Optimal Osmotic Dehydration of Pieceform Mango in a Semi-continuous Operation

*Oladejo Duduyemi ^a, P.O. Ngoddy ^b, B.I.O. Ade-Omowaye^c

^aDepartment of Chemical and Polymer Engineering, Lagos State University, Epe campus, Lagos, Nigeria.
 ^bDepartment of Food Science and Technology, University of Nigeria, Nsukka, Nigeria.
 ^cDepartment of Food Science and Engineering Ladoke Akintola University of Technology, Ogbomoso, Nigeria.

*E-mail: engrduduyemi@yahoo.com, Phn: +234-8023920152

Abstract : Osmotic dehydration of mango fruits (Mangifer indica L.) is directed towards reduction of its seasonal losses at glut. Effects of sucrose concentrations; 40 to 65°Brix; temperatures; 30 to 50 °C and time of immersion between 0 to 180 min were studied using samples of diced mango cubes of 2 cm3 dimensions. The measured response variables of water loss, solute gain and performance ratio were subjected to statistical analysis. The results were modelled and optimised on the premise of maximum WL and minimum SG and maximum PR as constraints using the modified distance approach of the response surface methodology (Design Expert 6.08). Optimal osmotic dehydration evaluated were 59.19oBx, 32.06oC and 156.min. of 53.03°Bx sucrose concentration yielded 42.32% WL, 3.41% SG and PR of 10.69 with a desirability of about 89.73%. The results showed that all the input process variables had a significant effect at 5% level of significance (P < 0.05). Optimal process conditions adopted in a semi-continuous pilot plant yielded about 47 (%) water loss and 7.1 (%) solute gain of the initial sample content. The system proved efficient in the treatment of mango with performance ratio in the range of 3 – 7 and could be used in the food industry for the preservation of fruits. More so, the model could be used to predict operational conditions for the OD of other similar fruits.

Running title: Optimal osmotic dehydration of mango cubes

Keywords: osmotic dehydration, kinetics, flow rate, impingement agitation, optimisation, performance ratio

1.0 INTRODUCTION

Most agricultural products are seasonal. They are characterised with excessive losses in season and utmost scarcity when out of season. In the environment of third world countries, appropriate post harvest technologies are far from adequate. These features combine to generate gross annual losses in food and nutritional values that have been estimated to be in excess of 40-percent [1, 2]. Mango is one of the most important commercial fruits cultivated worldwide for social, economic, and nutritional purposes. Nigeria was ranked 10th among the largest mango producing countries in the world [3]. Unripe mangoes are rich in vitamin C, while the ripe fruits are rich in provitamin A and contain; moderate levels of vitamin C and some other vitamins. All mango varieties represent a potential source of natural antioxidants [4, 5].

Osmotic dehydration (OD) is a complementary treatment in the processing of natural foods and it involves the immersion of food materials in hypertonic solution (osmolite) which imposes osmotic pressure as mechanism of dehydration. The

phenomenon imposes minimal damages to the colour and flavour of treated food, inhibits enzymatic browning and reduces the energy and cost of further drying [6]. In addition, it has been shown to be a good method to obtain minimally processed fruits with improved organoleptic properties due to considerable sensory similarity recorded between the dehydrated and 'in-natura' products [7, 8]. Nowadays, the consumption of fresh fruits and vegetables have been increasing in popularity to satisfy the growing market demand for commodities in a fresh-like state compared to canned products [9]. The practicality of osmotic dehydration as an pre-treatment has effective measure been demonstrated but mostly on laboratory batch scale operations.

One of the problems militating against adopting osmotic-dehydration in food industry is the dearth of information on the design and operational conditions for industrial processing [10, 11]. Investigated variables known to influence OD kinetics include: composition and concentration of the osmotic agent, specific surface, shape and thickness of samples, agitation mechanism, liquor-product ratio,

temperature of treatment [12-15]. In spite of the numerous studies reported, it is difficult to establish general rules about the variables that affect osmotic dehydration [16]. The processing steps involved in recycling of spent osmotic solution still remain proprietary in the form of patents [17]. Essentially new technologies are needed to enhance quality, reduce energy consumption, improve safety and reduce environmental impact of the process [18].

