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ScienceDirect
LWT - Food Science and Technology 41 (2008) 2044–2050
LWT
www.elsevier.com/locate/lwt

Optimization of osmotic dehydration of diced green peppers by response surface methodology

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Received 19 June 2007; received in revised form 4 January 2008; accepted 18 January 2008

Abstract

Osmotic dehydration of diced green peppers was optimized with respect to temperature (20–40 °C), time (15–600 min), salt (0–10 g/100 g) and sorbitol (0–10 g/100 g) concentrations through response surface methodology. Water loss (WL), solids gain (SG), salt uptake (SA) and sorbitol uptake (SO) were the responses in a 2^4 central composite rotatable design. Models developed for all responses were significant ($p \leq 0.01$) without significant lack of fit. Results suggested that optimum processing conditions of 5.5 g salt/100 g and 6 g sorbitol/100 g at 30 °C after 240 min would result in WL = 23.3%, SG = 4.1%, SA = 8 g/100 g dry pepper and SO = 2.4 g/100 ml extract.

Keywords: Osmotic dehydration; Green pepper; Optimization; Response surface methodology

1. Introduction

Osmotic dehydration (OD) has been used for many years to remove water from fresh fruits and vegetables and increase their storage stability. Fruits and vegetables are placed in an osmotic solution, which creates a concentration gradient between the solution and the intracellular fluid. This driving force results in the removal of water from the food through cellular membranes. These membranes are semi-permeable in nature, allowing water molecules to pass through easier than solute molecules (Raoult-Wack, 1994; Torreggiani, 1993).

The use of OD in food processing, particularly prior to drying and freezing operations, reduces energy requirements of these processes (Rahman & Perera, 1999; Raoult-Wack, 1994). OD also helps retain the functional and organoleptic properties of foods, particularly fruits and vegetables (Ertekin & Cakaloz, 1996; Torreggiani, Forni, Erba, & Longoni, 1995).

The effect of several factors including the type and concentration of osmotic agents (Biswal & Bozorgmehr, 1992; Collignan & Raoult-Wack, 1994; El-Aouar, Azoubel, Barbosa, & Murr, 2006; Lerici, Pinnavaia, Dalla Rose, & Bartolucci, 1985), processing temperature and time (Biswal & Bozorgmehr, 1992; Lenart & Flink, 1984), agitation (Lenart & Flink, 1984; Mavroudis, Gekas, & Sjoholm, 1998; Vijayanand, Chand, & Eipeson, 1995), tissue to solution ratio (Vijayanand et al., 1995) and raw material characteristics (Mavroudis et al.,1998) on the OD of fruits and vegetables, meat and fish have been investigated. Investigation of factors affecting the OD of a specific food product provides invaluable information on the most important processing variables and their levels prior to optimization studies (Ozen, Dock, Ozdemir, & Floros, 2002).

Response surface methodology (RSM) is an effective tool for optimizing a variety of food processes including osmotic dehydration (Azoubel & Murr, 2003; Corzo & Gomez, 2004). The principles and foundations of RSM were first introduced by Box and Wilson (1951). The main advantage of RSM is the reduced number of experimental runs that provide sufficient information for statistically valid results. RSM is faster and more

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0023-6438/$34.00
doi:10.1016/j.lwt.2008.01.010

informative than the classical one-variable-at-a-time approach or the use of full factorial designs. Quintero-Ramos, De La Vega, Hernandez, and Anzaldua-Morales (1993) used RSM to study the effect of sugar concentration, temperature and time on the OD of apples. The OD resulted in a 30% weight loss and protected the apple dices from adverse heat effects and oxidation during drying. Vijayanand et al. (1995) used RSM to optimize the processing conditions of OD of cauliflower. Cauliflower processed at 80 °C for 5 min in a brine to tissue ratio of 2:1, and an osmotic salt solution (12 g/100 g) lost about 40% of its water with a minimum salt uptake of 4.2 g/100 g. At these optimum conditions, enzymatic browning was prevented. Corzo and Gomez (2004) optimized the OD of the cantaloupe using the desired function methodology of response surface optimization. The optimal conditions for OD of the cantaloupe corresponded to temperature of 38 °C, concentration of 41.6 °Brix, and time of 132 min.

