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Modeling and Optimization of Ultrasound Assisted Osmotic Dehydration of Cranberry Using Response Surface Methodology

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ABSTRACT

In this study, we investigated the effects of osmotic process with or without ultrasound on solid gain (SG) and water loss (WL) of cranberries. Response surface methodology was used to model and determine the optimum processing conditions for WL and SG during osmotic dehydration of samples. Sucrose (40-60%) and salt (0-8%) concentrations, temperature (30-50°C) and frequency of ultrasound (0-130 kHz) were the factors investigated with respect to WL and SG. Experiments were designed according to a second-order Central Composite Design (CCD) in the form of a Face-Centered Cube (FCC) with these four factors, each at three different levels, including central and axial points. All experiments were conducted in triplicate. Experiments were conducted in a shaker with constant 150 rpm agitation and solution to sample mass ratio of 10/1 (w/w). Analysis of variance (ANOVA) was performed to check the adequacy and accuracy of the fitted models. Statistical analysis of results showed that the linear terms of all the process variables had a significant effect on WL. Except for temperature, all other parameters had a significant effect on SG. Optimum operating conditions were found to be sucrose concentration of 50.1%, salt concentration of 8%, temperature of 50°C and ultrasound frequency of 130 kHz.

Keywords: Cranberry, Modeling, Optimization, Osmotic dehydration, Response surface methodology, Ultrasound.

INTRODUCTION

Cranberry, *Vaccinium marcrocarpon Ait.,* is a member of the Ericaceae family, evergreen, creeping shrubs native to cool temperature, acidic soils and peat wetlands (Roper and Vorsa, 1997). It is cultivated in northern and north western parts of Iran and contains high concentrations of phytochemicals which have health promoting properties (He and Liu, 2006; Neto, 2007b; Torres *et al*., 2006). Some of these phytochemicals, which act as antioxidants such as anthocyanins, reduce the oxidative damage to cells that can lead to cancer, heart disease, and other degenerative diseases (Zafra-Stone *et al*., 2007). The antioxidant properties of cranberries are documented in the literature and cranberries are ranked one of the highest antioxidant activities among many other fruits (Sun *et al.,* 2002).

Recently, cranberry products have been used for preventing or treating urinary tract infections or *Helicobacter pylori* infections that can lead to stomach ulcers, or to prevent dental plaque (He and Liu, 2006). Cranberry is a seasonal and perishable fruit; therefore, a number of processes such as cold storage, concentration, reducing to paste, or drying are used to conserve it.

Dehydration is an important operation for preserving cranberries. The quality of

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dehydrated products is dominated by drying methods and conditions. Conventional hot-air drying results in extremely shrunken products with tough texture, severe browning, low rehydration rate, and low nutrition value. Moreover, it is energy intensive and consequently cost intensive due to its simultaneous mass and heat transfer processes accompanied by phase change (Deng and Zhao, 2008).

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 (Eren and Kaymak-Ertekin, 2007).

Osmotic dehydration has been found to be effective even at ambient temperature. It is known to protect the color, flavor and texture of food from heat and is used as a pretreatment to improve the nutritional, sensorial and functional properties of food (Fenandes *et al*., 2006; Singh *et al*., 2010).

Mass transfer rates during osmotic dehydration depends on factors such as temperature, concentration of osmotic medium, size and geometry of the samples, sample to solution ratio, and degree of agitation of the solution (Ispir and Türk Togˇrul, 2009; Kaymak-Ertekin and Sultanoglu, 2000; Rastogi and Raghavarao, 2004a, b; Singh *et al*., 2007; Fernandes *et al.,* 2007; Falade *et al.,* 2007; Rastogi *et al.,* 2002; Rahimzade Khoyi and Hesari, 2007). The rate of mass transfer during osmotic dehydration is generally low. Applying ultrasound is a method that can improve mass transfer rate (Simal *et al*., 1998) because the ultrasonic waves can cause a rapid series of alternative compressions and expansions, in a similar way to sponge when it is squeezed and released repeatedly (sponge effect). The forces involved in this mechanical mechanism can be higher than surface tension which maintains the moisture inside the capillaries of the fruit creating microscopic channels that may ease moisture removal and increase mass transfer (Fernandes *et al*., 2008; Simal *et al*., 1998). In addition, ultrasound can cause fast and complete degassing, initiate various reactions by generating free chemical ions (radicals) and enhance polymerization/depolymerization reactions (Stojanovic and Silva, 2007).

