OPTIMIZATION OF OSMOTIC DEHYDRATION OF CARROT SLICES UNDER PULSED VACUUM CONDITION IN TERNARY SOLUTION BY RESPONSE SURFACE METHODOLOGY

A THESIS

BY

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Student No: 1405199

Session: 2014-15

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MASTER OF SCIENCE (MS)

IN

FOOD ENGINEERING AND TECHNOLOGY

DEPARTMENT OF FOOD ENGINEERING AND TECHNOLOGY

HAJEE MOHAMMAD DANESH SCIENCE AND TECHNOLOGY UNIVERSITY, DINAJPUR-5200

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DECEMBER, 2016

DEDICATED

TO MY

BELOVED PARENTS

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The Author

CONTENTS

LIST OF TABLES

LIST OF FIGURES

ABSTRACT

The osmotic dehydration (OD) is a treatment in which large portion moisture is removed to obtain minimally processed product. The use of reduced pressure at the beginning of the process is called pulsed vacuum osmotic dehydration (PVOD). In this present work, the influence of independent variables (temperature, salt, and sucrose concentration, and restoration time) on water loss (WL), solute gain (SG) and color change (CC) of carrot slices was studied by response surface methodology. Analysis of results showed that restoration time was the most significant variables ($p<0.05$) for WL and SG while temperature significantly influenced the Change of color parameter. The optimum operating conditions were found to be restoration time of 182.50 min, solution temperature of 40.55°C, and solution concentration of 46.88^{o}Brix sucrose with 12.50% sodium chloride. Under this operating condition WL, SG and CC were 52.3964% (wb), 11.68% (wb) and 5.98, respectively. The predicted values for independent variables were validated by performing several experiments and the simulated data were found similar to the experimental ones.

CHAPTER I

INTRODUCTION

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INTRODUCTION

In Bangladesh, more than 30-40% of fresh fruits and vegetables are spoiled due to lack of proper and timely processing. In addition, the main cause of spoilage of fresh fruits and vegetables are their high moisture content which is more than 80% in nature (Karim & Hawlader 2005; Orsat et al. 2006). For this reason, to increase shelf life and promote food security over 20% of the world perishable crops are dried (Grabowski et al. 2003). Preservation of fruits and vegetables is usually accomplished by convective drying (Nijhuis et al. 1998). Furthermore, energy consumption and quality of dried products are the two important critical parameters for selection any drying process since drying process consumes about 20-25% of the energy during food processing (Kumar et al. 2014). However, osmotic dehydration reduces 20-30% of energy consumption during final drying and improve the quality of dried products by lowering thermal damage (Lenart 1996).

Out of the various methods of preserving perishable crops, osmotic dehydration (OD) is one of the simple and inexpensive processes that gain a lot of attention as a pretreatment prior to drying by other methods. This is because it requires very little energy and low capital investment (Sagar & Kumar 2010). Osmotic dehydration is a process in which water is partially removed by soaking foods, mostly fruits, and vegetables, in hypertonic solutions. The diffusion of water from plant tissue to solution is due to osmotic pressures of the hypertonic solution and plant tissue which is actually take place through a semipermeable membrane (Shi & Maguer 2002). The pulsed vacuum osmotic dehydration (PVOD) is an osmotic dehydration process in which vacuum pulse is applied for a short period of time at the beginning of the process, followed by an OD at atmospheric pressure (Consuegra & Corte 1997; Fante et al. 2011). Moreover, this process principally leads to an outflow of internal gas or liquid from the tissue and promotes the entrance of external solution through a hydrodynamic mechanism (HDM), which accelerates water loss and the uptake of external solutes (An et al. 2013). A number of researchers conducted research on optimization of osmotic dehydration of carrots (Singh et al. 2008; Rastogi & Raghavarao 1997). However, limited efforts have so far been made to study optimization of pulsed vacuum osmotic dehydration (PVOD) of carrots.

Optimization is defined as a technique through which best alternative solution obtained from a specified set of alternatives (Evans 1982). Response surface methodology (RSM) is an important statistical tool by which optimization techniques can be applied successfully. Furthermore, RSM enables the reseacher to determine the relationship between the response and the independent variables. In addition to, RSM is basically employed for mapping a response surface over a specified region of interest, optimizing the response or selecting operating conditions to achieve target specifications (Myers and Montgomery 2002). Therefore, during the last decades, RSM has been widely applied in the optimization of different processes in food applications.

From the above-mentioned literature and to fulfill the research work the following objectives are considered:

1) to investigate the effect of mass transfer and color change parameters during pulsed vacuum osmotic dehydration (PVOD) of carrots and

2) to optimize the PVOD process of carrots by using combinations of sucrose and sodium chloride by response surface methodology (RSM).

CHAPTER II

REVIEW OF LITERATURE

CHAPTER II

REVIEW OF LITERATURE

The aim of this chapter is to collect the available scientific literature related to osmotic dehydration of carrots as well as preparation of solution, effect of temperature, time and concentration. Moreover, pulsed vacuum osmotic dehydration (PVOD) of carrots and literature related to other authors on optimization were also reviewed in this chapter. Notably, before the commencement of this research, there was limited information in the scientific literature on the optimization of PVOD of carrot slices was found.

2.1 Osmotic dehydration

Eren & Kaymak-Ertek (2006) stated that osmotic dehydration, due to its energy and quality related advantages, is gaining popularity as a complementary processing step in the chain of integrated food processing. Osmotic dehydration is based on the principle that when cellular materials (such as fruits and vegetables) are immersed in a hypertonic aqueous solution, a driving force for water removal sets up because of the higher osmotic pressure (or lower water activity) of the hypertonic solution. Since the membrane responsible from osmotic transport is not perfectly selective, solutes from the solution diffuse into the product, as well. Therefore, osmotic dehydration can be defined as simultaneous counter-current mass transfer process. In addition, a leaching of the products own solutes (sugar, organic acids, minerals, vitamins, etc.) also occurs, which is quantitatively negligible compared with the first two transfers, yet essential regarding the final products composition.

Sethi *et al.* (1999) recommended that osmotic dehydration (OD) is a useful technique for the production of safe, stable, nutritious, tasty, economical and concentrated food obtained by placing the solid food, whole or in sliced in sugar or salt aqueous solutions of high osmotic pressure. The principle underlying osmotic dehydration is that water diffuses from dilute solution (Hypotonic solution) to concentrated solution (Hypertonic solution) through a semi-permeable membrane till equilibrium is established. The driving force for water removal is the concentration gradient between the solution and the intracellular fluid. If the membrane is perfectly semi-permeable, solute is unable to diffuse through the membrane into the cells. However, it is difficult to obtain a perfect semi-permeable membrane in food systems due to their complex internal structure and there is always some solid diffusion into the food which means that osmotic dehydration is actually a combination of simultaneous water and solute diffusion process.