Deepened knowledge of mass transfer mechanisms in OD revealed water loss, solute impregnation and leaching which is considered negligible as important and that the storage of osmo-dehydrated mango pieces at relative humidity between 64.8 to 75.5 per cent would be conducive for the retention of colour, flavour, texture and taste [19]. The fruit looses water and gains solids, high solute gain results in undesirable changes in the sensorial assessment [20]. One major way to harness the processing variables for the attainment of the desired moisture content that minimises microbial growth and degradation of product quality is by the optimisation of the most effective factors with the desired constraints. Statistical analysis is a powerful tool that has been used to account for the primary as well as the interactive influences of different process parameters. The application of response surface methodology (RSM) with central composite design method (CCRD) has been applied successfully to study osmotic dehydration [21, 22]. Also, recurrent artificial neural network architecture for modelling mass transfer kinetics has been used in the empirical study of relationships between numbers of input variables [23]. The aim of this study was to maximize the performance ratio (PR) defined as the ratio of water loss to solute gain in the osmotic dehydration of mango cubes. In this paper, the modified distance design of the response surface methodology (RSM) is used in the analysis and optimisation of the most significant variables of (OD) as conditions for optimal osmotic dehydration of fruits in a pilot plant developed for this purpose to operate on a semi-continuous basis.

2.0 MATERIALS AND METHODS

Mango fruits were obtained from a local farm in Epe, suburban region of Lagos, Nigeria. At the early stage of ripening, similar size (average weight of about 650 g), and average diameter of 2.8 cm without damages were selected. The fruits were washed and pieceform samples were cut into cubes of dimensions 1.0 cm³ by a manual dicer to maintain relatively equal size and weight. Commercial food grade sucrose was procured from a major sale distributor in Ojota, Lagos. Hypertonic solutions were prepared by dissolving the required quantity of sugar in distilled water under the conditions dictated by the experimental design. Table 1. The resulting sucrose syrup concentrations (°Bx) were confirmed with refractometer RFM(100) - Bellingham and Stanley, Kent. The samples were kept overnight to equilibrate before use [24]. The average moisture and initial sugar content in the fresh mango fruit was determined [25].

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

Factor	Name	Units	Type	Low Actual	High Actual	Low coded	High coded
X_1	Concentration	0 Bx	Numeric	40.00	60.00	-1.000	1.000
X_2	Temperature	0 C	Numeric	30.00	50.00	-1.000	1.000
X_3	Time	Min	Numeric	30.00	180.0	-1.000	1.000

In the first part of the assays involving determination of osmotic dehydration kinetics and optimisation, the samples were immersed in process solution at a ratio 1:5 to avoid solution dilution at specified temperatures in a controlled shaker agitator at a speed of 70 rpm. The modified distance set- up of the Design Expert 6.0.8 Start Ease Inc (2002) model was evaluated for two replicates with seventeen set of experiments .After the specified immersion times, the mango cube samples were removed from the osmotic solution and rinsed with water to remove surplus solvent adhering to the surfaces. These osmotically dehydrated cubes were then spread on absorbent paper to remove free water present on the surface.

During experimentation, it was assumed that the amount of solute leaching out of the mango samples during osmosis was negligible. Dehydrated samples were further dried to constant weight at 70°C

temperature. The water loss (WL), solute gain (SG) and performance ratio (PR) as response variables were evaluated using equations (1-3) in accordance with Azoubel and Silva, [26].:

$$WL(\%) = \frac{ww_o - (tw - ws)}{w_o} \times 100$$

$$SG(\%) = \frac{ws - ws_o}{w_o} \times 100$$

Performance Ratio =
$$\frac{WL(\%)}{SG(\%)}$$

Where: tw is the total wet weight of the mango fruit piece at the time of the sampling, ws is the total solids weight, ws₀ is the initial weight of solids, ww₀ the initial weight of water and w₀ the total initial weight of the sample.

Statistical significance of (p<0.05) was used in the analysis of variance (ANOVA). Optimisation of process variables were carried out by identifying the desirability of process variables with observed and predicted values [27]. A second order polynomial model from the regression analysis was fitted according to equation 4:

$$Y = a_0 + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 + b_{12} X_1 X_2 + b_{13} X_2 X_3$$
(4)

(where a_0 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,%); X_1 , X_2 , and X_3 are the coded independent variables for temperature (°C) and sucrose concentrations (°Bx) and Time (min) respectively.

