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Experimental Study on Kinetics, Modeling and Optimisation of Osmotic Dehydration of Mango (*Mangifera Indica L*)

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--Abstract--

Osmotic dehydration is a widely accepted pre-treatment method with diverse huddle effects in fruits and vegetable preservation but has not enjoyed amiable application in food processing industry. The objective of this work is to study the mass transfer mechanism during osmotic dehydration, the process modelling and osmotic dehydration optimisation. Factors affecting mass transfer during osmotic dehydration especially concentrations of osmotic agent, processing temperatures, and time were investigated using mango fruit (Mangifera indica L) as a case study. The combined effects of temperature, sucrose concentration, and process time were modelled and while water loss and solute gain as response variables were optimised using Response Surface Methodology (RSM) with Central Composite Rotable Design (CCRD). Optimised conditions of 53.5^0Bx sucrose concentration 30.0^0C temperatrure and immersion time of 160.0min removed about 42.6 % water content with minimum solute gain of 6.3% of the sample solid content. At the predicted optimum points, the observed water loss and solute gain were found to be 41.87 and 10.65% of the initial sample content respectively. These results are impressive for future design of versatile equipment for osmotic dehydration of fruits and vegetables for the food industries.

Keywords: *optimization, osmotic dehydration, transfer mechanism, water loss and solute gain.*

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I. INTRODUCTION

Dehydration is an important process to preserve raw food materials and products in the food industry. The basic objective of dehydration in food processing is the removal of water from the raw food materials to extend the shelf life and reduce the water activity in food products Nowadays, fresh fruits and vegetables have been increasing in popularity for consumption compared to canned fruits [1]. Researchers have looked for new ways to improve the quality of preserved food products; one of these methods is osmotic dehydration. It has been widely used as a pre-treatment step in food drying process since it can reduce the overall energy requirement for further drying process [1,3].

Osmotic dehydration removes water from the fruit up to a certain level, and the final water content is too high for adequate food preservation. Therefore, these foods are not stable and usually complementary drying process is required [4,5]. Osmotic dehydration involved the immersion of food material in highly concentrated osmotic solution which in turn imposed an osmotic pressure gradient to withdraw excess water from the material thereby reducing its water activity. During the process, excess solute uptake changed the organoleptic and nutritional characteristics of the food materials and there were loss of vitamin and mineral salt from food products, [6,7]. The current increase of interest in osmotic treatments arises primarily from the need for quality improvement and economic gains. Hence, minimal solute gain and high water loss are desired objective functions in the osmotic dehydration of mango.

Osmotic dehydration was characterised by the solute gain and water loss for both quantitative modelling and knowledge of the kinetics of mass transfer while the quality of osmotic dehydration were evaluated as the ratio of water loss to solute gain [8,9]. Mango (*Mangifera indica L.*) is one of the most important commercial crops worldwide in terms of production, marketing and consumption. Unripe mangoes are rich in vitamin C, the ripe fruits are rich in provitamin A and contain moderate levels of vitamin C. All mango varieties represent a potential source of natural antioxidants [10,11]. A vast diversity of products may be prepared from fresh mango. However, mangoes are extremely perishable like other farm produce especially fruits and vegetables.

The pre-treatment with osmotic dehydration to retain its nutritional qualities cannot be over emphasised. Hence, fast and efficient methods are required to conserve the quality of the fruit from harvest to consumption [12,13]. In conclusion of their investigation [13] reported that using proper dehydration techniques, dried mango with physicochemical and sensory qualities similar to those of fresh fruit could be obtained. Mango products treated either as slices or puree by osmotic dehydration and hurdle technology are reported [14]. For these cases, the water loss and solid gain were plotted against time of immersion. Quantitative modelling and knowledge of the kinetics of mass transfer (water loss and solute uptake) are necessary in studying osmotic dehydration process. Quality improvement is related not only to the water removal with minimal thermal stress but also to the impregnated solute and the modification of the structure [15 In order to develop an efficient technology for preservation of fruits and vegetables by osmotic dehydration, it is necessary to understand the mass transfer kinetics which characterises osmotic dehydration process until equilibrium is attained, when net rate of mass transport is zero. The objective of this work was to examine the kinetics of water loss and solute gain as a function of sucrose concentrations, temperatures and time of immersion during osmotic processing of mango mesocarp, model and optimise these functions necessary for qualitative information on osmotic dehydration process design and control.