Eroglu *et al.* (2010) reported that osmotic dehydration is an operation used for partial removal of water from foods such as fruits and vegetables. In this process, foods are placed in hypertonic (osmotic) solution. Three different mass transfer mechanisms occur in the osmotic dehydration; (i) water migration from food to the solution, (ii) solute migration from solution to food, and (iii) solute concerning product extracted to solution. Osmotically dehydrated fruits and vegetables are generally dried by hot air flow.

Fernandes *et al.*, (2009) descried that osmotic dehydration is a common pre-treatment used before air-drying. The technique consists of immersing the fruit in a hypertonic solution to remove part of the water from the fruit. The driving force for water removal is the difference in osmotic pressure between the fruit and the hypertonic solution. The complex cellular structure of the fruit acts as a semi-permeable membrane creating an extra resistance for diffusion of water within the fruit.

The osmotic dehydration (OD) is a process that partially removes water from food material by means of food immersion in a hypertonic solution (i.e. sugar and salt). Generally, OD is a slow process depending on the permeability of cell membranes and cell architecture (Anami *et al.,* 2007).

According to Azoubel and Murr (2004) two major simultaneous counter-current flows occur during osmotic dehydration; Water flows out of the food into the solution and a simultaneous transfer of solute from the solution into the food .There is also a third flow of natural solutes such as sugars, organic acids, mineral, salts, leaking from the food into the solution. All these mass exchanges may have an effect on the organoleptic and nutritional quality of the dehydrated product.

Madamba (2003) reported that osmotic dehydration is a water removal process involving soaking foods, mostly fruits and vegetables, in a hypertonic solution such as concentrated sugar syrup. Two major simultaneous counter-current flows occur during osmotic dehydration and important water flow out of the food into the solution and a simultaneous transfer of solute from the solution into the food.

Osmotic dehydration is one of the energy efficient means of removing moisture from a food product, as the water doesn’t have to go through a phase change to be released from the product. It is stated that some of the advantages of direct osmosis in comparison with other drying processes include minimized heat damage to color and flavor, and less decolonization of fruit by enzymatic oxidative browning (Krokida and Kouris 2003).

Osmotic dehydration (OD) is a preservation technique that may be used to decrease water activity (aw), as a pre-treatment for further processing, or applied to modify the product sensorial characteristics and increase product diversity. Dehydration techniques, including OD, induce significant changes in the dehydrated material such as volume reduction, membrane alteration or lysis, membrane separation from the cell wall or cell wall deformation among others (Alzamora *et al.*, 2000).

Torreggiani (1993) discussed that and ideal osmotic dehydration situation would be for a semi-permeable membrane to be permeated by the molecules of the solvent but not by those of the solute. Osmotic dehydration is mainly carried out on fruits and vegetables and these products that have cell wall membranes which are alive, and can stretch and expand when the product grows or generates pressure from within. The cellular membranes will freely allow molecules of the solvent to pass through them, but they also allow a small amount of the solute molecules to pass through; this is shown in Figure 2.1. This type of membrane should be classified as permeable, as opposed to semi-permeable.

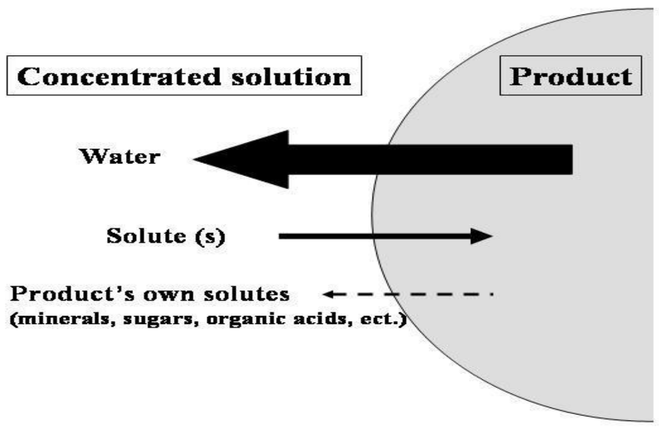


Figure 2.1: Mass transfers during the soaking process (Raoult-Wack, 1994).

2.2 Factors affecting osmotic dehydration

Osmotic dehydration of red paprika was studied using a combined sucrose and NaCl solution. It was found that the optimum conditions for sucrose concentration and NaCl concentration are 21.86g/10g and 2.02 g/10g, respectively for the appropriate criteria are achieved (Ade-Omowaye *et al.*, 2002).

Ternary sucrose and NaCl solution, multi-components salt sugar aqueous solutions have been studied to increase the driving force of the process. Mixture of salt and sucrose in different proportion can be used for materials of plant and animal origin to obtain higher weight loss to solid gain ratios (*WL*: *SL*) than with individual solutes in binary solution; this also reduces impregnation (Sacchetti *et al.*, 2001; Ade-Omowaye *et al.*, 2002).

Sucrose and NaCl solutions proved to be the best choices based on effectiveness, convenience and flavor. For apple sticks dehydrated using ternary sucrose and NaCl solutions, the addition of NaCl may help to attenuate the excessive sweetness of product processed with high sucrose concentration. It was also found that addition of NaCl at levels up to 1% did not have a detrimental effect on product acceptability when added to sucrose solutions having concentrations lower than 55% (Sacchetti *et al.*, 2001).

Osmotic dehydration of apples cut into a cylinder shape were carried out in binary aqueous solution of sucrose (40-50%) and NaCl (15-26.5%) with different concentrations and temperatures, as well as in ternary solutions of 30/10, 40/10, 50/10, 20/15, 30/15, 40/15 % of sucrose and NaCl, respectively. The ratio of water loss to solids gain (WL/SG) for each osmotic treatment was particularly high in the case of salt solutions, due to a low solids gain. In the case of ternary mixed solutions, intermediate values for WL/SG are obtained (Sereno *et al.*, 2001).

Increase in osmotic solution concentration resulted in corresponding increases in water loss to equilibrium level and drying rate (Conway *et al.*, 1983; Hawkes and Flink, 1978; Lenart, 1992). Therefore, increased osmotic solution concentrations lead to increased weight reductions. This was attributed to the water activity of the osmotic solution which decreases with the increase in solute concentration in the osmotic solution (Biswal and Le Maguer, 1989; Biswal *et al.,* 1991; Rahman and Lamb, 1990). According to Ravindran (1987) an increase in 10°Brix corresponds to an increase of 5% of the final water loss percentasge. Lazarides (1994) studied on the osmotic dehydration of apples using 45 and 65°Brix of sucrose solution. It was found that a higher sucrose concentration (65°Brix) a faster water loss (ca.30% increase). However, there was a much greater solid uptake (80% increase). He concluded that under increased osmotic solution concentration favored solid uptake and resulted in lower water loss to solids gain ratio. On the contrary, low concentration sucrose solution can cause minimal water loss which resulted in lower water loss to solid gain ratio (Karathanos *et al.*, 1995).