During osmotic dehydration, water removal from the product is always accompanied by the simultaneous counter diffusion of solutes from the osmotic solution into the tissue. Depending upon the process variables, the amount of diffusing solute is generally about 5-10% of the initial weight of the product. This amount not only modifies the composition and the taste of the final product, but also blocks the surface layers of the material, posing an additional resistance to mass exchange and lowering the rates of complementary and subsequent (vacuum, convection and freeze) dehydration. In such situations, it becomes more important to determine the optimum processing conditions that yield maximum water loss and minimum solid gain during osmotic dehydration.

Response Surface Methodology (RSM) is an important tool in process optimization and product quality improvement. RSM is a collection of experimental design and optimization techniques that enables the researcher to determine the relationship between the response and the independent variables. RSM is typically used for mapping a response surface over a particular region of interest, optimizing the response, or for selecting operating conditions to achieve target specifications or customer requirements (Eren and Kaymak-Ertekin, 2007).

In this paper, we are studying the osmotic dehydration of cranberries with or without ultrasound to determine the effects of process parameters on solid gain and water loss. Response surface methodology was

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used for modeling and optimization of process parameters.

MATERIALS AND METHODS

Osmotic Dehydration

Sample Preparation: The cranberries were purchased from a local market (Karaj, Iran). They were sorted visually for maturity and size, were washed with tap water and surface dried with a filter paper. To increase permeability of the skin, cranberries were dipped in NaOH (0.5 Molar) for 2 minutes (samples were washed after dripping to avoid any NaOH residual). The average initial moisture content was 85% on wet basis, gravimetrically measured using an oven at 105°C for 18 hour (time required to stabilize its weight (Deng and Zhao, 2008).

Osmotic Dehydration without the Application of Ultrasound: Osmotic dehydration was done in solution of sucrose-salt mixture having different concentrations. The concentration of osmotic solutions were 40, 50, 60% sucrose and 0, 4, 8% NaCl.

Cranberries were weighed and placed in the osmotic solution under dynamic conditions provided by agitation (150 rpm) at ambient temperature (25°C). The sample/solution ratio was high at 1:10 (w/w) to limit the decrease of ratio of sample to solution and thus to avoid significant dilution of the medium from the release of water and subsequent decrease of the osmotic driving force during the process (Emam-Djomeh *et al.* 2001).

Samples were then removed from the solution at different time intervals (2, 4, 6, 8, 10 and 12 hour), washed by distilled water and dried with an absorbent paper in order to remove the excess solution on the surface.

Osmotic Dehydration with the Application of Ultrasound: Experiments with ultrasound application were carried out in an ultrasound bath (Elma, D-78224 Singen/Htw, Germany). Water temperature inside the ultrasonic bath was maintained constant during the osmotic experiments. Two levels of frequency were tested: 35 and 130 kHz. The electrical power output was 100%. Samples were removed from the solution at different time intervals (10, 20, 30, 40, 50, 60, 70 and 80 minutes), washed with distilled water and dried with an absorbent paper.

Analytical Methods

Moisture Content: Measurements were carried out on fresh samples and after drying process in triplicate. Moisture content was determined gravimetrically by drying in an oven at 105°C for 18 hour (Deng and Zhao, 2008).

Measurement of Water Loss and Solid Gain: Measurements were performed on fresh samples and after osmotic process in triplicate. Water loss (WL) and solid gain (SG) of osmosised samples were calculated using the following relationships (Dehghannya *et al.*, 2006):

$$SG = \frac{(W_s - W_t)}{W_0} \times 100 \qquad (1)$$

$$WL = \left[\frac{W_0 - W_{0s}}{W_0} \times 100\right] + SG \qquad (2)$$

Where, W_t and W_S are, respectively dry weight of blank and dry weight of sample after osmotic process. W_{0s} and W_0 stand for weight of sample after osmotic process and the initial weight of sample, respectively.

