40	1:5	40	180	25.32	14.27	33.53
5	50	1:10	40	180	30.16	20.71	44.81
6	40	1:5	40	30	23.56	13.63	31.13
7	40	1:10	30	30	24.85	14.11	32.9
8	30	1:10	30	105	16.64	6.29	16.87
9	40	1:10	40	105	36.29	26.1	53.33
10	30	1:10	40	30	22.56	11.36	27.86
11	40	1:10	50	30	28.45	18.26	40.65
12	40	1:10	30	180	21.86	10.71	26.51
13	40	1:10	40	105	37.29	26.14	57.37
14	50	1:5	40	105	30.51	19.35	43.8
15	40	1:10	40	105	37.29	19.11	47.15
16	40	1:10	40	105	37.29	26.1	53.41
17	50	1:15	40	105	24.26	13.06	31.26
18	50	1:10	40	30	30.94	19.75	44.63
19	40	1:10	50	180	31.68	0.53	26.15
20	30	1:5	40	105	20.91	9.75	24.6
21	40	1:5	30	105	24.95	13.76	32.65
22	30	1:15	40	105	20.33	9.18	23.45
23	30	1:10	40	180	22.55	11.35	27.84
24	50	1:10	30	105	29.21	18.06	41.21
25	30	1:10	50	105	29.44	18.24	41.62
26	40	1:15	50	105	29.44	18.25	41.63
27	40	1:15	30	105	15.13	3.94	13.01
28	40	1:10	40	105	37.29	26.1	53.41
29	40	1:15	40	30	26.11	14.92	34.97

WR = Weight Reduction; SG = Solid Gain; WL= Weight Loss.

By applying multiple regression analysis on the experimental data, second-order polynomial equations were developed for the responses which can express the relationship between process variables and the responses. The final equations obtained in terms of coded factors are given below.

$$WR = 37.09 + 3.98X_1 - 1.18X_2 + 3.96X_3 - 0.25X_4 - 1.42X_1X_2 - 1.73X_1X_3 + 0.19X_1X_4$$
$$+ 3.30X_2X_3 - 1.49X_2X_4 + 1.56X_3X_4 - 4.96X_1^2 - 7.98X_2^2 - 4.94X_3^2 - 5.30X_4^2 \quad (1)$$

$$SG = 24.71 + 3.81X_1 - 1.30X_2 + 2.02X_3 - 1.98X_4 - 1.43X_1X_2 - 2.27X_1X_3 + 0.24X_1X_4$$
$$+ 3.30X_2X_3 - 1.21X_2X_4 - 3.58X_3X_4 - 3.69X_1^2 - 6.48X_2^2 - 6.20X_3^2 - 5.90X_4^2 \quad (2)$$

$$WL = 52.93 + 7.79X_1 - 2.47X_2 + 5.98X_3 - 2.23X_4 - 2.85X_1X_2 - 4.0X_1X_3 + 0.050X_1X_4$$
$$+ 6.60X_2X_3 - 2.70X_2X_4 - 2.03X_3X_4 - 7.25X_1^2 - 13.05X_2^2 - 9.73X_3^2 - 7.79X_4^2 \quad (3)$$

Pareto analysis of variance (ANOVA) was used to analyze the experimental data and the results are listed in Table 2. The higher model F-value (68.81for WR, 4.35 for SG, 11.86 for WL) and the associated lower p-values ($p < 0.0001$) demonstrated the significance of developed models and also indicated that most of the variation in the responses could be explained through the regression

equations. The high value of R^2 (0.9857 for WR, 0.9131 for SG, 0.9223 for WL) and adj-R^2 (0.9714 for WR, 0.9262 for SG, 0.9445 for WL) clearly demonstrated that the form of the model chosen to represent the actual relationship between the response and independent variables is well correlated and accurate. High values of coefficient of variance (3.86 for WR, 25.76 for SG, 12.38 for WL) exhibited

the high degree of precision and good reliability of the conducted experiments. In this study, the adequate precision (signal to noise ratio) was found to be > 29 for

all the responses, which indicated the best fitness of the developed models.