The specific effect of the osmotic solution is of great importance when choosing the solution. The solute cost, organoleptic compatibility with the end product and additional preservation action by the solute are factors considered in selecting osmotic agents (Torreggiani, 1995). Several solutes, alone or in combinations, have been used in hypertonic solutions for osmotic dehydration (Le Maguer, 1988).

Ponting *et al.* (1966) and Flink (1979) reported that an increase of osmotic solution to sample mass ratio resulted in an increase in both the solid gain and water loss in osmotic dehydration. To avoid significant dilution of the medium and subsequent decrease in the (osmotic) driving force during the process, a high ratio (at least 30:1) was used by most workers whereas some investigators used a much lower solution to product ratio (4:1 or 3:1) in order to monitor mass transfer by following the changes of the sugar solution concentration (Conway *et al.*, 1983).

2.3 Applications of osmotic dehydration process

Osmotic Dehydration is mainly related to the improvement of nutritional, organoleptic and functional properties of the product. As OD is effective at ambient temperatures, heat damage to color and flavor is minimized and high concentration of the sugar surrounding fruit and vegetable pieces prevent discoloration. Furthermore, through the selective enrichment in soluble solids and reduced acidity with little or no use of SO2, high quality fruits and vegetables are obtained with functional properties compatible with different food systems. These effects are obtained with a reduced energy input over traditional drying process (Torreggiani, 1993). The product thus obtained is an intermediate moisture food (IMF) having moisture content ranging from 65-75% and water activity (aw) in the range of 0.94-0.97 (Le Maguer, 1988). The commercial applications of OD process for tropical fruits such as banana, mango, apple, papaya sapota and strawberry has been critically reported by Bongirwar and Sreenivasan (1977).

Recently, Sharma (1996) has reported that osmo-vacuum dried apple rings after reconstitution in 15^0 Brix sugar syrup were better than canned products. Osmotic Dehydration can also be used instead of air drying in obtaining dehyro-frozen foods. The use of OD for partial concentration of fruits and vegetables reduces the moisture content of the material thereby reducing the refrigeration load during freezing, saving packaging and distribution costs and achieving higher product quality because of the marked reduction in structural collapse and dripping loss while thawing (Huxoll, 1982; Biswal *et al.*, 1991)

2.4 Response surface methodology (RSM)

According to Eren *et al.* (2007), Response surface methodology (RSM) is a statistical procedure frequently used for optimization studies. It uses quantitative data from an appropriate experimental design to determine and simultaneously solve multivariable problems. Equations describe the effect of test variables on responses, determine interrelationships among test variables and represent the combined effect of all test variables in any response. This approach enables an experimenter to make efficient exploration of a process or system.

Response surface methodology (RSM) is a useful and well known statistical tool applied in process optimization. Experimental condition that optimizes a process response as a specific characteristic of quality of the final product can be obtained by RSM. However, this condition may be different for another process response. Desirability function is thus a technique that can be used to formulate and solve this conflict as a constrained optimization problem. It allows finding an optimal experimental condition that meets all the process responses established as ideal (Montgomery, 2001).

Singh *et al.* (2006) conducted researches on osmotic dehydrations of carrot cubes in sodium chloride salt solutions at different solution concentrations, temperatures and process durations which were analyzed for water loss and solute gain. The osmotically pretreated carrot cubes were further dehydrated in a cabinet dryer at 65C and were then rehydrated in water at ambient temperature for 8–10 h and analyzed for rehydration ratio, color and overall acceptability of the rehydrated product. The process was optimized for maximum water loss, rehydration ratio and overall acceptability of rehydrated product, and for minimum solute gain and shrinkage of rehydrated product by response surface methodology. The optimum conditions of various process parameters were 11% salt concentration, 30^{o}C osmotic solution temperature and process duration of 120 min.

Ding *et al.* (2013) observed that Response surface methodology (RSM) of Box–Behnken design with 27 experimental runs and the desirability function method were used in the osmotic dehydration process of Chinese ginger (Zingiber officinale Roscoe) slices in ternary solution of water, sucrose and sodium chloride for maximizing water loss (WL), rehydration ratio (RR) and total phenolic content (TPC) and minimizing solute gain (SG) and hunter color change (HCC) of dehydrated product. The results indicated that the optimum operating conditions were found to be process duration of 102 min, solution temperature of 30 °C, solution concentration of 50 Brix sucrose + 7.31% sodium chloride and solution to food ratio of 8:1 (w/w). Under this condition, the WL, SG and TPC were 58.8% (wb), 12.56% (wb) and 1.46% (db), while its RR and HCC were 1.59 and 6.55, respectively. The immersion time was the most significant variable for WL, HCC, SG and RR, and for TPC it was temperature ($P < 0.05$).

Response surface methodology was used by Vieira *et al.* (2012) to assess the effects of osmotic solution concentration (40–60^{o}Brix), process temperature (20–40^{o}C) and vacuum pulse application time (0–20 min) at 100 mbar on water loss (WL), weight reduction (WR), solid gain (SG), water activity (aw), color parameters and mechanical properties of guava slices. Optimal process conditions were determined through the desirability function approach and quality characteristics of osmotically dehydrated guavas were analyzed. Only models obtained for WL, WR and aw were suitable to describe the experimental data. The desirability function showed that optimal conditions for osmotic dehydration of guavas were: osmotic solution concentration at 60_Brix, process temperature at 32^{o}C and 20 min of vacuum pulse application. Under optimal conditions, color and mechanical properties of treated guavas were similar to fresh fruit, presenting WL of 29.01 g ⁄ 100 g, WR of 25.91 g ⁄ 100 g, SG of 3.10 g ⁄ 100 g and a_w of 0.979.

Button mushrooms (Agaricus bisporous) were dried in a microwave vacuum dryer up to a final moisture content of around 6% (d.b.). The effect of microwave power level (115 to 285 W), system pressure (6.5 to 23.5 KPa), and slice thickness (6 to 14 mm) on drying efficiency and some quality attributes (color, texture, rehydration ratio, and sensory attributes) of dehydrated mushrooms were analyzed by means of response surface methodology. A rotatable central composite design was used to develop models for the responses.Analysis of variance showed that a second-order polynomial model predicted well the experimental data. The system pressure strongly affected color, hardness, rehydration ratio, and sensory attributes of dehydrated mushrooms. A lower pressure during drying resulted in better quality products. Optimum drying conditions of 202W microwave power level, 6.5 KPa pressure, and 7.7mm slice thicknesses were established for microwave vacuum drying of button mushrooms. Separate validation experiment was conducted at the derived optimum conditions to verify the predictions and adequacy of the models (Giri & Prasad, 2007).