Green peppers are widely used in various food products including salsas, pizzas, salads and other similar products without being osmotically dehydrated. OD can increase the stability of green peppers in these products. Osmotically dehydrated green peppers may then be used by restaurants and other food service establishments, or as an ingredient in the manufacturing of these products. The objective of this work was to determine optimum processing conditions during the OD of diced green peppers through the use of RSM. Such optimum conditions should provide a final product with maximum weight loss and minimum solids gain.

2. Materials and methods

2.1. Theoretical considerations

Factors affecting the osmotic dehydration of diced green peppers were investigated with a fractional factorial design by Ozen et al. (2002). The work provided very valuable information on the most important processing variables (factors, inputs or independent variables), their levels and responses (output or dependent variables) for the osmotic dehydration of diced green peppers. This information was used in the experimental design chosen to optimize the osmotic dehydration of diced green peppers. The factors studied were salt concentration (x_1), sorbitol concentration (x_2), temperature (x_3) and time (x_4). The responses measured were water loss (y_1), solids gain (y_2), salt uptake (y_3) and sorbitol uptake (y_4). Furthermore, it was assumed that four functions f_n ($n = 1, 2, 3, 4$) exist between each response and the input factors:

$$y_n = f_n(x_1, x_2, x_3, x_4) \quad (1)$$

Since the exact nature of the true function(s) is either unknown or too complex, these functions were approximated by second order polynomials (Floros & Chinnan, 1988):

$$y_n = \beta_{n0} + \sum_{i=1}^{4} \beta_{ni} x_i + \sum_{i=1}^{4} \beta_{nii} x_i^2 + \sum_{i=1}^{3} \sum_{j=i+1}^{4} \beta_{nij} x_i x_j \quad (2)$$

where β_{n0}, β_{ni}, β_{nii} and β_{nij} are constant coefficients, and x_i are the factors.

2.2. Experimental design and data analysis

A central composite rotatable design (CCRD) for four factors was used as the experimental design (Table 1). CCRD includes a 2^4 design with eight star points and six replicate center points. The rotatability property of the design provided a reasonably constant variance of response in the four-dimensional space. Since the design was orthogonal, it minimized the variance of the estimated regression coefficients. The RSREG procedure of SAS (SAS Institute Inc., 1990) was used to fit second order polynomial models and determine the β coefficients. A logarithmic transformation was done to obtain equal spacing between each time measurement.

2.3. Raw material preparation

Green bell peppers (*Capsicum annuum*) of variety Jupiter were harvested and stored at 5 °C and relative humidity (RH) > 95% for 8 h. Just before use, green peppers were cleaned and cut into small pieces of approximately 1 cm^2.

Table 1
Experimental design and data[a]

Factors				Responses			
Salt (x_1)	Sorbitol (x_2)	Temp (x_3)	Time (x_4)	Water loss[b] (WL)	Solids gain[c] (SG)	Salt uptake[d] (SA)	Sorbitol uptake[e] (SO)
2.5	2.5	25	240	16.27	0.89	4.52	1.12
2.5	2.5	35	36	10.80	0.45	2.58	0.74
2.5	7.5	25	36	13.50	1.02	2.25	1.47
2.5	7.5	35	240	20.65	2.48	3.76	2.91
7.5	2.5	25	36	15.24	1.68	7.63	0.42
7.5	2.5	35	240	19.47	4.23	12.95	1.15
7.5	7.5	25	240	24.41	5.58	9.69	3.41
7.5	7.5	35	36	15.97	2.85	7.45	1.62
5	5	30	96	16.33	2.76	7.24	2.04
5	5	30	96	19.20	2.12	7.59	1.31
2.5	2.5	25	36	12.40	0.38	2.62	0.56
2.5	2.5	35	240	20.40	1.23	4.01	0.96
2.5	7.5	25	240	23.14	1.91	3.38	2.21
2.5	7.5	35	36	13.61	0.99	2.04	1.80
7.5	2.5	25	240	22.86	3.79	11.39	0.98
7.5	2.5	35	36	15.20	2.08	7.77	0.67
7.5	7.5	25	36	16.81	2.72	6.73	1.38
7.5	7.5	35	240	27.65	5.76	10.45	2.86
5	5	30	96	18.15	3.44	5.92	1.55
5	5	30	96	18.35	3.64	5.38	1.45
0	5	30	96	5.26	0.04	0	0.86
10	5	30	96	24.81	4.47	10.47	1.47
5	0	30	96	18.87	1.24	7.67	0
5	10	30	96	23.31	3.26	5.53	2.23
5	5	20	96	18.99	2.03	5.71	1.40
5	5	40	96	21.49	2.50	6.85	1.26
5	5	30	15	9.95	0.82	3.85	0.93
5	5	30	600	24.11	3.94	8.37	2.42
5	5	30	96	20.03	2.50	6.36	1.45
5	5	30	96	20.08	2.25	6.16	1.33