II. MATERIALS AND METHODS

2.1 Materials

Fresh matured mango fruits, cultivar *(Mangifera indica L.)* obtained directly from a local farm in Epe town suburb of Lagos, Lagos State, Nigeria was used. Fruits in early stage of ripeness in terms of colour (greenish yellow), with similar size (average weight of about 650 g and average diameter of 2.8 cm) and without physical damage were selected. Mango samples were prepared by peeling off epicarp and slicing the mesocarp into slabs. Specimens of uniform cubes of 15.0±0.5 mm^3 dimensions were prepared using a metallic dicer manually to maintain approximately uniform samples, [16]. The average moisture content of sample was determined in a vacuum oven (Genlab MINO/50) at 70 ^{0}C to be 82.37±0.5 % on wet basis. The initial sugar content in the fresh mango fruit was determined to be 15.91±0.5 ^{0}Brix. [17].

2.2 Methods

The osmotic solutions were prepared using commercial sucrose (granulated sugar, 98% minimum purity) and distilled water at room temperature of 27±1.5 ^{o}C and in the range of 40 to 65 (^{o}Bx) confirmed in RFM-100 refractometer. The resulting sucrose syrup was kept overnight to equilibrate before use [18]. The sucrose concentrations of (w/w%) were prepared with distilled water. Weighed and labelled samples of mango cubes diced into 15.0±0.5 mm^3 dimensions were osmo-dehydrated in sucrose concentrations between 40 (^{o}Bx) to 65 (^{o}Bx) and 30^{0}C to 50 ^{0}C temperature for up to 180 minutes. A fruit to liquor ratio of 1:5 (w/w) was maintained through each set of experiment conducted in a steam bath stirred continuously to ensure effective agitation. The experimental design was evaluated using coded levels -1 to +1 according to levels number with Design-Expert software version 6.08 of Stat-Ease Inc. Minneapolis, MN-2002, as specified in Table 1.

TABLE 1: Experimental Design

Factor	Name	Units	Type	Low Actual	High Actual	Low Coded	High Coded
A	Conc	^{0} Bx	Numeric	40.00	65.00	-1.000	1.000
B	Temp	^{0}C	Numeric	30.00	50.0	-1.000	1.000
C	Time	Mins	Numeric	30.00	180.00	-1.000	1.000

2.3 Evaluation of OD in treated samples

The characteristic parameters for osmotic treatment mainly; water loss (WL) solute gain (SG), and moisture content on wet basis were determined by gravimetric measurement and calculations according to equations (1) - (3) and expressed in percentage (%) of initial compositions [19,20,21]

$$WL = \frac{M_{W\,\text{initial}} + (M_{\text{final}} - M_{S\ \text{final}})}{M_{inital}} \times 100 \quad (1)$$

$$SG = \{\frac{(M_{S\,\text{final}} - M_{S\,\text{initial}})}{\text{M}_{\text{initial}}} \times 100\} \quad (2)$$

$$MC = \frac{\%\,water \text{ removed by dehydration}}{\text{Total \%water content in sample}} \quad (3)$$

where WL = water loss %; SG = solid gain % ;MC=Moisture content in sample (wet basis)
M_W initial = initial water content before osmotic dehydration (g);
M_{final} g = final mass of sample after dehydration at set time (g);
$M_{s\ initial}$ = initial solid content in fresh sample (g);
$M_{s\ final}$ = final solid content after OD at specified time (g);
X and X_0 : represent the moisture contents at initial time and at times 't' respectively.