Table 2 Analysis of variance (ANOVA) for the developed second order polynomial models

Source	DF	Weight reduction(%)			Solid gain(%)			Water loss(%)		
		CE	SS	*p*-value	CE	SS	*p*-value	CE	SS	*p*-value
Model	14	37.09	1085.55	< 0.0001	24.71	971.11	0.0047	52.93	3382.64	< 0.0001
X_1	1	3.98	190.08	< 0.0001	3.81	173.81	0.0052	7.79	727.43	< 0.0001
X_2	1	-1.18	16.59	0.0018	-1.30	20.18	0.2796	-2.47	73.36	0.0785
X_3	1	3.96	188.26	< 0.0001	2.02	48.72	0.1023	5.98	428.53	0.0004
X_4	1	-0.25	0.74	0.4313	-1.98	47.01	0.1080	-2.23	59.54	0.1094
X_{12}	1	-1.42	8.04	0.0183	-1.43	8.18	0.4856	-2.85	32.43	0.2276
X_{13}	1	-1.73	11.90	0.0058	-2.28	20.70	0.2736	-4.00	64.00	0.0980
X_{14}	1	-0.19	0.15	0.7223	0.24	0.24	0.9051	0.05	0.01	0.9826
X_{23}	1	3.30	43.63	< 0.0001	3.30	43.63	0.1203	6.61	174.50	0.0110
X_{24}	1	-1.49	8.85	0.0141	-1.21	5.88	0.5534	-2.70	29.16	0.2514
X_{34}	1	1.56	9.67	0.0110	-3.58	51.34	0.0944	-2.03	16.44	0.3841
X_1^2	1	-4.97	159.90	< 0.0001	-3.69	88.10	0.0339	-7.25	340.70	0.0011
X_2^2	1	-7.98	413.19	< 0.0001	-6.48	272.09	0.0010	-13.05	1105.50	< 0.0001
X_3^2	1	-4.94	158.37	< 0.0001	-6.20	249.07	0.0014	-9.73	614.72	< 0.0001
X_4^2	1	-5.30	182.03	< 0.0001	-5.90	225.64	0.0021	-9.79	622.00	< 0.0001

DF= Degree of Freedom; CE=Coefficient estimate; SS=Sum of Squares.