[a] The total of 30 treatments were carried out in a random order.
[b] As % of initial weight.
[c] As % of initial weight.
[d] g/100 g dry pepper.
[e] g/100 ml extract.

2.4. Preparation of osmotic solutions

Salt (NaCl) was obtained from Mallinckrodt Baker (Paris, KY) and sorbitol from Sigma Chemical (St. Louis, MO). Osmotic solutions were prepared by dissolving required amounts of salt (0–10 g) and/or sorbitol (0–10 g) in distilled water (Table 1).

2.5. Osmotic dehydration experiments

Beakers with 30 g of diced peppers and the desired osmotic solution were placed in a water bath. Temperatures in the water baths were maintained at 20, 25, 30, 35 or 40 °C within ±1 °C as dictated by the experimental design (Table 1). Approximately 10 g of the 30 g diced peppers were contained in separate net-cages holding for water loss and solids gain determinations. According to the time required by the experimental design scheme (Table 1), diced peppers were removed from the osmotic solution, gently blotted dry with tissue paper for a few seconds and weighed. Tissue to solution ratio of 1:3 (w/v) was used.

2.6. Water loss, solids gain and salt uptake

Samples for water loss, solids gain and salt uptake measurements were dried in a vacuum oven at 70 °C overnight. Percent WL and SG were calculated from formulas given by Hawkes and Flink (1978):

$$\mathrm{WL} = \frac{W_0 - (W - S)}{S_0 + W_0} \times 100 \tag{3}$$

$$\mathrm{SG} = \frac{S - S_0}{S_0 + W_0} \times 100 \tag{4}$$

where W_0 is the initial weight of water, S_0 is the initial weight of solids, W is the final weight of tissue and S is the weight of solids at the end of the process.

The dried samples for salt uptake measurements were digested for 1 h at 180 °C using nitric acid (70 ml/100 ml) followed by H_2O_2 (30 ml/100 ml). The salt concentration was determined by an inductively coupled plasma/atomic emission spectroscopy using a Perkin Elmer Plasma 400 ICP/AES instrument (Norwalk, CT), and results were expressed in terms of dry basis.

Table 2
Analysis of variance for the four responses

Source	df	Sum of squares			
		Water loss (WL)	Solids gain (SG)	Salt uptake (SA)	Sorbitol uptake (SO)
Model	14	617.38*	64.74*	257.60*	15.73*
Linear	4	560.58*	61.44*	248.22*	14.29*
Quadratic	4	48.49	1.01	2.37	0.41
Cross product	6	8.31	2.29	7.01**	1.03
Residual	15	92.39	2.46	5.88	1.27
Lack of fit	12	88.23	2.20	5.65	0.99
Pure error	3	4.15	0.26	0.23	0.28
Correlation coefficient (R^2)		0.87	0.96	0.98	0.92

*Significant at $p \leq 0.01$.
**Significant at $p \leq 0.05$.