2.4 Modelling and optimization

The gravimetric evaluation of experimental data for water loss (WL) and solids gain (SG) for sample were subjected to analysis of variance (ANOVA) and F test in order to be fitted to a polynomial of the type in Equation 4:

$$Y = a_o + b_1 A + b_2 B + b_3 C + b_{11} A^2 + b_{22} B^2 + b_{33} C^2 + b_{12} AB + b_{13}BC \quad (4)$$

(where a_o is a constant, b_n are constant of regression coefficients for n= 0,1,2,3; Y represents the responses: water loss (WL,%) or solute gain (SG,%); A B and C are the coded independent variables for temperature (°C) and sucrose concentrations (°Bx) and time (min) respectively. Statistical significance of the terms in the regression equations was tested for errors, and test of significance considering probability at confidence limits with $p<0.05$ was used for analysis of variance and test of lack of fit. Response surface plots were also generated. [22,23]

III. RESULTS AND DISCUSSION

3.1 Effects of process variables

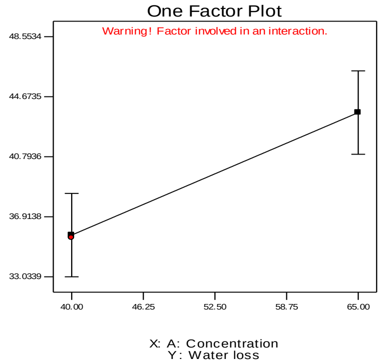
The results of the osmotic dehydration of mango evaluated with respect to water loss and solute gain are presented in Table 2.0. Each of the three variables used in the present study has its individual effect on water loss and solute gain in the osmotic dehydration of mango in sucrose solution. The effect of each of the variables was shown by the one factor plot generated for rate of water loss and solute gain Fig. 3 (a- f). Osmotic dehydration processing of mango was found to be directly related to sucrose concentration (SC) Fig.3 (a), thus showing that as sucrose concentration increases water loss was increased. But the effect of concentration on solute gain was affected negatively such that as SC is increased, solute gain decreases Fig, 3(b) especially at low temperature. This effect may be due to the increased viscosity and osmotic pressure of the sucrose syrup as the (SC) was increased. Consequently, higher water loss was achieved while the solute gain is hindered by the semi-permeable membranes of the mango tissue. The results is in agreement with the findings of other researchers on the modelling with Recurrent Artificial Neural Network that at higher medium concentration lead to a faster water loss, sucrose also enhanced the formation of a sugar surface layer, which becomes a barrier to the solute uptake [24].

Fig.3 (c) and (d) revealed that temperature did not have significant effect on water loss within temperature range of 30 to 45°C but the effect was more pronounced with solute gain Fig.3(d). The time of immersion had greater effect on water loss as observed in Fig 3e than for solute gain Fig.3f. The effect of solution concentration on mass transfer kinetics was higher than temperature effect in mango dehydration. A similar result was obtained on the evaluation of water and sucrose diffusion coefficient during osmotic dehydration of jenipapo fruit [25].