The models generated were optimised with characterizing constraints of maximum water loss minimum solute gain and maximum performance ration.

The second part of the assay was the semi-continuous osmotic dehydration in a pilot plant. The outcomes of the optimisation process were set as operating parameters in a customised semi-continuous flow osmotic dehydration pilot plant. The pilot plant powered by electric source was conditioned to a control mechanism containing an electric heater and a thermocouple connected to a PID controller system as a feedback temperature control device. The set up was charged with sucrose concentration of 52.5 °Bx in the ratio of 1:20 sample to sucrose solution (w/w of the total weight of the samples) to enhance agitation by liquid impingement and effective circulation with minimal dilution over the stationary phase for sample materials Figure 1:

Weighed and labelled mango samples introduced into the contactor were removed at intervals of 30 min, rinsed quickly with distilled water to rid-off adhering concentrated solution at the surfaces, carefully blotted with tissue paper and weighed. The samples were analysed for water loss (WL, %), solute gain (SG, %) and quality of dehydration by the performance ratio. The experiments were repeated up to 180 min, in triplicate and average values were recorded.



Figure 1: Pictorial section of the semi-continuous osmotic dehydration contactor

3.0 RESULTS AND DISCUSSION

3.1 Initial characteristics of mango sample

The average moisture content of the sample as determined at 70 °C after 24 hr in a vacuum oven (Genlab MINO/50) was 82.37±0.5 % on a wet basis. The initial sugar content in the fresh mango fruit was determined to be 15.91±0.5 °Bx. These results are in agreement with reported values of sugar concentration of 15.19±1.2 (°Bx) and water content of 80.85±0.11%, for fresh mango [28]. The moisture content of fruit pulp of different mango germplasms to was reported to be in the range of 74.58 to 86.36%.[29]

3.2 Osmotic dehydration kinetics on mango samples

Experimental design and evaluated responses of solute gain (SG), water loss (WL) and performance ratio (PR) are as presented in coded terms in Table 2. The overall effect of osmotic dehydration on the physical appraisal of mango sample revealed minimal structural deformation as observed on treated samples shown in Figures 2(a), 2(b), and 2(c). Osmotic dehydration, a commonly used pre-treatment for fruits, involves diffusion of water from food material to surrounding outside solution which is replaced by the solute from the same solution. Combination of dehydration and impregnation processes resulted in modified functional properties of food which are favourable for drying and result in better product quality when these properties are integrated.

TABLE 2: Evaluated Responses of osmotic dehydration to the input variables

Run	Factor 1 X ₁ :Conc	Factor 2 X ₂ :Temp	Factor 3 X ₃ :Time	Response 1 Water Loss	Response 2 Solute Gain	Response 3 Performance
	°Bx	°C	min	%	%	Ratio
1	40.00	30.00	180.0	37.09	7.77	4.77
2	52.50	50.00	30.00	20.29	8.11	2.50
3	65.00	30.00	180.0	45.67	5.86	7.79
4	65.00	30.00	30.00	19.58	2.86	6.85
5	40.00	50.00	105.0	30.82	12.55	2.46
6	65.00	40.00	30.00	20.01	3.81	5.25
7	52.50	30.00	180.0	40.50	3.81	10.63
8	65.00	50.00	180.0	47.23	6.96	6.79
9	40.00	30.00	30.00	10.62	7.71	1.38
10	65.00	50.00	180.0	48.21	11.64	4.14
11	40.00	50.00	30.00	13.72	8.11	1.69
12	40.00	30.00	30.00	10.25	7.24	1.42
13	40.00	40.00	180.0	35.55	7.64	4.65
14	65.00	30.00	30.00	18.43	2.94	6.27
15	52.50	40.00	105.0	28.34	2.87	9.88
16	52.50	40.00	105.0	29.90	2.95	10.14
17	65.00	50.00	30.00	30.64	9.96	3.08
18	65.00	30.00	105.0	35.44	3.21	11.04







