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Effects of Agitation by Convective Liquid Impingement on Osmotic Dehydration of Carrot Disk in a Semi-Continuous Operation

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Abstract

The effective agitation of medium is salient to osmotic dehydration (OD), a process for improving organoleptic qualities and preservation of agro-products. The convective liquid impingement alleviated the burden of handling large volume of osmotic dehydration and agitation of delicate biological tissues. The convective liquid impingement was used in a semi-continuous system as alternative to mechanical and electrical vibrators for the osmotic dehydration of cylindrical carrot pieces. The effects of sucrose concentrations (40, 50 and 60 °Bx) temperatures (30, 40 and 50 ºC) and time (0-180 min) were optimised for maximum water loss and minimum solute gain with response surface methodology of the Design Expert 6.08. The optimisation results of 53.03 ^{o}Bx sucrose concentration, 32.44 ^{o}C temperature and 160 min were applied at turbulent flow regimes of 6-8 litres/min via a sprinkler of a pilot plant system. The technique achieved 48.60 % water loss and 4.50% solute gain in 150 min of processing.
Practical Application: The convective liquid impingement mechanism enhanced OD and enabled the use high sample: liquor ratio for industrial adoption of process.

(Keywords: agitation, semi-continuous, sucrose concentration, kinetics, impingement, flow-characteristics).

1. Introduction

Carrot is one of the essential root vegetables loaded with bioactive substances like carotenoids and dietary fibres with appreciable levels of functional components having considerable health-promoting properties. The consumption of vegetables such as carrot and their products is growing steadily due to their recognition as an important source of natural antioxidants which have been implicated in lowering degenerative diseases [1]. Fruits and vegetables are however prone to rapid deterioration by way of physiological processes called senescence or ageing [2].

Most advanced technologies in fruits and vegetables preservation are cost intensive and require constant power supply which is erratic in supply in most third world countries. Typical convective dryers account for about 85% of all industrial dryers and are the most energy-intensive processes in food processing industries [3]. Appropriate post harvest technologies are far from adequate, gross annual losses in food and nutritional value was estimated to be in excess of 40-percent [4, 5]. Preservation technologies that prolong their shelf-life of fruits and vegetables without detrimental effects on their sensory and nutritional qualities are increasingly being sought the world over. Promising technologies explored to achieve this goal include: dehydration or drying, fermentation, pickling, canning, juice extraction and chilling storage, freezing, freeze-drying, the use of chemical preservatives, etc [6].

Osmotic dehydration as a minimal processing technique are increasingly been used because of its numerous advantages which include mainly the retention of organoleptic qualities of treated food, reduction of water activity and as pre-treatment measure to food preservation [7]. In spite of the numerous studies that have been carried out on this subject and its proven advantages, it is still difficult to establish general rules about the variables that affect osmotic dehydration [8]. Drying, a commonly used method of food preservation in the developing countries, being one of the most energy-intensive processes in food processing industries requires more innovations to minimise the energy demand and improve the quality of the final products. The synergistic combinations of two or more of the techniques have achieved enhanced "huddle-effects" in drying [9]. Many authors recommended that the quality (colour, flavour and texture) of air or freeze dried fruits and vegetables was improved by a prior osmotic dehydration step during air drying [10, 11].

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The relevant factors employed to speed up water transfer include a high concentration of osmotic solution, low molecular weight of osmotic agent, processing temperature, stirring process or some pre-treatment techniques [12]. The qualities of osmotic dehydrated products are related not only to the water removed with minimal thermal stress but also to the impregnated solutes and the modification of the structure [13]. Apart from the influences of sucrose concentration, temperature, and processing time, the devices for medium agitation should prevent the rupturing of cell structure and unnecessary modification of the organoleptic properties of treated food materials. The dewatering effect was enhanced by solute concentration. However, solute uptake was limited because of a parallel increase of the viscosity of the solution and was considered as a restricting factor to the mass transfer [14].