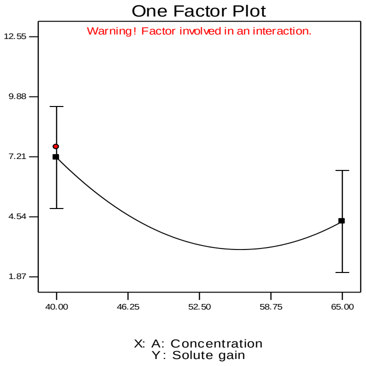
The combined effects of sucrose concentration, temperature and time were expressed in 3-D surface plot Fig 4. (a) to (a-f). It was evident that the responses were either enhanced or depressed by these combined effects. Figure 4 (a) and (b) revealed that immersion time had significant effect on water loss than on solute gain. The temperature exhibited relevance on solute gain at 45 ^{0}C and above, hence at higher temperature solute impregnation was enhanced. This may be attributed to rupturing of the cell structure at higher temperature and immediate solute impregnation which accounted for the sharp increase in solute gain. The effect of increased SC resulted in high water loss up to about 42.8% of initial moisture content Fig. 4(c). A suppressed effect was observed in solute gain as sucrose concentration increased until above 46.25 ^{0}Bx as seen in Fig.4 (d). The depression may be attributed to the fact that as concentration increased, the osmotic solutions were observed to be more viscous and therefore, the solutes had more difficulties in penetration. Figs. 4(e) and 4(f) revealed the dependency of water loss and solute gain on concentration and temperature. It was evident that the effect of concentration on water loss was higher than that of temperature while sucrose concentrations during the process were observed to be dependent on time to improve the degree of water loss. Therefore, time is a very important variable in the osmotic dehydration of mango fruits. A comparison of predicted and actual data used to monitor the osmotic dehydration process during the experiment and validation calculations is presented in Fig.5. There was a very good adequacy between predicted and observed data with correlation coefficient 'R' higher than 0.9897 for water loss and 0.8382 for solute gain. This fact also confirmed that the model equation is a good representation of the process and so can be used for process development purposes.

TABLE 2: Experimental result OD of mango samples by Design Expert.

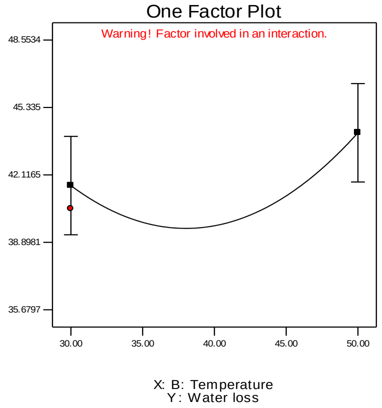
Expt No	Concentration (^{0}Bx)	Temperature (^{0} C)	Time (mins)	Water Loss (%)	Solute Gain (%)
1	-1.000	-1.000	1.000	37.09	7.770
2	0.0000	1.000	-1.000	20.29	8.110
3	1.000	-1.000	1.000	45.67	5.860
4	1.000	-1.000	-1.000	19.58	2.860
5	-1.000	1.000	0.0000	30.82	12.55
6	1.000	0.0000	-1.000	20.01	3.810
7	0.0000	-1.000	1.000	40.50	3.810
8	1.000	1.000	1.000	47.23	6.960
9	-1.000	-1.000	-1.000	11.62	8.710
10	1.000	1.000	1.000	48.21	11.64
11	-1.000	1.000	-1.000	13.72	8.110
12	-1.000	-1.000	-1.000	10.25	7.240
13	-1.000	0.0000	1.000	35.55	7.640
14	1.000	-1.000	-1.000	18.43	2.940
15	0.0000	0.0000	0.0000	26.34	1.870
16	0.0000	0.0000	0.0000	29.90	2.950
17	1.000	1.000	-1.000	30.64	9.960
18	1.000	-1.000	0.0000	35.44	2.510



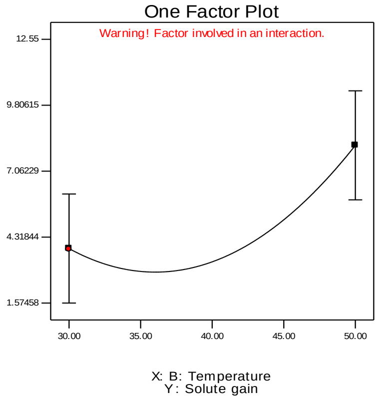
(a) Effect of Concentration on water loss