(b): Osmotically dehydrated mango samples after $180\ \mathrm{min}.$

(a): Piece-form mango samples



(c): Dried, osmotically dehydrated mango samples at 70 $^{\circ}\mathrm{C}$

Figure 2: (a) Picture of Fresh, (b) Dehydrated and (c) Dried samples of mango

3.3 Effects of process variables

The result of water loss based on the effects of the process variables is revealed in Figures 3 and 4. One factor plot of concentration, time and temperature is presented in Figures. 3 (a), 3(b) and 3(c) respectively. The effect of concentration revealed a consistent loss of water from beginning to the end of the process as shown in Figure 3(a). Hence, water loss increased with sucrose concentration. This is apparently due to high osmotic driving force between germplasms/sap of the fresh fruit and the surrounding hypertonic medium [30]. It could also be deduced from Figure 3(b) that the time of immersion had the highest dehydration effect, implying that the longer the residence time of processing the better. Faster water loss with greater diffusivity had been reported to result in greater dehydration at 65 °C [31]. However, this is limited to the attainment of equilibrium or saturation. The least WL was recorded by temperature as a factor of dehydration, the differential effect between 30 to 50 °C had very small percentage yield Figure. 3(c). It indicated the feasibility of OD even at the ambient temperatures. The 3-D surface plots Figure. 4(a), 4(b) and 4(c) for WL revealed the quadratic as well as interactive effects of all the variables evidenced by a resultant improvement in the percentage of water loss of about 44%.

The one factor plot of solute gain showed that SG decreased as the sucrose concentration is increased Figure. 5(a) but increased with both temperature Figure. 5(b) and time Figure. 5(c). A marked increase in SG occurred above 45°C as SG of over 8% was recorded Figure. 5(b). High temperature was reported

to denature biochemical components of samples especially at the periphery of the tissues which ruptured to accommodate more solute infusion [32]. Increased solute gain was reported to have resulted in increasing internal resistance to mass transfer as observed in apple [33]. These findings may have been responsible for the response observed in Figure. 6(a). Minimum SG was recorded at high concentration above 52.5°Bx and temperature below 40°C as observed in Figure. 6(b). The suppressive effect of the combined interaction among the input variables is displayed such that as SG increased slightly with time, there were no effects at sucrose concentration above 46.25°Bx (Figure. 6 c). Higher sucrose concentration is higher in viscosity and this may affect contact effectiveness between solvent medium and product surface. In addition, sucrose allows the formation of sugar surface layers, which become a barrier to both the withdrawal of water and solute uptake during osmotic dehydration. The combination of temperature ranging up to 40 °C with sucrose concentration ranging between 40 °Bx and close to 60 °Bx or higher concentrations resulted in a lower solute gain. The interactive effects of the process variables viz-a-viz concentration, temperature and time were considered and 3D contour and surface response plotted showed that the effect of temperature on solute gain was dominant. It increased exponentially as the temperature of the medium was increased above 45 °C. The resultant effects might have led to blockage of trans-membrane pores rather than infiltration of solute so that in all likelihood, they enhanced water loss in comparison to SG. Therefore, the use of osmotic solutions at low concentration favoured impregnation at the expense of dehydration which is in agreement with findings in literature [34].

The performance ratio is a measure of the product quality, depending on the desired attribute of product. During OD, maximum WL is often desired and high PR is preferred. The one factor plot showed that high temperature is not desirable as the PR is continuously degraded Figure. 7(a), but was favoured by increasing sucrose concentration Figure. 7(b). Figure. 7(c) shows that even as the time of immersion was observed to favour OD, it had an effective time range between 105 to 142 min [Figure 8(c) and Figure. 8(a)]. High concentration with low temperature was shown to favour PR Figure. 9 (b) and that the highest PR resulted at sucrose concentrations about 58 °Bx in Figureure. 9(c). The PR of the process was in the range of 5 to 11. The lower and upper limits of each

parameter for the contour plot were defined, water loss 35.97-48.18% (of initial water content in fresh mango samples) and solute gain in the range of 4.40-11.24% (of initial solid content in fresh samples) were achieved in the investigation in order to reach the highest performance and best possible product quality. These values compared favourably with reported results for similar fruits, Indian gooseberry or aonla (*Phyllanthus emblica* L.) for which the range of values reported were: 32.0 to 49.2% for WL and 4.8 to 18.9% for SG [29].