Effect of process variables

During the early stages of osmotic dehydration of *M.acuminata* slices, the increase in osmotic solution concentration from 30 to 46ºBrix enhanced the mass transfer properties (WR, SG and WL) (Fig.1). This is due to the fact that smaller molecular weights of the solute (sugar) vigorously infiltrate into the plant tissue and enhances the outflow of water from the inner tissue rapidly to the osmotic solution and increased the WR and WL **(Spiazzi & Mascheroni, 1997)**. Furthermore, the higher concentration of the solution augments the osmotic pressure gradient and the succeeding loss of functionality of plasmatic membrane of the cell which allows the solute to penetrate into the plant tissues and boosted the SG during the early stages of osmotic dehydration (Fig.1). However, the mass transfer properties of *M.acuminata* slices decreased beyond 46ºBrix level of sugar concentration (Fig.1) which described that the diffusion strength of osmotic solution (sugar solution) in to the plant material and the comparative moisture content on the outside of product were independent on the concentration of the solution and hence reduced the moisture transport phenomenon of the *M.acuminata* slices **(Lenart & Flink, 1984)**.
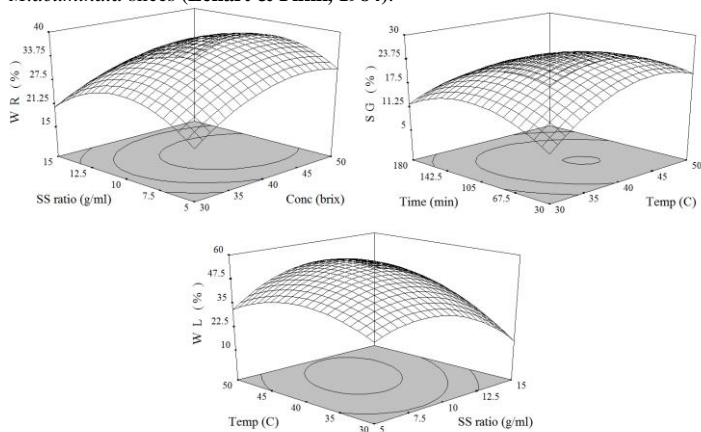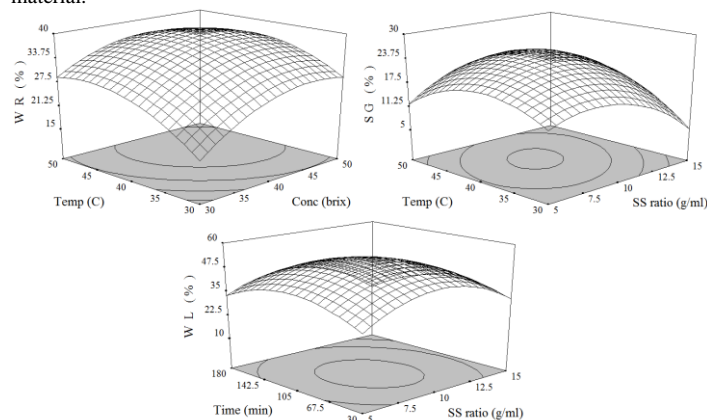

Figure1 Effect of process variables on the mass transfer properties of *M.acuminata* slices

The significance of sample to solution ratio (SS ratio) on the mass transfer properties of *M.acuminata* slices was appraised and the outcomes were exhibited in Fig.1. It was observed that the mass transfer properties on the *M.acuminata* slices during osmotic dehydration showed an increasing trend for the sample to solution ratio from 1:5-1:11 g/ml. This increase in trend may be substantiated by the fact that the higher SS ratio accelerated the driving force of the osmotic solution, enhanced the diffusion of solids from the osmotic solution to plant materials which improved the discharge of water from the plant materials to the surrounding medium **(Uddin *et al*., 2004)** and improved the mass transfer properties (Fig.1) of the *M.acuminata* slices **(Tortoe, 2010)**. However, a decline in the trend was noted beyond the SS ratio of 1:11 g/ml which stated that the osmotic solution became diluted due to the higher amount of water released from the plant tissues during osmotic dehydration which negatively affected the mass

transfer properties and confirmed the results obtained by **Bongirwar & Srinivasan (1977)**.

One of the vital parameters which control the osmotic dehydration properties of the *M.acuminata* slices is the temperature of the osmotic solution. The effect of osmotic solution temperature on the mass transfer properties of the *M.acuminata* slices and the consequences were depicted in Fig.2. Based on the results of the experiments conducted, it was observed that there was an enhancement in water loss from the plant materials and simultaneous uptake of solids (Fig.2) **(Ispir & Toğrul, 2009 ; Khan, 2012)** through swelling and plasticizing of cell membranes **(Contreras & Smyral, 1981 ; Lazerides & Mavroudis, 1997)** while the temperature of the osmotic solution was increased from 30 to 47°C. However, beyond osmotic solution temperature of 47°C, the mass transfer properties of *M.acuminata* slices (Fig.2) decreased which may be due to the predominance of thermal denaturation and affected the cell membrane permeability, decreased the viscosity of osmotic solution and enhanced the altercation of solids into the plant material.