2.5 Pulsed vacuum osmotic dehydration (PVOD)

Correa *et al.* (2016) stated that the osmotic dehydration (OD) is a treatment that reduces partially the moisture content and the water activity of a food. The use of reduced pressure in the first minutes of an OD is called pulsed vacuum osmotic dehydration (PVOD). In PVOD, the expulsion of occlude gases and the entrance of the solution in the food matrix are induced, with consequently mass transfer improvement. In the present work, the influences of independent variables (temperature (T), pressure of vacuum pulse (PV), sodium chloride concentration [NaCl] and sucrose concentration [suc]) on water loss (WL), solid gain (SG), weight reduction (WR), water activity (aw) and moisture content (U) of tomato slices were studied. The optimum condition obtained had its kinetics tested with three models from the literature. The optimized condition (T 40 °C, PV 56.25 mbar, [NaCl] 7.5 % and [suc] 32.5 %) resulted in the maximum values of WL, and WR (42.2, and 36.1 %, respectively) and minimum SG, aw and U (4.0, 0.948 and 76.5 kg water/100 kg sample, respectively). With respect to the kinetics, the best agreement was obtained with the model that considers variable diffusivity.

The effects of osmotic dehydration (OD) with or without pulsed vacuum (PV) on hot-air drying kinetics and quality attributes of cherry tomatoes were investigated by Li *et al.* (2013). Both OD and PVOD pre-treatments were performed for 3 h at 50^{o}C in 50 and 70^{o}Brix sucrose solutions with a solution-to-fruit mass ratio of 4:1, and PVOD was applied for 15 min before OD at atmospheric pressure. Samples were further dried at air temperature of 70^{o}C. Effective moisture diffusivity (D_{eff}) of osmotically dehydrated samples increased gradually while the D_{eff} curve of fresh samples had a plateau stage during hot air drying. Lower glass transition temperature, T_g, values of osmotically dehydrated samples indicated that they needed a lower storage temperature. Both OD and PVOD pre-treatments had advantages in shortening drying cycles and improving quality of products. Compared with air drying, osmo-air drying decreased the total drying time, color change, and hardness of dried samples by 32.26%, 18.11%, and 88.21%, respectively, and increased volume ratio and vitamin C retention rate by 72.31% and 125.82%. As compared with OD, PVOD decreased color change and hardness by 28.48% and 45.17%, increased volume ratio and vitamin C retention rate by 27.41% and 17.77%, but there was no significant difference shown in drying time. Therefore, osmotic pre-treatment can shorten the total dehydration time, and improve the general quality of dried cherry tomatoes.

The influence of operating pressure during osmotic dehydration on mass transfer and mechanical properties in pineapple fruits was analyzed by Ramallo et al. (2013). Dehydration trials were performed at atmospheric pressure (OD) and by applying a vacuum pulse (VPOD), in sucrose solution at 60°Brix and 40°C for 300 min. Seven operation conditions were implemented with a vacuum pulse of 100 mbar or 250 mbar for 0, 5, 15 or 25 min at the beginning of the process. The decrease of pressure favored the solute uptake, but the water loss has not been significantly affected. No significant effect of vacuum time was observed. However, solute uptake in trials with vacuum pulse of 100 mbar was significantly higher than in OD process. In general, mechanical properties and shrinkage were not affected by operation conditions. Osmotic dehydration process (both OD and VPOD) originates a more resistant tissue structure than the one in fresh pineapple fruit.

The combination of pulsed vacuum osmotic dehydration and microwave drying of garlic bulbs were examined by Hamledari et al. (2012). The response surface methodology (RSM) was used to determine the effect of NaCl concentration (8%-20%), osmotic solution temperature (25-65°C), operation pressure (240-830 mbar), immersion time (20-300 min) and microwave power level (100-600 W) on water loss (WL), solids gain (SG), weight reduction (WR), hardness and shrinkage of samples. Analysis of the results showed that by increasing the osmotic solution concentration, temperature and immersion time, WL and WR will increase. The effect on SG was almost the same as WL except the effect of temperature. Increasing temperature resulted in an initial increase in SG for a period of time, followed by a decrease. NaCl concentration, temperature and immersion time showed the significant influence on hardness.

Correa et al. (2014) stated that Pulsed vacuum osmotic dehydration (PVOD) is an efficient process for obtaining semi-dehydrated food. The effects of temperature (30–50°C), solute concentration (NaCl 0–15 kg per 100 kg solution, sucrose, 15–35 kg per 100 kg solution) and vacuum pulse application (50–150 mbar and 5–15 min) on water loss (WL), solid gain (SG), water activity (aw) and total colour difference (ΔE) of previously blanched pumpkin slices were assessed through Plackett–Burman experimental design. Temperature was not statistically significant in the process. Later, with the aid of a central composite design (CCD), it was found that concentration of sucrose and NaCl was influent on the WL, SG, aw and ΔE, and the pressure and time of application of vacuum were influent on WL and SG. The optimal conditions of process were stabilized with the desirable function, and the simulated data were similar from the experimental ones.

CHAPTER III

MATERIALS AND METHOD

CHAPTER III

MATERIALS AND METHOD

The experiment was conducted in the laboratories of the faculty of Engineering, Hajee Mohammad Danesh Science and Technology University (HSTU), Dinajpur.

3.1 Materials

Fresh carrots (*Daucus carota* L.) were collected from the local market and kept in cool storage at 4-5^{o}C. Prior to osmotic dehydration experiments, carrots were thoroughly washed to remove the dirt and graded by size to eliminate the variations in respect to exposed area. Then the carrots were cut into the thickness of 10mm slices through sharp stainless steel knife. Before commencement of the research work, the moisture content of fresh carrots was determined by drying the samples in a vacuum oven at 70^{o}C for 14-16h (AOAC, 2000). The initial moisture content of carrot slices was 85.2% (wb).

3.2 Osmotic agent preparation

Osmotic solutions were prepared by mixing sucrose and salt with the proper amount of distilled water using magnetic stirrer. Concentrations of osmotic solutions were checked by a digital refractometer (model-HI96801).