The residence time of immersion is crucial in osmotic dehydration with respect to water loss and solute gain and is affected by sucrose concentration and temperature variations. As time of dehydration increased, water loss percentage increased in a polynomial pattern such that at 180 min of dehydration about 33% water loss was achieved.

However, at higher concentration of 65 °Bx and about 150 min, the percentage water loss of 42% on a wet basis was recorded thus showing the combined effects of the two variables. Minimum water loss was attained at the lowest concentration (40 °Bx) and time (30min), at the same temperature of 30 °C. This confirms that maximum water loss occurred at the highest sucrose concentration. This may be due primarily to increased osmotic concentration gradient between the fruit and the solution. This, in all probability, is due to the fact that concentration and time had a quadratic effect on water loss. The least result was recorded under the influence of temperature and time having a water loss of 40.8% in a sucrose concentration of (40 °Bx) Therefore, temperature and concentration of osmotic solutions are directly correlated with high osmotic dehydration

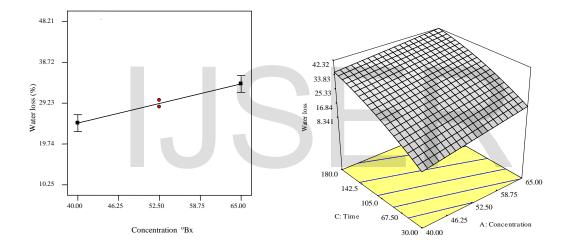
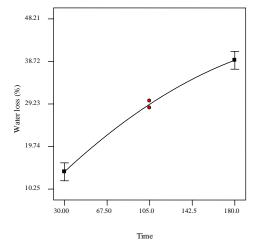


Figure 3(a): Plot of WL versus Concentration.

Figure 4(a): 3D Plot of time and conc. on WL



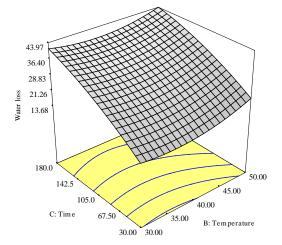
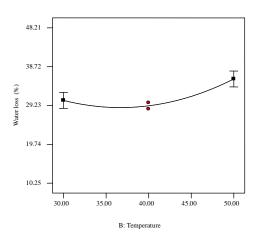


Figure 3(b): Plot of WL versus time.

Figure 4(b): 3D Plot of time and temperature on



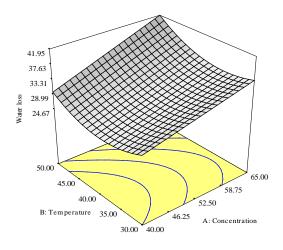


Figure 3(c): Plot of WL versus Temperature.

Figure 4(c): 3D Plot of temp and conc. on WL

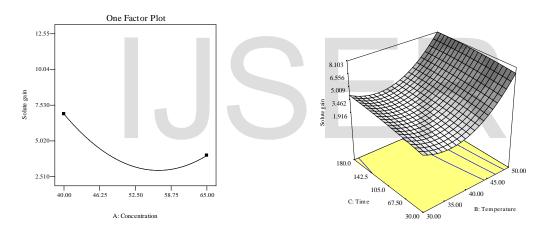


Figure 5(a): Plot of WL versus Concentration.

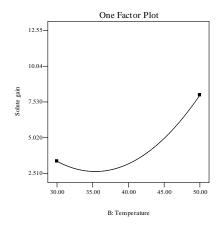


Figure 6(a): 3D Plot of time and conc. on WL

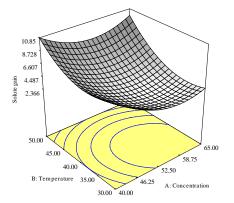
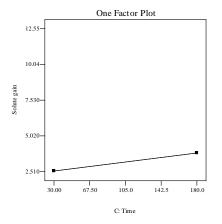


Figure 5(b): Plot of WL versus time.

Figure 6(b): 3D Plot of time and temperature on WL



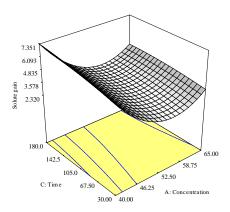


Figure 5(c): Plot of WL versus Temperature.