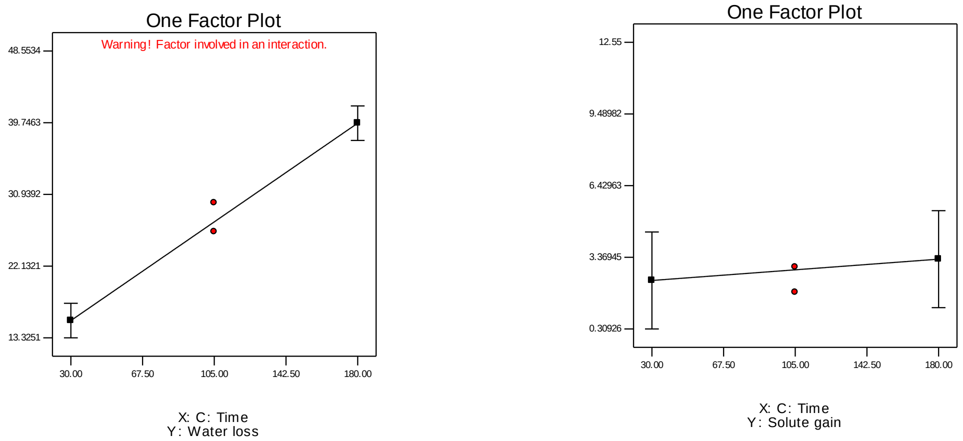
(b) Effect of concentration on solute gain



(c) Effect of temperatureon water loss



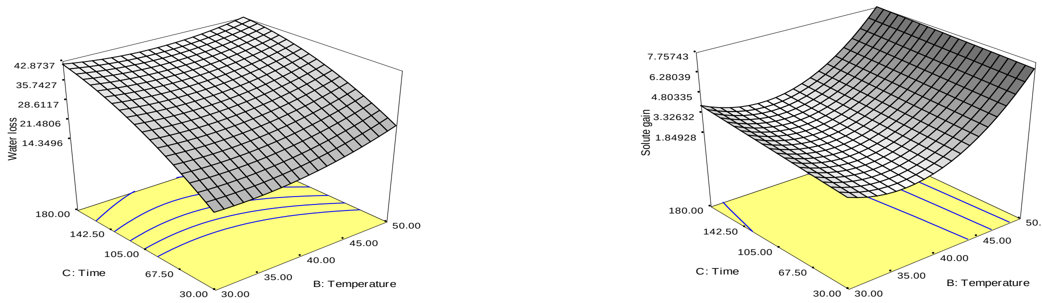
(d) Effect of Temperature on solute gain



(e) Effect of time of immersion on solute gain

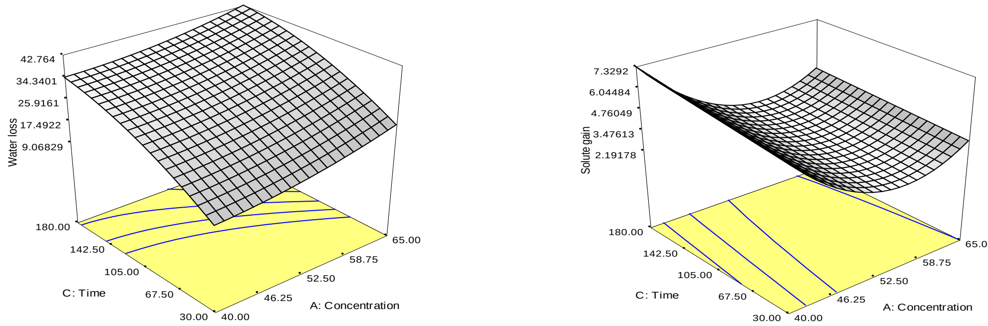
(f) Effect time of immersion on water loss

Figures 3 (a) – (f) showing the one factor plot the effects of variables on water loss and solute gain