Experimental Design

The response surface methodology (Minitab 14 software) was used to estimate the main effects of process variables on water loss (WL) and solid gain (SG) during osmotic dehydration of cranberries. Sucrose concentration (X_1), NaCl concentration (X_2), temperature (X_3) and frequency of ultrasound (X_4) were selected as independent variables by means of literature survey and preliminary experiments. A second-order Central Composite Design (CCD) in the form of a Face-Centered Cube (FCC) with four factors (sucrose concentration, NaCl concentration, temperature and frequency of ultrasound) at

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three levels each was used for cranberries. All experiments were conducted in triplicate (Changrue *et al*., 2008). The actual factor values and corresponding coded values (-1, 0, 1) for cranberries are given in Table 1.

Model Development

The model was developed from regression coefficients under a range of experimental factors. The coefficient of determination (R^2) was used to indicate how the model fits the variability of the results. The terms of second-order polynomial model consist of linear, quadratic (squared) and interaction terms as shown by the following equation:

$$\begin{aligned} Y_1 &= b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{11}X_1^2 \\ &+ b_{22}X_2^2 + b_{33}X_3^2 + b_{44}X_4^2 + b_{12}X_1X_2 + b_{13}X_1X_3 \\ &+ b_{14}X_1X_4 + b_{23}X_2X_3 + b_{24}X_2X_4 + b_{34}X_3X_4 \end{aligned} \quad (3)$$

Where, b_n parameters are the regression coefficients; Y_1 is response either *WL* or *SG* of cranberries; X_1, X_2, X_3 and X_4 in Equation (1) are sucrose concentration (%w/w), NaCl concentration (%w/w), temperature (°C) and frequency of ultrasound (kHz), respectively. Analysis of variance (ANOVA) was performed to check the adequacy and accuracy of the fitted models (Changrue *et al*., 2008).

Optimization

During optimization of industrial processes, usually several response variables describing the quality characteristics and performance measurements of the systems, are to be optimized while some are to be minimized. In many cases, responses are competing, i.e., improving one response may have an opposite effect on another one, which further complicates the situation. Several approaches have been used to tackle this problem. One approach uses a constrained optimization procedure, the second is to superimpose the contour diagrams of the different response variables, and the third approach is to solve the problem of multiple responses through the use of a desirability function that combines all responses into one measurement. The advantages of using desirability functions include the following: (1) responses that have different scaling can be compared, (2) the transformation of different responses to one measurement is simple and quick, and (3) both qualitative and quantitative responses can be used (Singh *et al*., 2010).

It is based on the idea that the "quality" of a product or process with complex characteristics is not acceptable, when one of its parameters is outside of "desired" limits. The method finds operating conditions x that provide the "most desirable" response values.

In the present study, desirability functions developed for the criteria of maximum water loss and minimum solid gain are given in Tables 1 and 2.

Table 1- Selected levels for factors.

Factors/Levels	Low (-1)	Medium (0)	High (+1)
Sucrose concentration (%)	40	50	60
NaCl concentration (%)	0	4	8
Temperature (°C)	30	40	50
Frequency of ultrasound (kHz)	0	65	130

Table 2- Selected points for optimization of solid gain and water loss of cranberries.

	Goal	Lower	Target	Upper	Weight
SG (%)	Minimize	-	3	4	1
WL (%)	Maximize	20	56	-	1

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RESULTS ANDD DISCUSSION

Effect of Sucrose and NaCl Concentrations on *WL* and *SG*

Results indicated that by increasing sucrose and NaCl concentrations in osmo process with and without ultrasound, WL and SG were increased, which is due to increasing osmosis pressure gradient. Figure1(a, b, c, d, m and n)

Effect of Temperature on *WL* and *SG*

Effect of temperature on *WL* and *SG* was explained by Arrhenius law. Increasing the temperature intensifies diffusive coefficient. In addition, increasing the temperature decreased the viscosity of osmotic solution that caused convective mass transfer coefficient to increase thereby resulting in higher mass transfer. (Figure1-c, d, g, h, k, l, p, s, t)

Effect of Frequency of Ultrasound on *WL* and *SG*

Water loss and solid gain rates were faster when ultrasound was used to carry out the osmotic dehydration. (Figure 1-i, j, e, f, k, l, q, r, u, v, x,w)

In the treatments without ultrasound, the required time to attain equilibrium was approximately 12 hours whereas in the treatments with ultrasound it decreased approximately to 40-60 minutes (Ispir and Türk Togˇ rul, 2008; Simal *et al.,*1998).