Table 3
Values of the second order polynomial regression coefficients for the four responses

Regression coefficient[a] (β_n)	Water loss (WL) ($n=1$)	Solids gain (SG) ($n=2$)	Salt uptake (SA) ($n=3$)	Sorbitol uptake (SO) ($n=4$)
β_{n0}	6.564	−9.412	0.214	−0.113
β_{n1}	2.543	0.064	0.182	0.003
β_{n2}	−1.830	0.326	0.184	−0.118
β_{n3}	−1.051	0.342	−0.132	0.108
β_{n4}	12.587	1.762	1.212	−1.559
β_{n11}	−0.151	−0.020	−0.043	−0.009
β_{n22}	0.091	−0.019	0.011	−0.011
β_{n33}	0.014	−0.005	−0.001	−0.001
β_{n44}	−2.727	−0.523	−0.293	0.467
β_{n12}	0.011	0.009	−0.031	0.010
β_{n13}	−0.006	−0.003	0.018	−0.005
β_{n14}	0.096	0.337	0.525	0.104
β_{n23}	0.005	−0.006	0.002	0.001
β_{n24}	0.639	0.110	−0.211	0.199
β_{n34}	0.121	0.006	0.048	−0.026

[a] These are coefficients of Eq. (2) and subscripts 1, 2, 3 and 4 represent salt concentration, sorbitol concentration, temperature and time, respectively.

2.7. Sorbitol uptake

Juice from approximately 20 g of osmotically dehydrated tissue was extracted after smashing the pepper samples in a blender. The extract was centrifuged with a Dynac centrifuge (Becton, Dickinson and Company, Parsippany, NJ) at 1150 rpm for 10 min. The supernatant was then used for sorbitol determinations.

An HPLC system with an Aminex HPX-87H (300 × 7.8 mm I.D.) column (Biorad Laboratories, Hercules, CA) was used for sorbitol analysis. Column temperature was maintained at 70 °C. The sample was eluted using a mobile phase of 0.05 N H_2SO_4 at a flow rate of 0.4 ml/min. A refractive index detector (model 156, Beckman Industries, Inc., San Ramos, CA) and a Linea recorder–integrator (model 1200, Graphic Controls, Buffalo, NY) were used for quantification.

Table 4
Analysis of variance for the overall effect of the four factors on the four responses

Input process variables	Sum of squares			
	Water loss (WL)	Solids gain (SG)	Salt uptake (SA)	Sorbitol uptake (SO)
Salt concentration	205.98**	37.79*	211.35*	0.54
Sorbitol concentration	58.64	8.21*	7.54**	10.91*
Temperature	5.32	1.08	2.04	0.14
Time	349.63*	20.31*	43.47*	5.10*

*Significant at $p \leq 0.01$.
**Significant at $p \leq 0.05$.