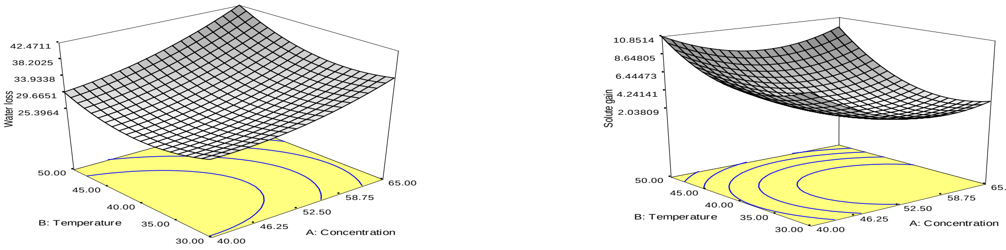
(a) Effect of temperature and time on WL

(b) Effect of temperature and time on SG



(c) Effect of Concentration and time on WL

(d) Effect of concentration and time on SG



(e) Temperature and temperature on WL

(f) Temperature and temperature on SG

Figure 4. (a-f): Effects of combined process variables on water loss (WL) and solute gain (SG)

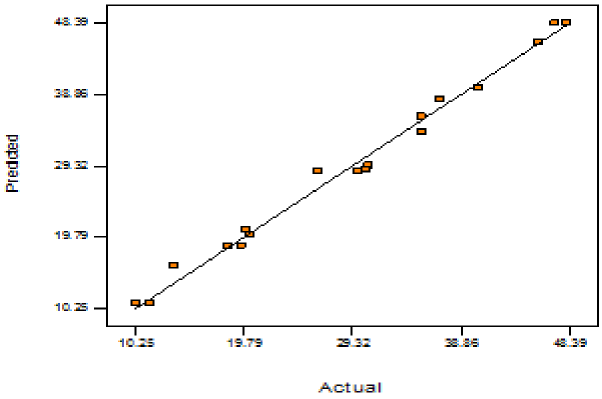


Figure 5: Predicted versus actual data plot for the water loss

3.2 Modelling and optimization process

Process variables of sucrose concentration, temperature, and immersion time were modeled for water loss and solute gain. The modified final equations in terms of coded factors A, B and C are presented in Eqns. (5) and (6) as fitted for variables defined in equation (4). The evaluation of osmotic dehydration to achieve specific water losses and solute gain was predicted from

$$\text{Water loss} = 28.64+5.15A+1.89B+11.70C+2.29A^2+2.99B^2-3.66C^2+1.30AB-1.91BC \quad (5)$$

$$\text{Solute gain} = 2.77-1.45A+2.17B+0.48C+2.43A^2+2.83B^2+0.80AB-0.16AC \quad (6)$$

To test the fit of the model, the regression equation and coefficient (R^2) were evaluated. Table 3 and 4 summarised the results for the response surface quadratic model for water loss and solute gain respectively. Some non-significant terms ($P < 0.05$) were eliminated to improve the status of the model. The ANOVA for the models as fitted show significance ($P < 0.05$). The Lack of Fit designed to determine whether the selected model is adequate described the lack of fit, not significant at ($P > 0.05$) F–test. The low probability value (< 0:0001) for both WL and SG indicated that the model is significant.Therefore, the models as fitted provide an approximation to the true system. The model F-value of 86.71 implies the model is good and values of "Prob > F" less than 0.0500 indicate model terms are significant. At this instance, variables A, B, C, B^2 ,C^2 and BC are significant model terms for water loss while A, B, A^2 and B^2 are significant model terms for solute gain. Thus the dependency of solute gain on sucrose concentration and temperature of dehydration and not significantly on time of immersion was demonstrated. In both cases, the lack of fit were insignificant with p> 0.05 confidence limit, hence the models represented the experiments data effectively and so can be optimised.

The desired criteria for effective osmotic dehydration process were to have maximum water loss and minimum solute gain. The results of evaluated optimization conditions based on the stated criteria is presented in Table 5.