In ultrasound process with low frequency waves (16-100 kHz), effects such as streaming, cavitation and interface instabilities were observed. Mechanisms of these effects consist of:

Cavitation: The formation, growth and violent collapse of small bubbles or voids in liquids as a result of pressure fluctuation.

Rectified diffusion: When high intensity acoustic energy travels through a solid medium, the sound wave causes a series of rapid and successive compressions and rarefactions with rates depending on their frequency.

Acoustic streaming: At liquid/solid or gas/solid interfaces, acoustic waves cause extreme turbulence known as “acoustic streaming” or “micro streaming”.

These mechanisms lead to a decrease in the thickness of the boundary layer which exists between a suspended solid and a liquid, an increase in the temperature of the medium and structure deformation such as the production of many fractures on the surface of the fruit and creation of micro channels in cell walls resulting in an increase in mass transfer (Fernandes *et al.*, 2008; Rastogi *et al.*, 2002; Tarleton *et al.*, 1998.

By increasing the frequency of ultrasound from 65 to 130 kHz, cavitation, compression and rarefaction, localized pressure, fractures on the surface and micro channels in cell walls were increased thereby increasing mass transfer. Therefore, the frequency of 130 kHz had higher water loss than 65 kHz whereas solid gain at 65 kHz was more than that at 130 kHz probably because at the frequency of 130 kHz high water flux prevented solid intake.

Interaction Effects of Variables on Water Loss and Solid Gain

Figure1 a to l show that the interaction effects of sucrose and NaCl concentration, sucrose concentration and temperature, sucrose concentration and frequency of ultrasound, NaCl concentration and temperature and NaCl concentration and frequency of ultrasound on water loss were significant. By increasing sucrose and NaCl concentration, sucrose concentration and temperature, sucrose concentration and frequency of ultrasound, NaCl concentration and temperature and NaCl concentration and frequency of ultrasound water loss was increased. Because high water loss in osmotic dehydration is very important, high

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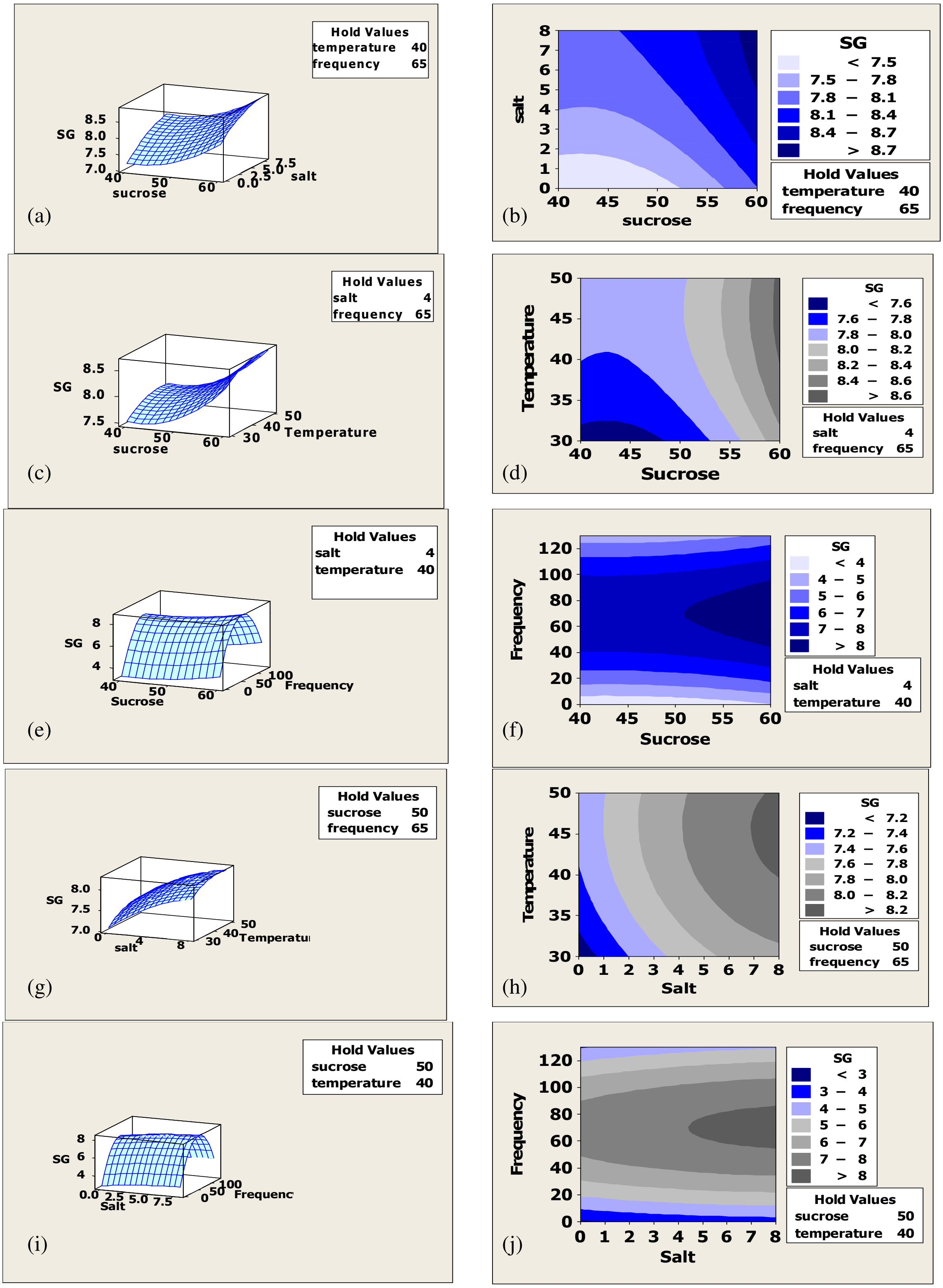