Figure 6(c): 3D Plot of temp and conc. on WL

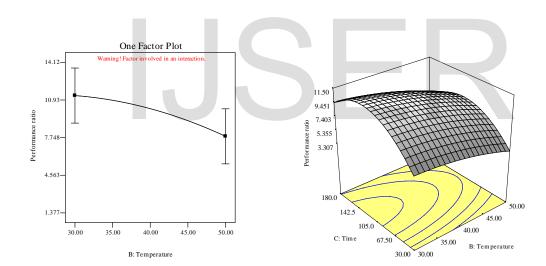


Figure 7(a): Plot of WL versus Concentration.

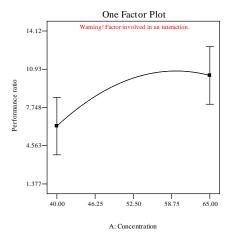


Figure 7(b): Plot of WL versus time.

Figure 8(a): 3D Plot of time and conc. on WL

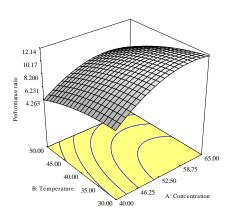


Figure 8(b): 3D Plot of time and temperature WL

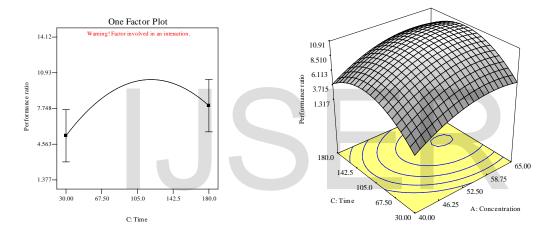


Figure 7(c): Plot of WL versus Temperature.

Figure 8(c): 3D Plot of temp and conc. on WL

3.4 Optimisation

The analysis of variance (ANOVA) of the results of the responses is given in Tables 3. The second-order model showed that each of the responses were significant (P < 0.05), overall non-significant lack of fit of the process made the results to be desired. The least "Pred R-Squared" of 0.6617 is in reasonable agreement with the "Adj R-Squared" values for WL, SG, and PR responses as a true representation of the process. The R - squared for WL, SG and PR were 99.5%, 81.1% and 87.5%, respectively. Therefore, the model was used to navigate the design space for optimisation of the desired characteristics of osmotic dehydration.

Regression analysis for different models indicated that the quadratic models fitted accounted for more than 95% of the variation in the experimental data. The value of regression coefficients and P levels for the coded form of process variables are presented in Tables 4. The regression coefficients of "Prob > F" less than 0.0500 values indicated that the model terms are significant and were fitted to polynomials equation 4. All linear terms of the process variables have a significant effect (P < 0.05), whereas the quadratic terms of concentration and interactions of "concentration temperature" have nonsignificant effect at 5% level of significance (P >0.05) on the water loss. The fitted responses were optimised for maximum water loss, incorporation of solute and maximum performance

ratio in order to obtain product that resemble their non-processed form. The values of input variables at optimisation were: 59.19 °Bx, 32.06 °C and 156.min

immersion time.. These were adopted as operating conditions for the pilot plant from the points of view of economic and energy requirements.

Table 3 Analysis of variance for water loss (WL) solutes gain (SG) and performance ratio (PR) in the osmotic dehydration of mango fruit in sucrose solution

	Water Loss		Solute Gain		Performance Ratio	
Source	SS	Prob>F	SS	Prob>F	SS	Prob>F
R sqrd	0.9948		0.8116		0.8753	
Adj.R sqrd	0.9890		0.7331		0.8233	
Pred. R sqrd	0.9593		0.6047		0.6617	
Lack of fit	non significant	0.08905	non	0.3972	non	0.06403
			significant		significant	
Pure error	2.43		11.07		11.07	
Residual	13.19		25.68		31.59	
Model terms	significant	0.0001	significant		significant	

Where s.s., sum of square; k., constant; m.s., mean square; n.s., non significant at 5% level.