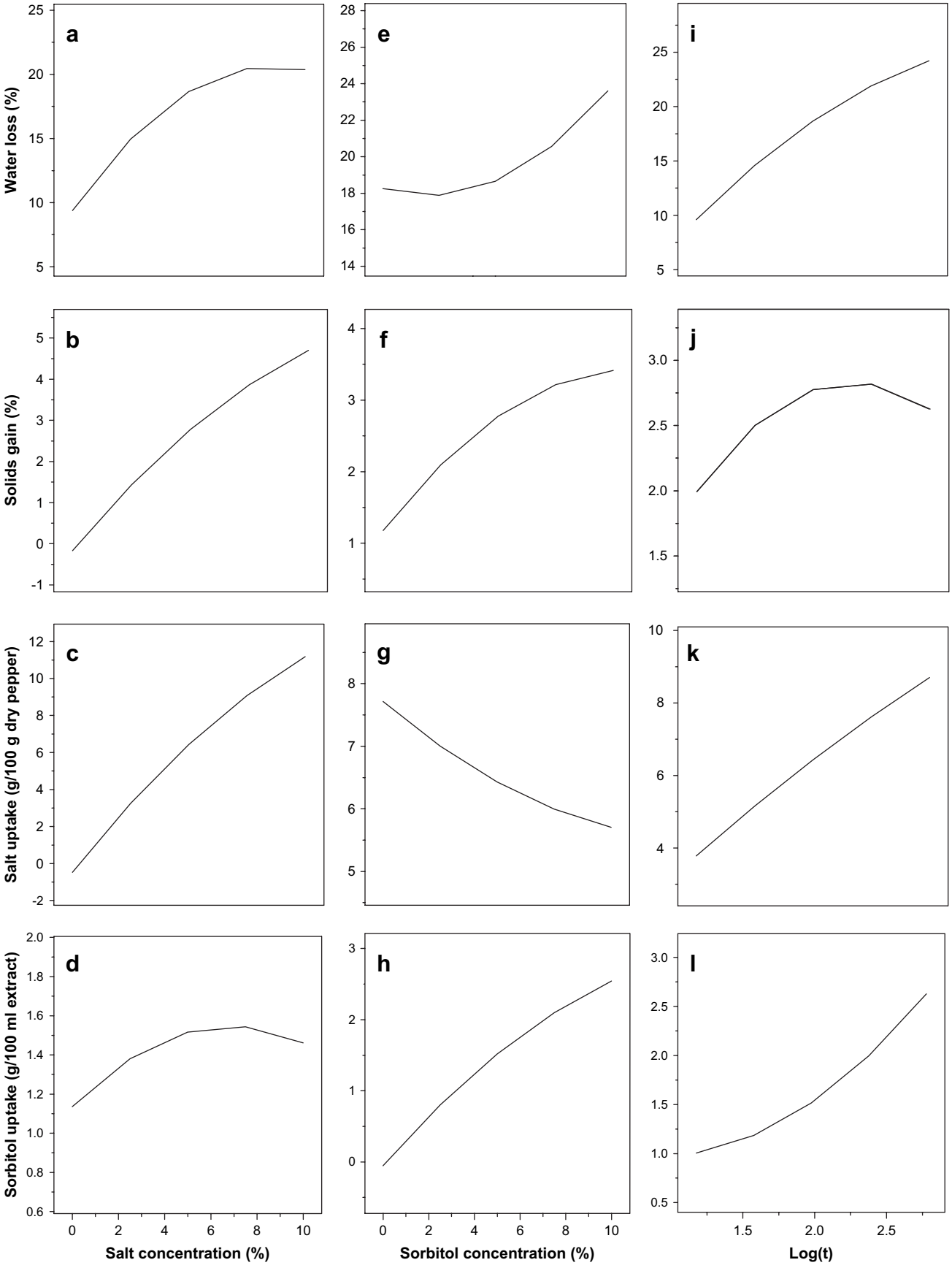

Fig. 1. Effects of significant factors on all four responses.

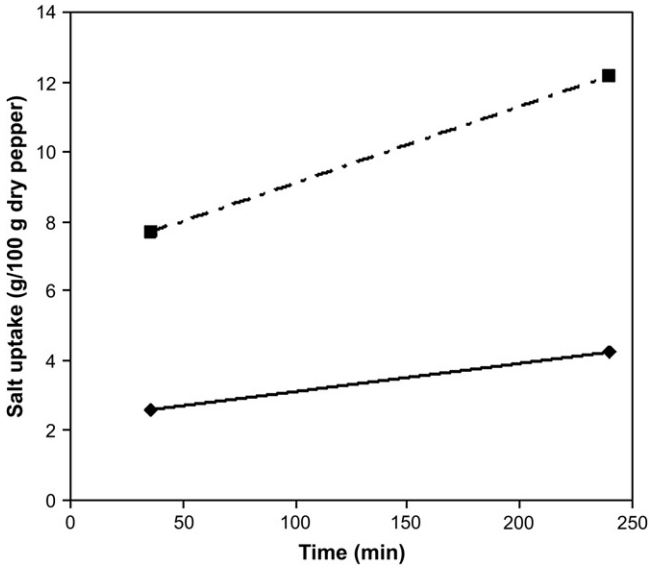

Fig. 2. Salt uptake vs. time plot at low (2.5 g/100 g) and high (7.5 g/100 g) salt concentrations while keeping the sorbitol concentration constant at 2.5 g/100 g.