Figure1. Surface plots (left) and contour plots (right) showing the effect of different variables on water loss (WL) and solid gain (SG) in cranberries during osmotic dehydration.

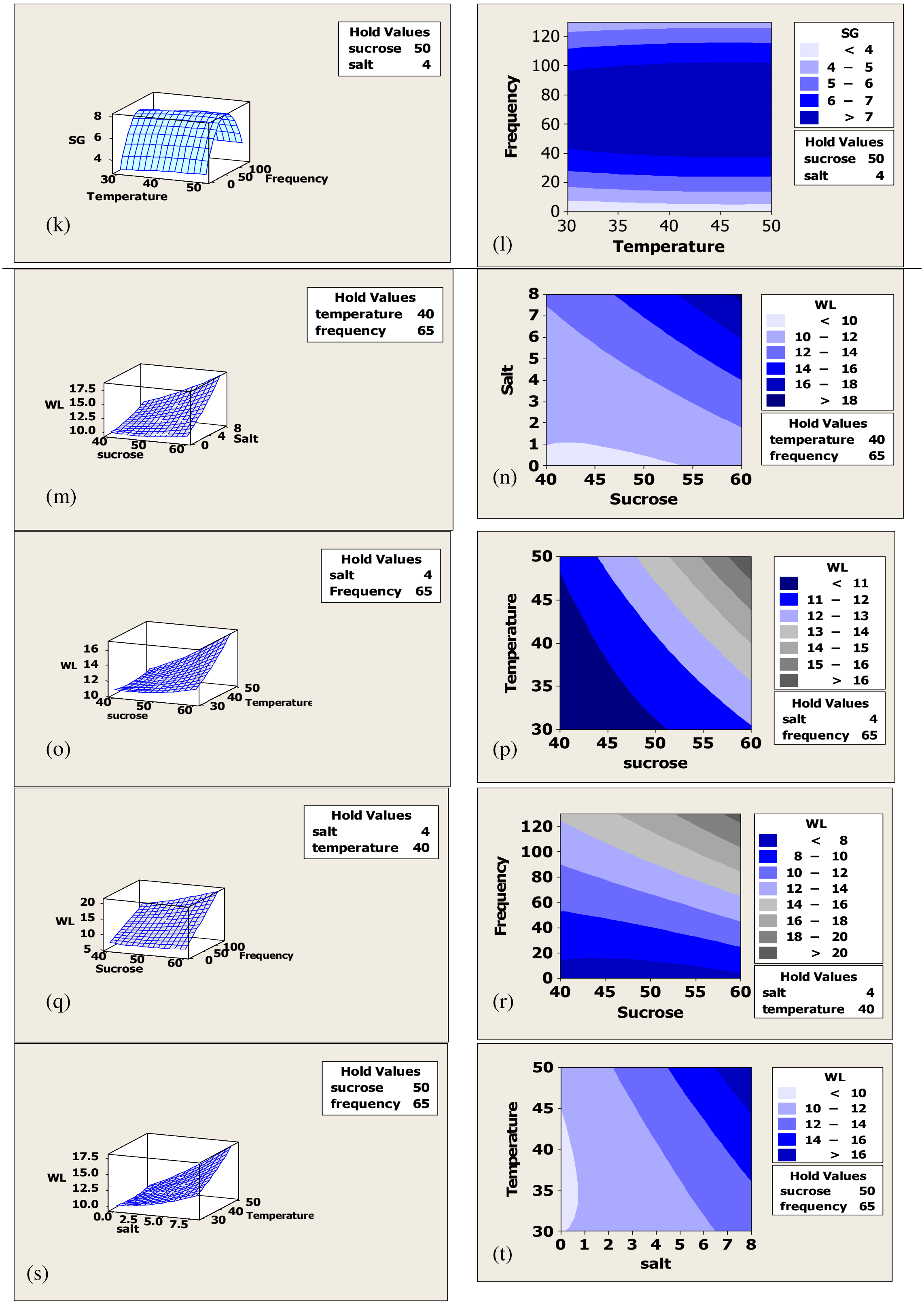