Figure 2 Effect of process variables on the mass transfer properties of *M.acuminata* slices

Osmotic dehydration experiments were performed at different time levels (30-180 min) in order to investigate the effect of immersion time on the mass transfer properties of *M.acuminata* slices during osmotic dehydration and the consequences were shown in Fig.2. The results revealed that, increase in the immersion time of the sample in the osmotic solution upto 140 minutes caused an improvement in the mass transfer parameters of the samples (Fig.2). This enlightened the fact that the transport phenomena become very rapid during the early stages of dehydration and aided to the progression of mass transfer of solutes due to the feasible membrane enlargement or plasticizing, which forced to enhance the cell membrane permeability to the sugar molecules to infuse into the plant material **(Lazerides & Mavroudis, 1997)** and advanced the loss of water from the plant tissues **(Lazarides *et al*., 1995 ; Ertekin & Çakaloz, 1996 ; Shi & Maguer, 2002)**. It was also visualized that after an osmotic dehydration time of 140 minutes, equilibrium level was attained between the sample and the osmotic solution which brought a negative trend in mass transfer rate and decreased the mass transfer properties of the slices **(Ertekin & Çakaloz, 1995)**.

Optimization and validation of dehydration parameters

In this study, desired function methodology was used to identify the optimal osmotic dehydration condition. By using this method and from the results of the osmotic dehydration experiments, the optimized conditions obtained were : osmotic solution concentration of 42.36 ºBrix, sample to solution ratio of 1:10.17 g/ml, osmotic solution temperature of 50°C and osmotic dehydration time of 164.21 minutes and the corresponding values of mass transfer properties were : WR 34.08%, SG 12.68% and WL 40.25%. However, considering the operability in real manufacture, the most favorable condition can be customized as follows: osmotic solution concentration of 43%, sample to solution ratio of 1:10 g/ml, osmotic solution temperature of 50°C and osmotic dehydration time of 164 minutes. To compare the predicted results with experimental value, triplicate experiments were carried out at the modified optimum condition and the experimental yield (WR of 34.26 ± 0.48%, SG of 12.63± 0.34% and WL of 40.419± 0.19%) matched well with predicted values.

Microstructural analysis at optimal condition

Micrographs of osmotically treated and untreated samples are depicted in Fig. 3. The Fig. 3a illustrates that, cytoplasmic membrane and cell walls appeared as the bright regions. The appearance of cells in a tattered and uneven in shape exhibits the existence of several blank regions (Fig.3a). The Fig.3b confirms that, the cells materialized with decrease in size and unclear. It could be possibly due to the solubilization of carbohydrates, loss of water and the salt concentration on the exterior of the plant materials during the process (**Raoult-Wack *et al*., 2007; Torreggiani & Bertolo, 2001; Garcia *et al*., 2010**). In addition, loss of water might have stimulated the plasmolysis of cells and the stability to the tissues by solute gain (**Nunes *et al*., 2008).**
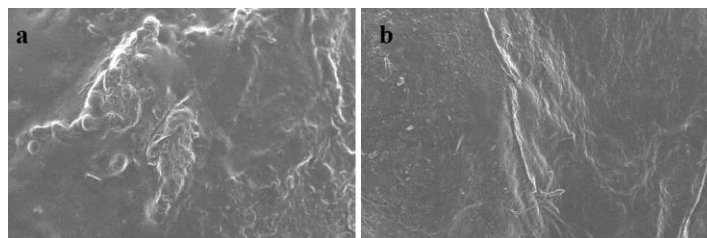
Figure 3 SEM micrograph of raw and osmotically dehydrated *M.acuminata* slices

CONCLUSION

Four factors, three levels Box-Behnken experimental design was effectively utilized in this study to evaluate and identify the optimal osmotic dehydration condition in order to prepare osmotically dehydrated *M.acuminata* slices using sugar solution as an osmotic agent. Three second order polynomial models were framed for the responses (WR, SG and WL) from the observed data. The results exhibited that, all the independent variables have considerable influence on the osmotic dehydration process of *M.acuminata* slices. The optimum condition was identified by desired function methodology and validated the optimal condition experimentally.

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