3.3 Pulsed vacuum osmotic dehydration (PVOD) procedure

The pulsed vacuum osmotic dehydration (PVOD) experiments were carried out in stainless steel cases. The temperature of the osmotic solution was measured by a thermocouple. The vacuum pressure was obtained by a vacuum pump. During PVOD process, the samples were immersed in a tertiary osmotic solution of water, salt, and sucrose. In this experiment, the fruit to solution ratio was maintained approximately 1:45 (Vieira et al. 2012). The vacuum pulse was maintained in the first 10 min (Corrêa et al. 2010; Viana et al. 2014) of each experiment at 500 mm of Hg; after that, the process was continued at atmospheric pressure. The total atmospheric restoration time was maintained 240 min (Corrêa et al. 2016; Singh et al. 2008). For each experiment, ten samples of known weight were used. After the PVOD process, the osmotic solution and carrot slices were taken in Erlenmeyer flasks which are placed in a thermostatic water bath. The Erlenmeyer flasks were covered with a sheet of aluminum film to prevent evaporation of osmotic solutions. The temperature was maintained constant by the thermostatic water bath during osmosis and agitation was given for reducing the mass transfer resistance at the surface of the carrots and for good mixing (Mavroudis et al. 1998). The agitation speed of 100 rpm was used and maintained constant (Gomes Alves et al. 2005). After specified times, the carrot slices were removed from the osmotic solutions and rinsed with tap water to stop the dehydration. Then the osmotically dehydrated samples were spread on the absorbent paper to remove free water present on the surface. Meanwhile, a proportion of carrot slices were used for determination of final moisture content using hot air drier at 70^{o}C. All experiments were performed in duplicate and the data are given an average of these results. The reproducibility of the experiments was within the range of ±2.47%.

3.3.1 Measurement of water loss and solute gain

The mass transfer parameters i.e water loss and solute gain were calculated by the equations given by El-Aouar et al. (2006):

$$WL(\%) = \frac{(w_i X_i - w_f X_f)}{w_i} \times 100 \quad \text{....................................(3.1)}$$

$$SG(\%) = \frac{(w_f(1-\frac{X_f}{100}) - w_i(1-\frac{X_i}{100})}{w_i} \times 100 \quad \text{......................(3.2)}$$

Where w_i and w_f are initial and final (time,t) samples weights (g) respectively; X_f and X_i are the final (time,t) and initial moisture content, respectively, (g/100g initial wet of carrot slices).

3.4 Color change

The color of fresh and osmotically dehydrated sample was measured by using Miniscan XE plus Hunter Lab Colorimeter (USA), Model 45/0-L. The color of fresh and dehydrated carrot slices was assessed in terms of 'L', 'a', and 'b' after making a paste of the sample. Before measuring the color of the sample, the colorimeter was calibrated using white plates provided. For determining the color of the dehydrated sample, the paste was completely filled in Petri dish hence no light can pass during the measuring process. The obtained values were recorded and compared with the values of fresh carrot sample. The color change (ΔE) was calculated by the equation given by (Alam et al. 2010)

$$CC = \sqrt{[(L-L_0)^2 + (a-a_0)^2 + (b-b_0)^2]} \quad \text{............(3.3)}$$

Where L_0, a_0, and b_0 represent the readings of fresh carrot sample, respectively.

3.5 Experimental design

The variables chosen for PVOD experiments were temperature (A), sucrose concentration (B), salt concentration (C), and restoration time (D). The variable levels were selected on the basis of previous studies described by several authors (Jokić et al. 2007; Singh et al. 2007; Telis et al. 2004). Thirty experiments were conducted according to a central composite rotatable design (CCRD) with five levels of each variable. The levels of the variable in coded form and actual units are given in Table 3.1.

Table 3.1: Process variables and their five levels of experimental design

Variable	Name	Units	Levels				
			-2	-1	0	1	2
A	Temperature	^{o}C	35	40	45	50	55
B	Sucrose concentration	^{o}Brix/%	40	42.5	45	47.5	50
C	Salt concentration	%	5	7.5	10	12.5	15
D	Restoration time	min	10	67.5	125.0	182.5	240

The experimental design of process variables in uncoded forms and values of various responses are shown in Table 3.2

Table 3.2 Central composite rotatable design (CCRD) with experimental values of response variables

Uncoded process variables					Responses		
Run	**Temperature (^oC)**	**Sucrose conc. (%)**	**Salt conc (%)**	**Time (min)**	**Water loss (%)**	**Solute gain (%)**	**Color change**
1	40.0	42.5	7.50	182.5	45.59	10.99	8.11
2	45.0	40.0	10.0	125.0	41.88	9.20	8.70
3	45.0	45.0	10.0	240.0	46.6	12.24	5.40
4	45.0	45.0	10.0	125.0	45.15	10.91	4.09
5	45.0	45.0	10.0	10.0	29.82	5.10	3.97
6	50.0	47.5	12.5	67.5	43.82	10.94	5.44
7	50.0	42.5	7.50	182.5	41.92	11.79	7.99
8	50.0	47.5	7.50	67.5	41.28	11.52	5.95
9	45.0	45.0	10.0	125.0	43.0	12.13	6.02
10	45.0	45.0	15.0	125.0	46.31	10.40	5.14
11	40.0	42.5	12.5	182.5	47.02	11.96	7.97
12	40.0	42.5	12.5	67.5	31.03	10.58	8.90
13	50.0	47.5	12.5	182.5	43.49	16.21	6.52
14	50.0	42.5	12.5	182.5	41.81	11.98	5.80
15	40.0	47.5	7.50	182.5	51.56	11.37	6.43
16	50.0	47.5	7.50	182.5	45.70	16.10	7.18
17	45.0	50.0	10.0	125.0	49.57	11.59	8.44
18	40.0	42.5	7.50	67.5	31.40	8.10	4.58
19	45.0	45.0	10.0	125.0	46.91	12.27	5.05
20	40.0	47.5	12.5	67.5	39.75	6.95	7.44
21	45.0	45.0	5.00	125.0	41.89	10.78	5.88
22	55.0	45.0	10.0	125.0	47.28	12.05	5.58
23	40.0	47.5	7.50	67.5	36.26	8.05	6.36
24	50.0	42.5	7.50	67.5	41.28	8.25	4.60
25	45.0	45.0	10.0	125.0	46.34	12.95	5.15
26	45.0	45.0	10.0	125.0	46.04	11.62	5.27
27	35.0	45.0	10.0	125.0	50.64	9.96	9.57
28	40.0	47.5	12.5	182.5	51.41	12.02	6.14
29	45.0	45.0	10.0	125.0	45.68	12.83	5.30
30	50.0	42.5	12.5	67.5	41.54	12.51	6.34

The center points in the CCRD design was repeated six times to estimate the reproducibility of the method. Response surface methodology was used to determine the relative contributions of A, B,C and D to various responses under study such as water loss (WL), Solute gain (SG), and color change (ΔE) of osmotically dehydrated carrot slices. The second-order polynomial

response surface model (Eq. (3.4)) was fitted to each of the response variables (Y_k).

$$Y_k = b_{k0} + \sum_{5}^{5} b_{ki} + \sum_{i=1}^{5} b_{kii} X_i^2 + \sum_{i \neq j=1}^{5} b_{kij} X_i X_j \text{ ....(3.4)}$$

Where b_{k0}, b_{ki}, b_{kii}, and b_{kij} expressed are the constant, linear, quadratic, and cross product regression coefficients, respectively, and X_is are the coded independent variables of A, B, C, and D.