Figure1. Continued

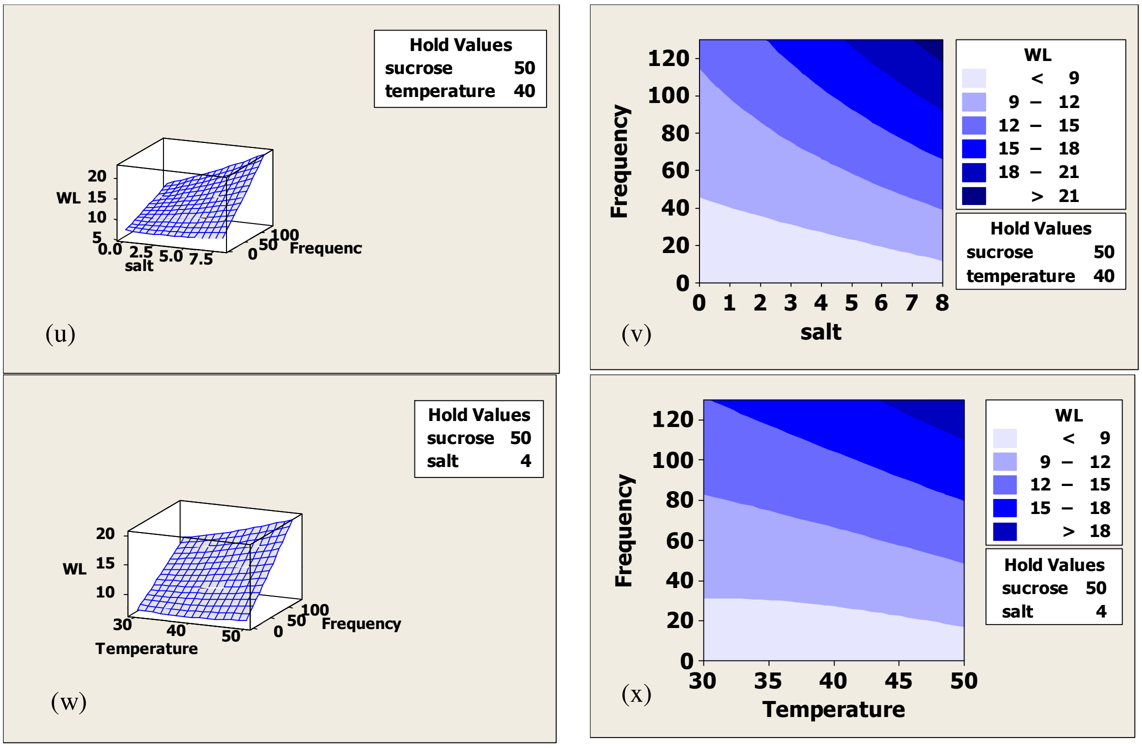

Figure1. Continued

values of all of these parameters are desirable. The interaction effects of variables on solid gain were not statistically significant.