The intensity of agitation is correlated with the Reynolds numbers (N_{Re}) of the flowing fluid and to the geometrical factors in the flow system, all of which have been shown to affect osmotic dehydration [15]. Majority of research work proclaiming the viability of OD as effective pre-treatment are primarily based on laboratory scale [16, 17]. The modes of agitation had been by mechanical and electrical shaker mechanism. Agitation was necessary for reducing the mass transfer resistance at the surface of the carrots and for good mixing and close temperature control in the osmotic medium [18]. Batch operations are characterised by enormous volume of solution and significant quantities of solutes on the industrial adoption of OD. Electrical and Mechanical shakers may not be effective especially in handling large volumes. These constraints and others might have made OD process unpopular in the food industries, especially to handle fragile food materials of fruits and vegetables.

The provision of automated operation control and online measurement facilities is a necessity to OD operation and acceptability [19]. Therefore, special attention is required for equipment design and this study focused on assessing the impact of agitation by convective liquid impingement on osmotic dehydration of carrot which has not been reported elsewhere.

2. Materials and Methods

2.1 Sample preparation and characteristics

The raw materials used in this study were carrot (*Daucus carota L*), commercial food grade sucrose procured from a local market in Epe town, Lagos. Selected carrot samples were deep yellow in colour and defect-free of average sizes of 2.50 to 4.0±0.5 cm diameter and between 10.0±0.5 cm to 15.0±0.5 cm length. The carrot was refrigerated at 7.0±2 ^{o}C in a custom-made airtight container to prevent contamination, humidification and evaporative drying in air before use. The carrots were washed in clean water, allowed to drain, peeled and diced to cylindrical disks of about 15.0±0.5 mm diameter and 15.0±0.5 mm length to maintain relatively equal size and weight.

Osmotic solution were prepared by dissolving determined quantity of sucrose in known volume of distilled water with sample to solution ratio of 1:5 (w/w) in order to minimize significant changes in concentration [20, 21]. The sugar content of the carrot samples measured in a RFM300 refractometer at 20 ^{o}C was 5.20 ^{o}Bx. The average initial moisture content of the fresh carrot samples and total solid content were determined gravimetrically by drying the samples in a vacuum oven at 70°C until a constant weight was obtained according to [22]. The average initial moisture content was determined to be 89.24 % (wet basis).

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2.2 Experimental designs and optimization

The variables studied were sucrose concentrations (40, 50, and 60 ^{o}Bx); temperatures (30, 40 and 50 ^{o}C) and immersion time from 0 to 180 min. The effects of these variables were studied by regression and optimized by response surface methodology of the Design Expert 6.0.8 Start Ease Inc (2002) software package. The independent uncoded variables and their coded values at the specified conditions are presented in Table 1.

Table 1: Coded and uncoded values of different process variables for optimization.

Factor	Name	Units	Type	Low Actual	High Actual	Low coded	High coded
A	Concentration	^{0}Bx	Numeric	40.00	60.00	-1.000	1.000
B	Temperature	^{0}C	Numeric	30.00	50.00	-1.000	1.000
C	Time	Min	Numeric	30.00	180.0	-1.000	1.000

Prepared carrot samples were weighed, labelled, osmotically dehydrated and the final weights recorded at the respective conditions of variables. Experiments were replicated thrice and samples analysed for water loss, and solute gain and the average values were recorded. The OD was achieved using an agitated water bath (Gallenkamp, Leicestershire, U.K.) at measured sucrose concentrations and regulated temperatures.

The optimisation of the process variables were constrained and evaluated in terms of maximum water loss (WL) and minimum solute gain (SG) within the respective range of variables. The results were subjected to Analysis of variance (ANOVA) using the General Quadratic Models procedure (GQM). At $p<0.05$ confidence limit, models were generated and the respective values of variables for the optimization process recorded. The outcome of the optimisation process was set as operating parameters in the pilot plant designed for this purpose.