3. Results and discussion

Four models were obtained as a result of fitting Eq. (2) to experimental data shown in Table 1. These models were tested for adequacy and fitness by analysis of variance (Table 2). Results of this analysis showed that the models developed for all four responses (WL, SG, SA and SO) were significant with no significant lack of fit suggesting that they adequately represented the relationship between responses and factors. The model coefficients (β's) are given in Table 3.

A joint test was performed to determine the overall effect of the four factors on all responses (Table 4). This analysis tested the hypothesis that all parameters involving one particular factor are zero. Results indicated that processing time was the most important factor because it was significant for all four responses ($p \leq 0.01$). As expected, salt significantly affected SG and SA ($p \leq 0.01$) as well as WL ($p \leq 0.05$), but had no effect on sorbitol uptake. Sorbitol influenced SG and SO at $p \leq 0.01$, and SA at $p \leq 0.05$, but surprisingly did not have a significant effect on WL. Processing temperature within the temperature range tested (20–40 °C) was found to be statistically not

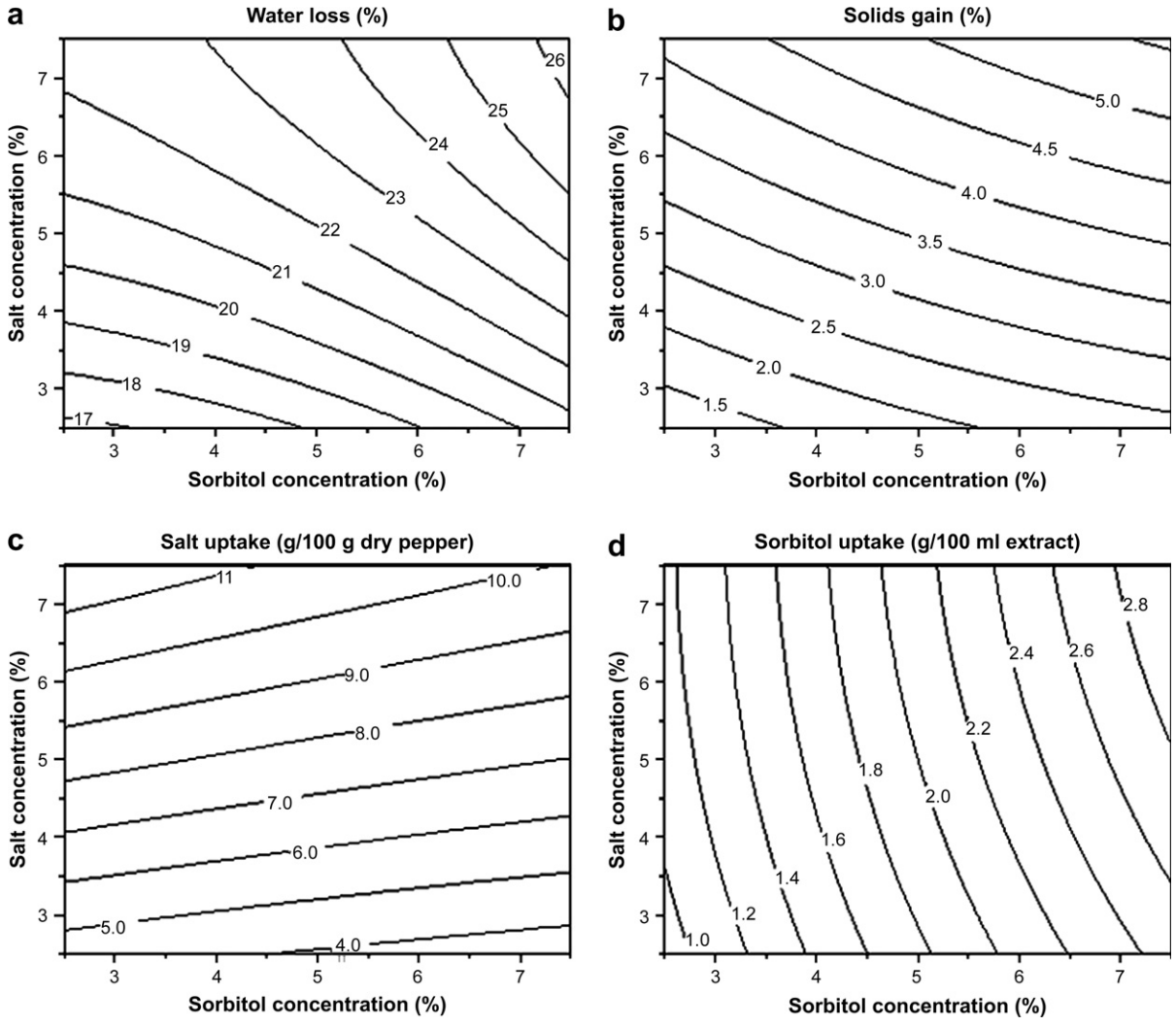

Fig. 3. Contour plots of equal response values for WL, SG, SA, and SO (all plots were generated for constant temperature = 30 °C and time = 240 min).

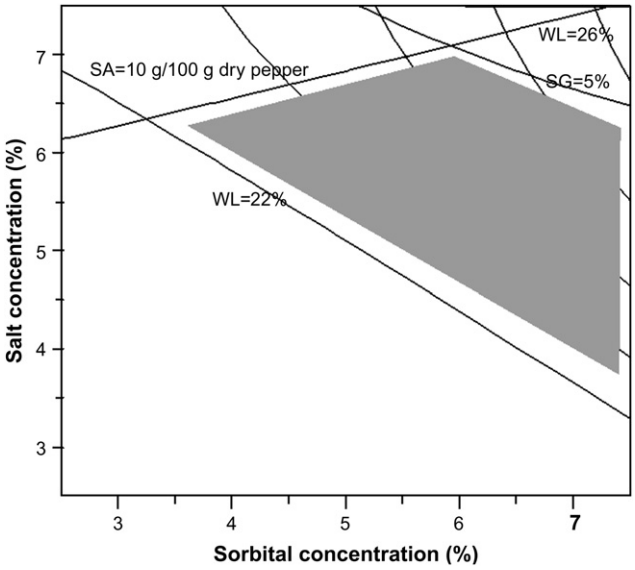

Fig. 4. Optimum region generated for constant temperature = 30 °C and time = 240 min by superimposing contour plots of all four responses WL, SG, SA, and SO.