3.6 Analysis of data

Response surface analysis of the experimental data was carried out using a commercial statistical package Design Expert, version 7.0 (Stat Ease Inc., Minneapolis, MN). Regression analysis and analysis of variance (ANOVA) were calculated for fitting the model represented in Eq (3.4) to the experimental data and to examine the statistical significance of each response. The model adequacies were checked by using model analysis, lack-of -fit test, and co-efficient of determination (R^2) analysis as outlined by various researchers (Lee et al. 2000; Weng et al. 2001). The co-efficient of determination (R^2) is a measure of the degree of fit and defined as the ratio of the explained variation to the total variation. Co-efficient of variance (CV) is the relative dispersion of the experimental points from the model prediction. Response surfaces were also generated by the Design Expert software.

3.7 Numerical optimization technique

Design Expert software was used for numerical optimization of the multiple responses. For optimization, some of the variables are to be maximised and some are to be minimized. Moreover, the independent factors are kept within the expermental domain. For finding a solution, the goals were combined into an overall composite function called the desirability function (Giri & Prasad 2007), which is defined as:

$$D(x) = (d_1 \times d_2 \times \text{.......................} \times d_n)^{1/n} \text{...(3.5)}$$

where d_1, d_2 are responses and n expressed as the total number of responses in the measure.

The function D (x) considers as the desirable ranges for each response (D_i), Desirability is an objective function that ranges from zero to one. The maximum point for desirability function considers as optimize point. The goal-seeking starts at a random starting point and keep forward the steepest slope to a maximum point. Because of curvature nature in the response surfaces there may be two or more maximums and their combination into the desirability function. For validating the obtained predicted values, several experiments were conducted using the conditions established by the optimisation. Moreover, the predicted values obtained by the models were evaluated to the experimentally obtained values.

CHAPTER IV

RESULTS AND DISCUSSION

CHAPTER IV

RESULTS AND DISCUSSION

The present study was conducted to evaluate the mass transfer and color change parameters during pulsed vacuum osmotic dehydration (PVOD) of carrot slices and to optimize PVOD process by using combination sucrose and salt. The data was analyzed by using multiple regression techniques to develop a response surface model. Moreover, the results of the experiments were handled based on the experimental design and regression coefficient in terms of the codified units and different models were tested for their adequacy using ANOVA technique.

Full second order model of the form was fitted to data and the regression coefficient was calculated, the results of which are given in Table 4.1. The sign and magnitude of coefficient indicate the effect of independent variables and their interaction on the various responses. The response surface generated from the various fitted models actually reflect the influence of two independent variables on water loss (WL), solute gain (SG) and color change (ΔE) of PVOD carrot slices whereas the third variable was fixed at the center point.

4.1 Effect of process variables and vacuum pulse on water loss (WL)

The coefficients of WL regression model with coded variables are listed in Table 4.1. In addition to, its coefficient of determination (R^2) was 0.94, the coefficient of variation (CV) was 4.39% and the lack of fit was non-significant at 95% level of confidence, which all implied that the model was adequate for predicting water loss (WL). The linear, interaction and quadratic terms of corresponding variables would be more significant if the F-value was greater with a lower p-value (Atkinson 2011). Thus, the most significant variable for WL was restoration time followed by sucrose concentration, salt concentration, and temperature. It implies that increased water loss with the increase of restoration time consequently sucrose solution concentrations also. This phenomenon supports with many early reports (Azoubel & Murr 2004; Conway et al. 1983; An et al. 2013). Therefore, the regression equation for describing the effects of process variables on water loss in terms of coded values of variable is given as-

$$WL=45.52+4.167E\text{-}003A+1.96B+0.57C+3.99D-1.01AB-0.24AC-3.26AD+0.15BC-2.500E\text{-}003BD-0.43CD+0.52A^2-0.29B^2-0.70C^2-2.17D^2 \quad \text{..............(4.1)}$$

From Table 4.1 it is observed that the interaction term 'sucrose concentration and salt concentration' have a positive effect on water loss while others have a negative effect.

Additionally, the interaction terms of 'temperature and time' has a significant effect on water loss at 5% level of significance. The quadratic terms of all process parameters have a negative effect on water loss except temperature. It indicates that an increase of that variables significantly decreased in water loss.

To visualize the combined effects of the two factors on the response, the response surface and contour plots were generated for the fitted model in function of two variables, while other two variables keeping at the central point. From Fig 4.1 it is observed that temperature and restoration time has a significant effect on water loss. It explains that water loss increases when restoration time increases even though temperature of the solutions decreased from 50 to 40^{o}C. Moreover, water loss was highly influenced by restoration time due to vacuum pulse application at the beginning of the process. It is due to the hydrodynamic mechanism which open pores for an external liquid phase thus enhancing water transfer rate. Similar trends were also reported by several researcher during pulsed vacuum and under atmospheric condition as well (Fito 1994; Fito & Pastor 1994; Moreno et al. 2000).

Table 4.1 Regression summary and ANOVA for water loss, solute gain and color change

Source	df	Water loss				Solid gain				Color Change			
		β	Sum of square	F- value	P level	β	Sum of square	F- value	P level	β	Sum of square	F- value	P level
Model	14	45.52	827.87	16.32	0.0001[a]	12.12	134.68	7.78	0.0002[a]	5.15	54.87	6.42	0.0005[a]
Temp (A)	1	4.2E-03	4.167E-004	1.15E-04	0.9916	0.98	22.93	18.54	0.0006[a]	-0.59	8.27	13.55	0.0022[a]
Sucrose Conc (B)	1	1.96	92.28	25.47	0.0001[a]	0.49	5.78	4.68	0.0472[a]	-0.14	0.47	0.77	0.3952
Salt Conc (C)	1	0.57	7.84	2.16	0.1619	0.26	1.61	1.30	0.2715	0.078	0.15	0.24	0.6322
Time (D)	1	3.99	381.60	105.33	0.0001[a]	1.66	66.0	53.37	0.0001[a]	0.39	3.67	6.02	0.0269[a]
AB	1	-1.01	16.40	4.53	0.0504	0.84	11.36	9.18	0.0084[a]	0.22	0.79	1.29	0.2738
AC	1	-0.24	0.96	0.27	0.6142	0.061	0.060	0.049	0.8286	-0.41	2.71	4.45	0.0522
AD	1	-3.26	169.91	46.9	0.0001[a]	0.013	2.5E-03	2.02E-03	0.9647	0.24	0.90	1.47	0.2439
BC	1	0.15	0.38	0.1	0.7511	-0.55	4.86	3.93	0.0660	-0.26	1.06	1.73	0.2082
BD	1	-2.5E-03	1.0E-004	2.76E-05	0.9959	0.69	7.51	6.07	0.0063[a]	-0.27	1.19	1.96	0.1823
CD	1	-0.43	3.03	0.84	0.3751	-0.2	0.62	0.5	0.4911	-0.62	6.14	10.06	0.0063[a]
A^2	1	0.52	7.27	2.01	0.1769	-0.1	0.30	0.24	0.6295	0.61	10.24	16.79	0.0010[a]
B^2	1	-0.29	2.37	0.65	0.4316	-0.26	1.81	1.47	0.2447	0.86	20.28	33.23	0.0001[a]
C^2	1	-0.7	13.44	3.71	0.0733	-0.21	1.19	0.96	0.3421	0.095	0.25	0.40	0.5343
D^2	1	-2.17	129.46	35.73	0.0001[a]	-0.69	13.0	10.51	0.0055[a]	-0.11	0.34	0.56	0.4669
Lack of fit	10		44.96	2.39	**0.1737**[b]		15.62	2.67	**0.1453**[b]		7.23	1.87	**0.2529**[b]
R^2		**0.94**					**0.88**				**0.86**		
C.V. %		**4.39**					**10.01**				**12.38**		