2.3 Analysis of data

The calculations of the solute gain (SG), water loss (WL) and moisture content (MC) were determined by gravimetric measurement [23, 24]. Equations 1 to 3:

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

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

$$Percentage\ \text{moisture content} = \frac{\text{W}_0 - W_t}{W_0} \times 100\% \qquad (3)$$

where WL = water loss %; SG = solid gain % ;
M_W initial = initial water content before osmotic dehydration (g);
M $_{\text{initial}}$ = total weight of fresh sample before dehydration (g);
M $_{\text{final}}$ = final mass of sample after dehydration at set time (g);
M $_{s\ \text{initial}}$ = initial solid content in fresh sample (g);
$M_{s\ \text{final}}$ = final solid content after OD at specified time (g);
W_o and W_t = total moisture contents in fresh sample and after dehydration at time 't' respectively.
A dimensionless group called the Reynolds number was used to verify the presence or absence of turbulence in the system [25]. It was defined according to Equation 4:

$$N_{\text{Re}} = \frac{DV\rho}{\mu} = \frac{\rho V^2}{\mu V/D} = \frac{\rho N d^2}{\mu} \qquad (4)$$

where V is the average velocity in the pipe (flow rate), ρ is the fluid density, μ is the fluid viscosity and D is the tube diameter, N is the impeller revolution speed (rev/s) and d is the impeller diameter (m) for mixing tanks.

The intensity of agitation was correlated with flow characteristics of the system. The agitated systems were characterized by $N_{\text{Re}} > 10{,}000$ for turbulent flow across the sprinkler.

2.4 The pilot plant

The contactor vessel and the reservoir were calibrated to measure volume of liquid required in ml for each process run, and re-constitution of sucrose syrup in the bio-reactor for osmotic dehydration. Pump was set to operate at turbulent flow regime of 6 l/min of the liquid delivered into the osmotic chamber through 0.635 cm pipe by a 10 cm diameter pin hole sprayer cap to impinge the carrot samples continuously at the regulated flow rate imposed by the pump. The effect of agitation at the turbulent flow regime by liquid impingement enhanced homogeneous circulation of OD solution in the contacting chamber.

The medium heated electrically was equipped with a thermocouple connected to a (Proportional + Integral + Differential) feedback controller system to maintain relatively constant medium temperature at desired set point. The connection pipes and accessories were made of plastic fittings to prevent corrosion and contaminations. The pictorial view of the contactor of the semi-continuous pilot plant is shown in Plate 1.

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

2.5 Process description

The contactor of the pilot plant was filled in the ratio of 1:20 sample to sucrose solution (w/w) of the total weight of the carrot samples to be treated with 53.03°Bx sucrose concentration prepared and measured over a 300 RFM Refractometer. The PID controller device was set at 33 ^{0}C temperature, and

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the medium was allowed to circulate at the regulated flow rate of 6-8 litres /min for about 5 min to attain the set temperature. A voltage regulator assisted the pump to sustain turbulent flow regimes of agitation.

The cylindrical carrot samples prepared, weighed and labelled were introduced into the contactor chamber at the same time. At intervals of 15, 30, 45, 60, 90, 120, 150 and 180 min, the carrot samples were removed from the osmotic solutions, rinsed quickly with distilled water (within 30 sec.) to remove adhering solution at the surface and carefully blotted with tissue paper. Each sample was subjected to gravimetric analysis as outlined in equations 1 to 3. The experiments were repeated thrice and the average (±SD) values of weighed carrot samples before and after dehydration were recorded. The extent of water loss (WL, %) and solute impregnation/solute gain (SG, %) were evaluated.