Predictive Model for Water Loss and Solid Gain of Cranberries

The coefficients of determination (R^2) for *WL* and *SG* of cranberries were determined to be 77.1 and 88.5%, respectively. Regression coefficients of Equations (1 and 2) for predictive models *WL* and *SG* of cranberries as shown in Table 3 and 4 provided the predictive equations in actual terms (uncoded) as the following:

$$WL = 11.8717 + 1.6915X_1 + 2.5413X_2 + 1.3159X_1 + 5.0412X_4 + 1.3702X_1X_2 + 1.1501X_1X_3 + 1.5337X_1X_4 + 1.1161X_2X_3 + 2.3001X_2X_4 + 1.2342X_3X_4$$

(4)

$$SG = 7.93643 + 0.40319X_1 + 0.41373X_2 + 0.15743X_3 + 0.59583X_4 - 4.02788X_4^2$$

(5)

Where, X_1 is sucrose (%w/w), $40 < X_1 < 60$, X_2 is NaCl concentration (%w/w), $0 < X_2 < 8$, X_3 is temperature (°C), $30 < X_3 < 50$ and X_4 is the frequency of ultrasound (kHz), $0 < X_4 < 130$.

Optimization of Water Loss and Solid Gain

Optimization of dependent variables was done by using the information in Tables 2 and 3. Figures 2 and 3 show that desirability in the second method (Figure 3) was higher. However optimum operating conditions were found to be sucrose concentration of 50.1%, salt concentration of 8%,

Table 3- Selected points for optimization of solid gain (SG) and water loss (WL) of cranberries.

	Goal	Lower	Target	Upper	Weight
SG (%)	Minimize	-	3	7	1
WL (%)	Maximize	12	56	-	1

Table 4- Regression equation coefficients for water loss (WL) and solid gain (SG) of osmotically dehydrated cranberries.

	SG Coefficients	p-value	WL Coefficient	p-value
Model	7.93643	0.000	11.8717	0.000
Linear				
X_1(Sucrose)	0.40319	0.001	1.6915	0.000
X_2(NaCl)	0.41373	0.000	2.5413	0.000
X_3(temperature)	0.15743	0.164	1.3159	0.003
X_4(frequency)	0.59583	0.000	5.0412	0.000
Quadratic				
X11	0.26932	0.364	0.4374	0.703
X22	-0.14485	0.625	0.4708	0.681
X33	-0.12755	0.667	0.3702	0.747
X44	-4.02788	0.000	0.1599	0.889
Interaction effect				
X_1X_2	-0.02570	0.829	1.3702	0.004
X_1X_3	0.00344	0.977	1.1501	0.015
X_1X_4	0.01226	0.918	1.5337	0.001
X_2X_3	-0.01827	0.878	1.1161	0.018
X_2X_4	0.07837	0.511	2.3001	0.000
X_3X_4	-0.00555	0.963	1.2342	0.009
R^2	88.5%		77.1%	
R^2-Adj	86.5%		72.9%	
CV	0.8228		3.187	

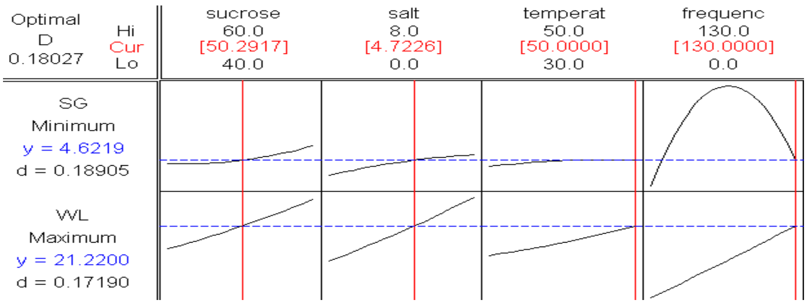

Figure 2. Optimization of WL and SG for parameters in Table 2.