[a] Significant at 5% ($p<0.05$) level of significance

[b] Non-significant at 5% ($p>0.05$) level of significance

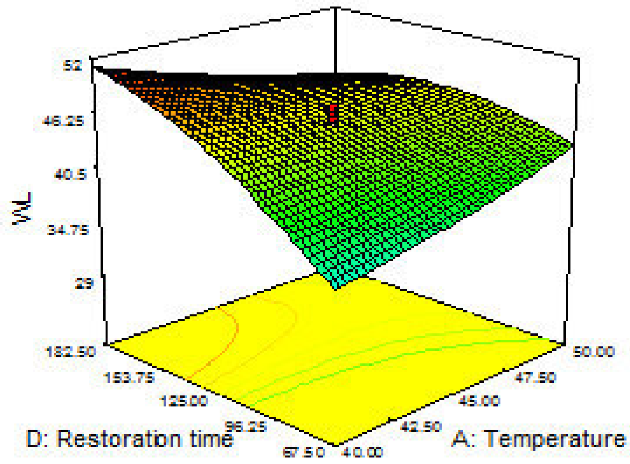


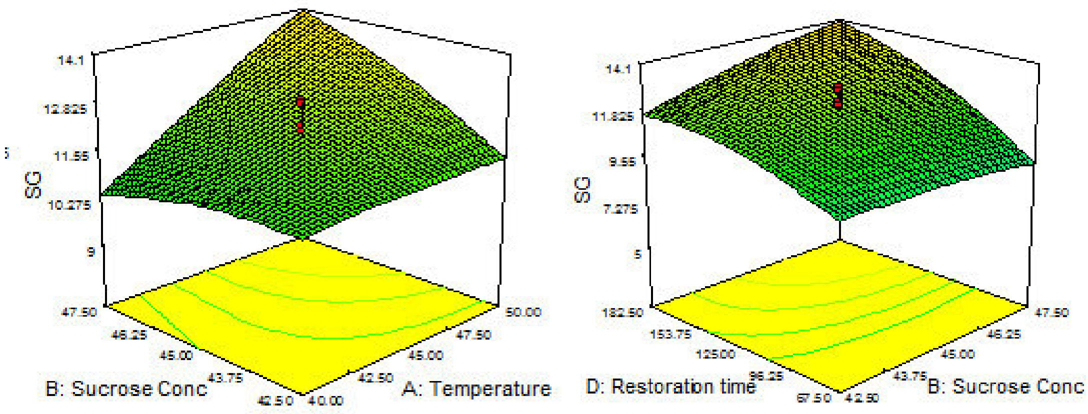
Fig 4.1: Response surface and contour plots for water loss (WL)

4.2 Effect of process variables and vacuum pulse on solute gain (SG)

The coefficient of SG regression model is listed in Table 4.1. The coefficient of determination R^2 was 0.88, the CV was 10.01% and the lack of fit was non-significant ($p>0.05$), which all indicated that the model was adequate for predicting solute gain (SG).The F-values, β values and P values in Table 4.1 indicates that the most significant variable for SG was restoration time, followed by temperature, sucrose concentration, but the effect of salt (β=0.26) was negligible on the solute gain. These results reveal that an increase in solute gain with an increase in process duration and solution temperature. The pronounced effects of linear terms such as time and solution temperature on SG was in accordance with the results of (Singh et al. 2010; An et al. 2013; Singh et al. 2010). Thus, the mathematical expression of relationship to the response with coded variables of solid gain is given in below-

$$SG = 12.12+0.9A+0.49B+0.26C+1.66D+0.84AB+0.061AC+0.013AD-0.55BC+0.69BD-0.20CD-0.10A^2-0.26B^2-0.21C^2-0.69D^2 \quad (4.2)$$

It is noted that the interaction terms 'sucrose concentration and salt concentration' and 'salt concentration and restoration time' have a negative effect on SG whereas all others have the positive effect on the solute gain (Table 4.1). Moreover, the interaction terms 'temperature and sucrose concentration' and 'sucrose concentration and time' has notable effect on the solute gain at 5% level of significance. It indicates that an increase in of that variables results in an increase in solute gain. However, all quadratic terms have a negative effect on solute gain. Apart from this, the quadratic term of restoration time has a significant effect on SG ($p<0.05$). This is due to the application of vacuum pulse at the begining of the process which exchanged gas occluded in the tissue structure hence solute gain was increased as the progress of time. The response surface and contour plots [Figure 4.2(a)-(b)] are generated for the fitted model to visualize the combined effect of different variables on the solute gain. Figure 4.2 (a) indicates that as the sucrose concentration and osmotic solution temperature increase solute gain also increased. From Table 4.1 it also points out that the osmotic solution temperature has the most pronounced effect on the solute gain in comparison to solution concentration. Campos et al. (2012) described that temperature is the most influential parameter in the osmotic dehydration. A similar effect was also reported by Singh et al. (2007) for carrot cubes in sucrose-salt solution. The possible explanation for this behavior is the collapse of the cell membrane at higher temperatures as a result higher rate of osmosis took place resulting better mass transfer rate (Kalse et al. 2012). It is observed from Fig 4.2 (b) that SG highly influenced by the increase in time and sucrose concentration. This phenomenon notably observed when sucrose concentration and time rises above 45^{o}Brix and 150 min, respectively.Similar results were reported by several researchers (Jokić et al. 2007; Alam et al. 2010).