3. Results and Discussion

The responses of carrot samples to osmotic dehydration (OD) in varying sucrose concentrations, SC, temperature (^{0}C) and processing time (min) were evaluated in terms of water loss (WL) and solute gain (SG). The trend and magnitude of water loss at different sucrose concentrations in the preliminary experiments were plotted in Fig. 1. It shows that water loss increased as sucrose concentration increased. The response of the samples to OD was observed to be non-linear as the rate was higher at the beginning of each of the experiments and reduced exponentially thereafter within the effective process time of 180 min.

A maximum WL of about 44.40% of initial water content was achieved at the highest SC of 60 ^{0}Bx, 180 min of dehydration at 40 ^{o}C temperature by the regression approach. Previous research findings revealed that higher concentrated solutions enhanced WL and SG due to greater osmotic pressure gradients [26, 27, 28]. A critical assessment of the trend depicts a closer profile of dehydration between 50 and 60 ^{o}Bx of SC than between 40 and 50 ^{o}Bx. This may be adduced to the manifestation of the effects of high SC and its characteristic osmotic pressure at the peripheral of the carrot samples membranes.

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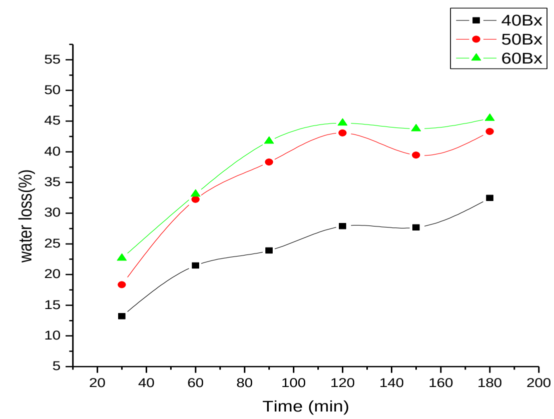

Figure. 1: Effects of sucrose concentration on water loss

The progress of dehydration with time showed an initial high rate of dehydration which reduced progressively towards attaining a state of equilibrium as shown in Fig. 2. The trend line equations for the time dependent operation are presented in Table 2. This could be used to predict the fraction of water loss at a given time. The trends of dehydration tended towards linearity at the maximum time of 180 min when the process is assumed to have attained saturation and further dehydration may not be feasible at the prevailing conditions.

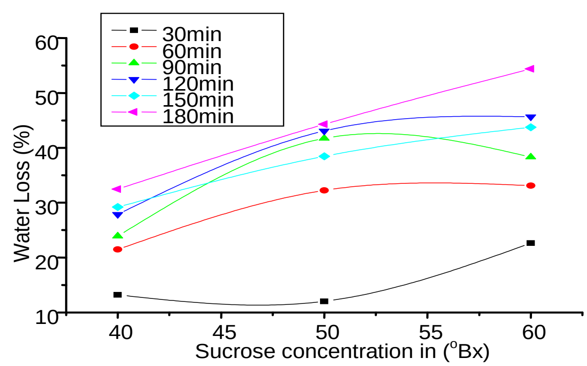

Fig. 2: Effect of immersion time on water loss at different sucrose concentrations

Table 2: The regression equations for water loss as function of time

Time (min)	Trend line Regression equations for water loss
30	$WL = -0.0086t^2 + 1.9576t - 32.061$
60	$WL = -0.0495t^2 + 5.5345t - 120.67$
90	$WL= -0.106t^2 + 11.324t - 259.37$
120	$WL = -0.0196t^2 + 2.6917t - 47.017$
150	$WL = -0.063t^2 + 7.1915t - 158.97$
180	$WL = -0.0086t^2 + 1.9576t - 32.061$

The magnitude of solute gain decreased as the SC increased, this is probably due to the increased viscosity of the sucrose solution that hinders the diffusion of solute into matrices of the sample. The solute gain in the process was more drastic at the beginning of dehydration especially at low SC which attained an assumed saturation before 150 min of processing (Fig. 3). The SC of 60 °Bx attained higher solute gain faster than the lower SC but with the least overall solute gain. This was an indication of peripheral blockage rather than solute impregnation of samples. It could be seen that higher SC favours both water loss and solute gain at minimal process time. High osmotic pressure gradient and increased viscosity of high sucrose concentrations might be responsible for this observation. The high SC might lead to loss of plasmatic cell membrane functionality at the periphery of the samples [29]. The magnitude of WL in percent of sample mass compared very well to previous findings for Indian gooseberry or aonla (*Phyllanthus emblica* L.) for which the range of values reported were; 32.0 to 49.2% for WL, 4.8 to 18.9% for SG [30].