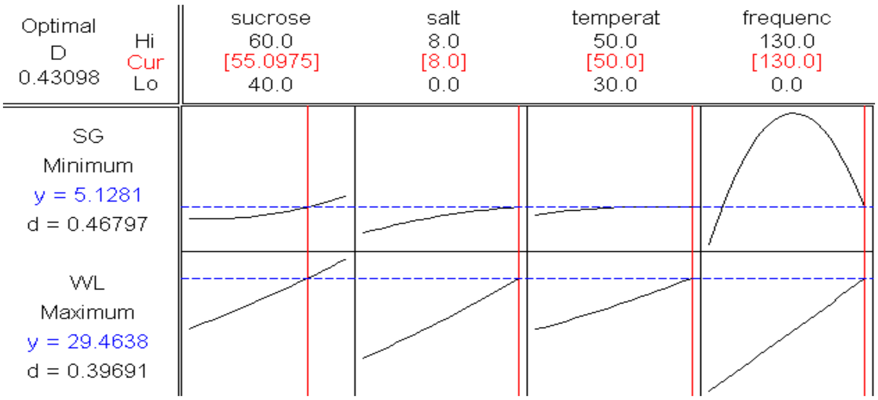

Figure 3. Optimization of WL and SG for parameters in Table 3.

temperature of 50°C and frequency of 130 kHz. At the optimum point, predicted responses were 5.13 and 29.46% for solid gain and water loss, respectively.

CONCLUSIONS

This study confirmed that using ultrasonic process decreased time of osmotic dehydration from 9 hours to 40 minutes. In addition, ultrasound waves increased water loss and solid gain. With increasing the frequency of ultrasound water loss was increased but solid gain was decreased as a result of high water flux.

Parameters of these processes such as sucrose and NaCl concentration, temperature and frequency of ultrasound considerably affected solid gain and water loss. Results showed that with increasing sucrose and NaCl concentration water loss and solid gain were increased because of intensifying osmotic pressure. Raising temperature caused an increase in water loss and solid gain because it increased the activation energy and decreased the viscosity. Results also showed that the linear terms of all process variables had significant effects on *WL*. Except for temperature, the other parameters had significant effects on *SG*. Optimum operating conditions were found to be sucrose concentration of 50.1%, salt concentration of 8%, temperature of 50°C and frequency of 130 kHz.

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مدل‌سازی و بهینه‌سازی خشک کردن اسمزی-اولتراسوند کرن‌بری با روش سطح پاسخ

س. شمایی، ز. امام جمعه، و س. معینی

چکیده

در این پژوهش اثر خشک کردن اسمزی همراه با کاربرد اولتراسوند بر روی جذب مواد جامد و افت آب در کرن‌بری بررسی گردید. روش سطح پاسخ به منظور مدل‌سازی و تعیین نقطه‌ی بهینه‌ی فرایند خشک کردن اسمزی، جهت دستیابی به بیشینه‌ی کاهش محتوای رطوبتی ماده‌ی غذایی و کمینه‌ی جذب مواد جامد از محلول اسمزی مورد استفاده قرار گرفت. غلظت ساکارز (٪۶۰-۴۰ وزنی/ وزنی)، غلظت نمک کلرید سدیم(٪۸-۰ وزنی/ وزنی)، درجه حرارت (۵۰-۳۰°C) و فرکانس اولتراسوند

(۱۳۰-۰ کیلو هرتز) فاکتورهایی بودند که تاثیر آنها بر روی کاهش محتوای رطوبتی ماده‌ی غذایی و جذب مواد جامد از محلول اسمزی مورد ارزیابی قرار گرفت. آزمایش‌ها بر اساس طرح کامپوزیت مرکزی با در نظر گرفتن سه سطح شامل نقاط مرکزی و محوری برای هر یک از فاکتورهای یاد شده، انجام شدند. در آزمایش‌های انجام شده دور همزنی برابر ۱۵۰ دور/ دقیقه و نسبت محلول اسمزی به نمونه ۱/۱۰ وزنی/ وزنی در نظر گرفته شد. برای بررسی صحت و دقت مدل، آنالیز واریانس انجام شد. آنالیز آماری نتایج نشان داد که اثر خطی کلیه پارامترهای فرایند بر روی افت آب معنی‌دار بوده است درحالی‌که برای جذب مواد جامد اثر کلیه فاکتورها به جز دما معنی‌دار می‌باشد. شرایط بهینه عملیاتی غلظت ساکارز ۵۰/۱٪، غلظت نمک ۸٪، دما ۵۰ °C و فرکانس اولتراسوند kHz ۱۳۰ تعیین گردید.