(a) (b)

Fig 4.2 (a-b): Response surface and contour plots for solute gain (SG)

4.3 Effect of process variables and vacuum pulse on color change

The coefficient of CC regression model is given in Table 4.1. The lack of fit was non-significant at 5% level of significance, the coefficient of determination (R^2) was 0.86, the CV was 12.38%, which all revealed that the model was adequate for predicting color change. The P and F-values indicated that the most significant variable for the color changes of carrot slices was temperature, followed by restoration time, sucrose concentration and salt concentration. This result describes that as the temperature of the osmotic solution increased color of carrot slices changes significantly. This is because carrots are root crop contains a high content of carotenes dominated by α and β-carotene which may oxidize with the increment of temperature resulting bleaching of carotene which leads to color change (Kidmose et al. 2002). This result supports the findings of other researcher reported by Lee & Lim (2011) for pumpkin slice and Alam et al. (2010) for aonla slice. Thus, after solving second order polynomial equations the final equation for color change is given in below-

$$CC = 5.15 - 0.59A - 0.14B + 0.078C + 0.39D + 0.22AB - 0.41AC + 0.24AD - 0.26BC - 0.27BD - 0.62CD + 0.61A^2 + 0.86B^2 + 0.095C^2 - 0.11D^2 \quad (4.3)$$

The interaction term 'salt concentration and restoration time' has the positive and significant effect ($p<0.05$) on color change even though others have the negative effect (Table 4.1). The β values in Table 4.1 show that the quadratic terms of temperature, sucrose concentration and salt concentration have the positive and significant effect on color change except for restoration time. The response surface and contour plot (Fig 4.3) are generated by combining the effect of various independent variables on the color change. Figure 4.3 describes that color change showed a significant increase with the increase in salt concentration and time. This is due to the presence of salt/sodium chloride in osmotic solution which may affect the structure of cellular membrane results in changes in physical properties (Sereno et al. 2001).

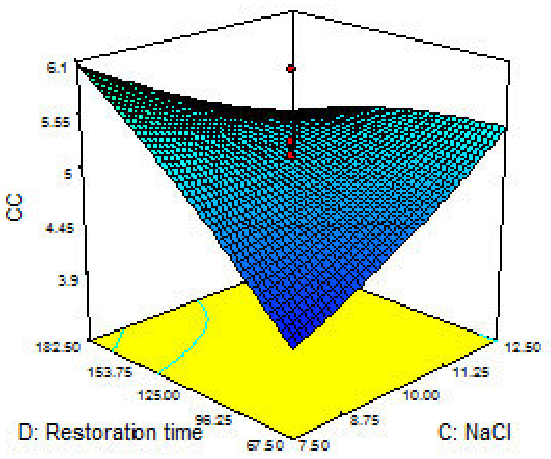


Fig 4.3: Response surface and contour plots for color change (CC)

4.4 Optimization of PVOD for carrot slices and Experimental validation

To determine the workable optimum conditions a graphical multi-response optimization technique was adopted for pulsed vacuum osmotic dehydration of carrot slices. So, the optimization of the osmotic process for carrot slices using the desirability function was performed, considering the process condition that results on minimum SG and CC and maximum WL. The Design Expert program was run for the optimum conditions and the solutions obtained are presented in Table 4.2. The table indicates the optimum conditions of various independent variables and also predicted values of various responses (WL,SG, and CC). The solution having maximum desirability value was selected as the optimum conditions for PVOD of carrot slices.

Table 4.2: Solution for optimum conditions

Sol. No.	Temp. (°C)	Sucrose Conc. °Brix	NaCl (%)	Restoration Time (min)	WL (%)	SG(%)	CC (ΔE)	Desirability
1.	**40.55**	**46.88**	**12.50**	**182.50**	**52.3964**	**11.68**	**5.98**	**0.715**
2.	40.70	47.04	12.50	182.50	52.3913	11.71	5.95	0.715
3.	40.54	46.85	12.50	182.50	52.36	11.69	5.97	0.715
4.	40.60	46.91	12.50	182.50	52.37	11.70	5.96	0.715

Osmotic dehydration experiments of carrot slices were performed for validating predicted data. Moreover, the experimental values of various responses were determined by using the predicted optimum values of independent variables. The experimental values (mean of three measurements), as well as the predicted values of various responses, experimental values shows the lower standard error of mean which is presented in Table 4.3.

Table 4.3: Comparison of experimental values with predicted values

Response	Predicted value	Experimental value ±SE
Water loss (%)	52.3964	51.19±0.69
Solute gain (%)	11.68	10.83±0.42
Color change (ΔE)	5.98	5.50±0.24

CHAPTER V

SUMMARY AND CONCLUSION

CHAPTER V

SUMMARY AND CONCLUSION

In this study, the pulsed vacuum osmotic dehydration (PVOD) of carrot slices was carried out under different processing conditions. Moreover, response surface methodology (RSM) was applied to analyze the effect of different process variables. In addition to, RSM was also used to predict the optimum operating conditions that yield maximum water loss (WL), minimum solute gain (SG) and color change (CC) during osmotic dehydration of carrot slices. For this purpose, different processing conditions such as process temperature (^{o}C), sucrose and salt concentration (%) and restoration time (min) were designed, developed, evaluated and optimized. In order to obtain a better understanding of different process variables the developed models was studied for analysis of variance (ANOVA) and presented as response surface and contour plots for each response. Second order polynomial models were obtained for predicting water loss, solute gain and color change.

The restoration time has the most significant effect on WL and SG followed by sucrose concentration, salt concentration, and temperature at 5% level of significance. It implies that increase on WL and SG as the restoration time proceeds. This behavior is due to the influence of the application of vacuum pulse at the beginning of the process which removes the air from the porous spaces of the food. Thus, the higher rate of mass transfer obtained during osmotic dehydration at atmospheric pressure. However, the color change was significantly influenced by the temperature of osmotic solution ($p<0.05$) followed by restoration time, salt and sucrose concentration. It indicates that color changes with enhanced temperature of the osmotic solution. This is because of the presence of carotene which is responsible for the actual color of carrots. Further, as the temperature increased non-enzymatic browning reaction took place resulting oxidation and isomerization of carotene during processing and degraded the color of the carrot slice. The desirability function technique was useful for finding optimum conditions during PVOD process of carrot slice. Optimum solution by numerical optimization obtained was 52.3964% (wb), 11.68% (wb) and 5.98 for water loss, solute gain and color change, respectively. After obtaining optimum conditions for various predicted independent variables, PVOD experiments of carrot slices were carried out to validate predicted values for each response. The experimental values measured for WL, SG and CC were near about with the predicted values.

Therefore, pulsed vacuum osmotic dehydration of carrot slice could effectively be carried out prior to further drying to remove a large portion of moisture at the low temperature, which is beneficial in terms of energy saving and to maintain the natural property of the product.

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