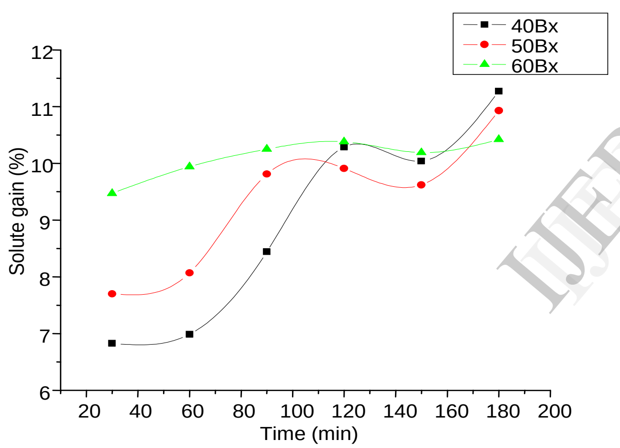

Figure 3: Effects of sucrose concentrations and time on solute gain

The effect of temperature on water loss at the same 40 °Bx SC but varying temperatures of 30, 40, and 50 °C, within 0 to 180 min was illustrated (Fig 4). The magnitudes of WL were positive and non- linear for each temperature. Average water losses (WL) on wet basis were 30.5, 38.4 and 32.8% for 30, 40, and 50 °C respectively. Highest water loss was recorded at 40 °C. It implies that the effect of temperature on water loss did not increase proportionately with temperature but are more effective at moderate temperature. The temperature effect on SG is presented in Fig. 5. It shows linear dependency of solute gain on temperature. These results are similar to those reported for osmotic dehydration of potatoes where increasing process temperature up to 45 °C resulted in increased WL and SG rates, in favour of higher WL/SG ratios [31].

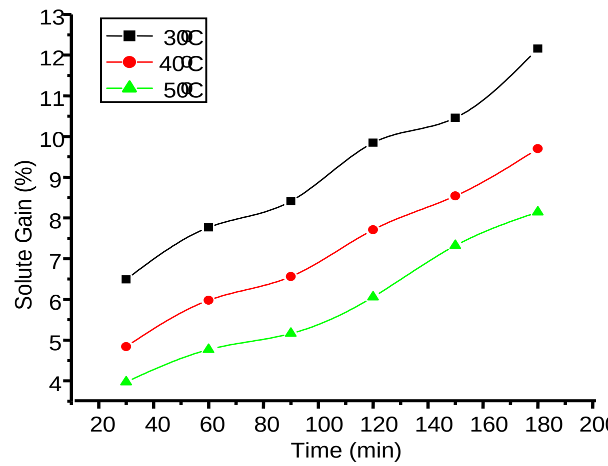

Figure 5: Effect of temperature and time on solute gain in 40 °Bx

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In this case, the influence of temperature was observed to increase the kinetics of mass transfer at the moderate temperature which could be attributed to diffusion rate enhancement by the thermodynamic properties of the media. Solution viscosity is directly related to temperature, and therefore increasing temperatures led to great reductions in viscosity. Consequently, temperature increases the flow characteristics of the medium. However, higher temperatures ≥ 50 °C temperatures have been reported to damage the structures of the samples. In several studies, membrane destruction at higher temperature has been reported and this led to higher solid uptake by plant-based materials during osmotic treatment [32].