TABLE 4: Final Model Equation in Terms of Coded Factors:

Coefficients	Water loss	Solute gain	Performance ratio
k	29.26	3.18	10.20
A	5.08	-1.46	2.12
В	2.10	2.33	-1.74
C	11.92	0.63	1.33
A^2	n.s.	2.27	-2.20
\mathbf{B}^2	3.38	2.51	n.s.
C^2	4.06	n.s.	-3.92
AB	n.s.	n.s.	n.s.
AC	-0.79	n.s.	n.s.
BC	-1.67	n.s.	n.s

where n.s.- non-significant (P > 0.05); A-concentration; B-temperature; C-time.

3.5 Semi-continuous dehydration

The purpose of the optimisation was carried out to generate operational conditions for processing food materials in the pilot plant for maximum performance. It was observed that the processes did not require the use of wire-gauss shielding to prevent floatation of samples as the impinging characteristics of pilot plant kept the samples in a continuously soaked condition. The sucrose concentration and temperature selected were kept constant while varying residence time was used to test the efficacy of the pilot plant as a tool for osmotic dehydration. Water loss was observed to increase linearly as the time of processing increased till about 180 mins of operation when maximum dehydration would have been attained as in Figure. 9, The perfomance ratio and quality assesment of dehydrated mango after 45min of treatment ranged from 6.0 to 9.5. This compared favourably and better than 5.16 reported under optimized conditions of 44 °C, 65.5 °Brix for

uncoated osmotically dehydrated mango cubes [20]. Trends in OD parameters evaluated for both WL and SG followed a similar pattern of continuous increase as processing time increased, albeit in an undulating manner. The SG increase was however kept minimal as desired while WL increased continuously.

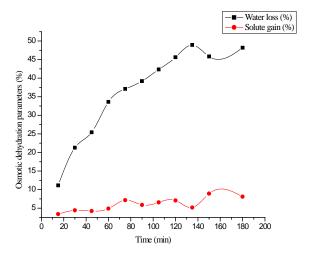
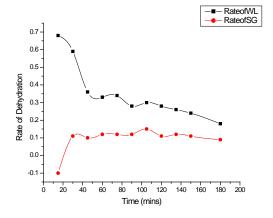


Figure 9: Rate of water loss in mango treated in semicontinuous operation

The peak of dehydration was attained between 120 and 130 min of operation which is an indication that osmotic dehydration in the pilot plant was faster and more efficient at the optimised conditions of the variables The un-interrupted pattern of dehydration was apparently due to the sustained prevalence of osmotic driving potentials between the sample and the surrounding hypertonic solution, enhanced with high liquor-to-sample ratio. The rate of dehydration helped to determine most active period of dehydration which occured within 90 to 120 min of dehydration Figure 5.



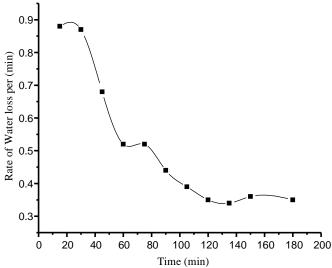


Figure 10: Evaluated rate of osmotic dehydration in the pilot plant

4.0 CONCLUSIONS

Osmotic dehydration in a pilot plant would appear to have facilitated the processing of mango samples typical of tropical fruits with its attending advantages of retention of initial fruit piece structural characteristics and organoleptic properties. The order of influence of the processing variables for WL is Time>Concentration >Temperature and for SG is Temperature > Concentration > Time in the decreasing order. The modified distance approach of response surface methodology is adequate for dehydration as numerical optimal osmotic optimisation yielded 42.32% WL, 3.41% SG and PR of 10.69 with a desirability of about 89.97.73% at input variables of 59.19 °Bx, 32.06°C and 156.min. The semi-continuous operation demonstrated a high degree of enhancement over counterpart batch operations with over 45% reduction in water content and minimal solute gain of about 6%. A reduction in process time from 160 min to about 120 min accounted for about 40% increase in efficiency in terms of residence time. The results demonstrate clearly that using the pilot plant accelerated the process of dehydration at optimal conditions and brought about the attainment of equilibrium/stability in significantly shorter time. Reduced drving time combined, on the one hand, with effective reconcentration and syrup re-cycling, on the other, within the pilot plant could significantly increase overall productivity, reduce operating cost and add value to the finished product and make fruits available all the year round.

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