TABLE 3: Analysis of variance (ANOVA) of water loss

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	2493.48	8	311.69	108.11	< 0.0001	significant
A	27.09	1	327.09	113.45	< 0.0001	
B	44.00	1	44.00	15.26	0.0036	
C	1690.30	1	1690.30	586.30	< 0.0001	
A^2	10.91	1	10.91	3.79	0.0836	
B^2	22.18	1	22.18	7.69	0.0216	
C^2	26.41	1	26.41	9.16	0.0143	
AB	13.87	1	13.87	4.81	0.0559	
BC	30.73	1	30.73	10.66	0.0098	
Residual	25.95	9	2.88			
Lack of Fit	17.53	5	3.51	1.67	0.3205	not significant

TABLE 4: Analysis of variance (ANOVA) of solute gain

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	150.08	6	25.01	9.41	0.0008	significant
A	26.881	26.88	10.11	0.0088		
B	61.271	61.27	23.04	0.0006		
C	2.75 1	2.75	1.04	0.3307		
A^2	16.381	16.38	6.16	0.0305		
B^2	19.651	19.65	7.39	0.0200		
AB	6.80 1	6.80	2.56	0.1382		
Residual	29.2511	2.66				
Lack of Fit	16.647	2.38	0.75	0.6520not significant		

TABLE 5: Solutions of optimisation for OD in mango samples

No	Conc	Temp	Time	Water Loss	Solute Gain	Desirability
1	55.70	46.22	175.5	38.97	4.145	0.8058
2	53.49	30.00	160.0	35.57	3.303	0.7793
3	52.11	30.00	166.4	35.98	3.601	0.7777

The choice of 53.49^0Bx, 30.0^0C and 160 minutes were preferred for economic reasons and to save energy requirement in achieving further dried products. The low temperature close to non-thermal processing will also help to minimize the undesirable effects of conventional thermal processing which may include charring, de-naturation and thermal decomposition. The 3D-plots of the osmotic dehydration process is overlaid in the contour plots for desirability, water loss and solute gain Fig 6 (a-c).

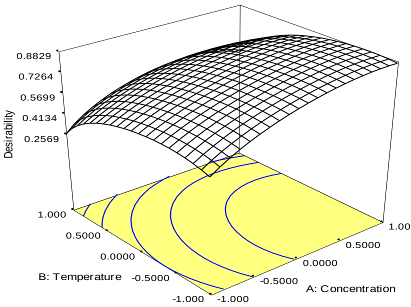


Figure 6a: 3D plot for Desirability of the OD process

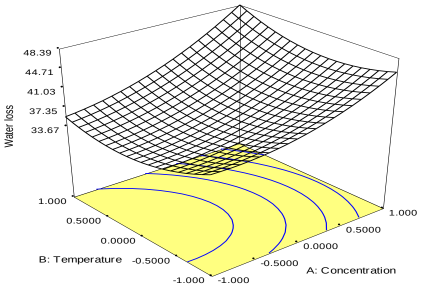


Figure. 6b: 3D plot for Water loss optimisation

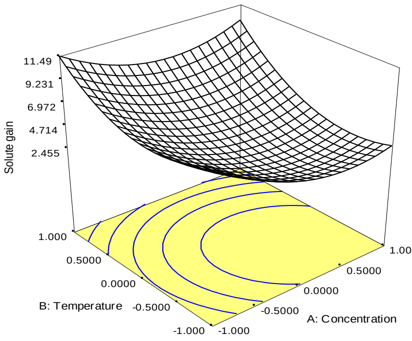


Figure 6c: 3D plot for solute gain optimization

IV. CONCLUSION

An increased concentration of sucrose solutions is associated with increase in the dewatering effect but not with an increased uptake of sucrose, the viscosity of the solution being a limiting factor. Water loss and solute gain during osmotic dehydration were significantly influenced by sucrose concentration and temperature especially at higher temperature values above 45 ^{0}C. Temperature had a positive influence on water and solids diffusivity resulting in greater water loss and solids gain. Time was a very important variable in the osmotic dehydration of mango fruits on which other variables depend for the process to attain an equilibrium. The optimized conditions of 53.49^{0}Bx, 30.0^{0}C and 160 minutes capable of removing about 42.6% of initial water content in mango sample can be used in the design and control of an OD process.

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