The results for the process variables were optimised using Design Expert 6.0.8 Start Ease Inc (2002) software package. The optimum conditions for osmotic dehydration of carrot sample were found to be 53.03°Bx sucrose concentration and 32.44°C temperature and 160 min to achieve 46.87% water loss and 7.33% solute gain. This compares favourably with optimum process conditions of 52.5°Brix sucrose syrup concentration, 49 °C osmotic solution temperature and 150-min process duration reported for carrot cubes [18].

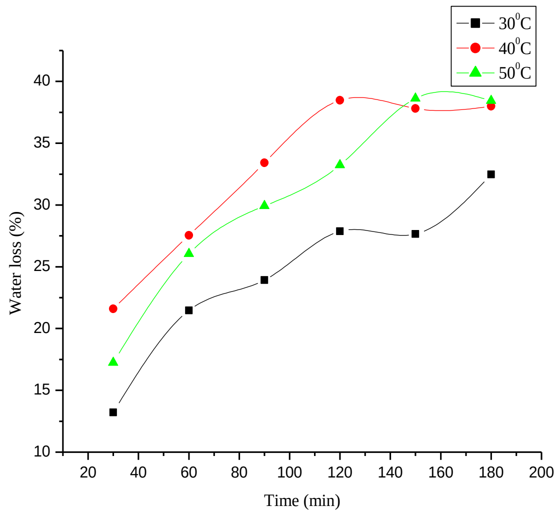

Figure 4: Effects of temperature and time on water loss in 40 ^{o}Bx.

The result of agitation by the convective liquid impingement on osmotic dehydration of carrot samples was also evaluated in terms of water loss and solute gain. The WL was observed to increase continuously with processing time up to about 180 min of operation as shown in Fig. 5. The un-interrupted pattern of dehydration was apparently due to osmotic pressure potentials between fresh carrot samples (stationary phase) and the surrounding hypertonic solution (continuous phase).

This sustained potential difference may be as a result of high sample: liquor ratio on one hand and the high agitation by the impinging characteristics of fluid flow on the other. The maximum WL of 48.6 % of the initial water content in fresh carrot sample was attained at about 150 min of processing. The fluid agitation by turbulent flow defined by Re > 10,000 was observed to show no eventualities of rupturing the sample and it exhibited higher degree of dehydration. The impinging effects also prevented solute deposition on sample surface. Hence, the essence of using wire gauze to submerge samples in viscous concentrated sucrose syrup/solution was un-necessary.

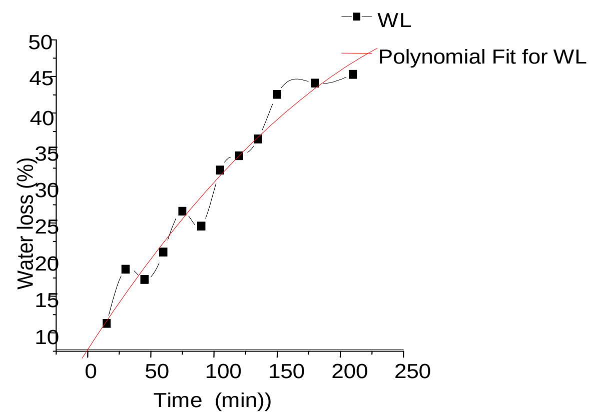

Figure 5: Water loss kinetics of carrot during OD process in the pilot plant

The solute gain was observed to increase steadily with time after an initial relapse between 0 and about 10 min (Fig. 6). This may be attributed to the leaching of nutrients from samples or that effective solute impregnation commenced from about 15 min of contact. The SG was in the range of 3.0 to 4.5% for a duration of 180 min which shows that the variables had little influence on SG but depended on the internal mechanism. Similar investigation indicated that mass transfers in tomato were probably governed by a predominantly diffusional mechanism, and the only resistance to this solute transport was the internal resistance ([33]

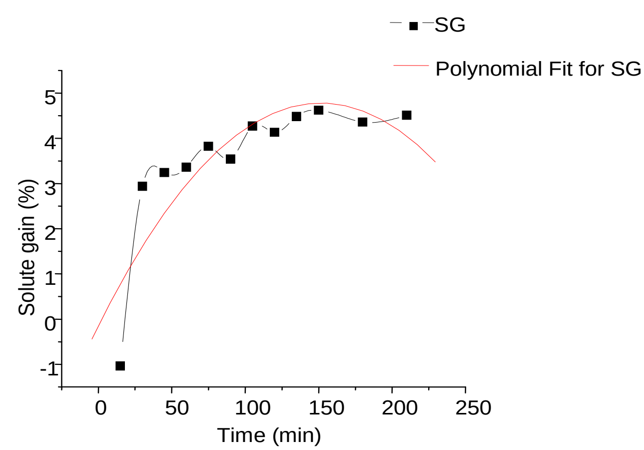

Figure 6: Solute gain kinetics of carrot during OD process in the pilot plant

The statistical analysis of WL and SG data from the convective liquid impingement is presented in Table 2 using the 'OriginPro' 2007. The regression-square (R^2) of 96.57% at confidence limit of $p<0.05$ generated equations 6 and 7 for WL and

SG respectively. This infers that the equations are good representation of the process and that equations guiding the process are also of second order polynomial but with positive constant term of 7.74 for water loss as compared to trend line equations in Table 1.

Table 2: Regression analysis of the WL and SG Data from the pilot plant process

Parameter	Water loss Value	Error	Solute gain Value	Error
A	7.74006	2.18302	-0.14678	0.82085
B1	0.26565	0.04527	0.06499	0.01702
B2	-3.76907E-4	1.99747E-4	-2.14375E-4	7.51078E-5
R-Square (R^2)	0.9657	0.73744		
SD	2.3196	0.8722		
N	12	12		
P	<0.0001	0.00244		

Where A, B1 and B2 are constants of the polynomial order, SD is the standard deviation, N is the number of sample points and P is the probability test point and t represents the time dependent variable of the semi-continuous operation.

The model equations for the water loss and solute gain of the treated samples in the pilot plant are given in Equations 6 and 7:

$$WL = 7.74006 + 0.26565t - 3.76907E\text{-}4\ t^2 \quad (6)$$

$$SG = -0.14678 + 0.06499t - 2.14375E\text{-}4\ t^2 \quad (7)$$

These results show that the time dependent OD with convective liquid impingement is efficient and preferable in osmotic dehydration because it was faster. In all cases of drying associated with drying time, volume of moisture in a material to be removed or the rate at which drying is accomplished is highly related to efficiency and energy demand. An improvement in energy efficiency by only 1% could result in as much as 10% increase in profits [3, 34] Therefore, OD as an upstream pre-treatment, using the convective fluid impingement process could be adopted to make industrial application of OD easier and could be applied to different classes of fruit and vegetables.

4. Conclusions

The sucrose concentration, temperature, immersion time and agitation are important factors influencing the extent of osmotic dehydration in carrot samples. Increased sucrose concentration affected both water loss and solute gain positively. A maximum WL of about 44.40% of initial water content of carrot was achieved at the SC of 60^0Bx, 180 min of dehydration at 40^oC for the preliminary experiments by regression. The optimum conditions for osmotic dehydration of carrot sample were 53.03°Bx sucrose concentration and 32.44^oC temperature and 150 min to achieve 46.87% water loss and 7.33% solute gain. Convective liquid impingement method was associated with enhanced WL of 48.69 % of the initial water content in fresh carrot sample at reduced process time of 150 min with minimum solute uptake of about 4.5 %. The beneficial effects of applying osmotic dehydration with impingement on a semi-continuous process was demonstrated to improve OD process with less process time and is suitable for delicate food samples such as fruits and vegetables.

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