significant. Collignan and Raoult-Wack (1994) also observed that the effect of temperature on WL was small during OD of cod at a temperature range of 0–40 °C. Low processing temperatures reduce energy requirements and help maintain better organoleptic properties while higher temperatures effectively increase mass transfer rates (Lenart & Flink, 1984; Quintero-Ramos et al., 1993). On the other hand, higher temperatures (>45 °C) lead to enzymatic browning and flavor deterioration in fruits and vegetables (Torreggiani, 1993).

Each response was plotted against each significant factor using the predicted models to better understand the relationship between factors and responses (Fig. 1). As shown, WL increased as salt concentration increased from 0 to 7.5 g/100 g and then leveled off (Fig. 1a). SG (Fig. 1b) and SA (Fig. 1c) increased fairly linearly with increasing salt concentration. SG also went up fairly linearly up to a sorbitol concentration of 7.5 g/100 g, but then an increase in sorbitol concentration did not substantially affect SG (Fig. 1f). A slight decrease in SA was observed with increasing sorbitol concentration (Fig. 1g). Similar effects between sugar and salt were also observed by Collignan and Raoult-Wack (1994). They reported that sugar can hinder the entrance of salt into cod tissue during its dewatering in concentrated sugar and salt solutions. This behavior can be explained by the formation of a concentration gradient around the tissue due to sorbitol, which hinders the entrance of salt into the product. SO was only affected by sorbitol concentration (Fig. 1h) and time (Fig. 1l) in which an SO of 2.5 g/100 ml extract was attained at 10 g/100 g sorbitol solution, while SO reached 2.8 g/100 ml extract at the end of the OD. A nearly linear rise was observed in WL (Fig. 1i) and SA (Fig. 1k) with increasing time. On the other hand, SG increased in the early stages, remained almost constant for a short period of time and finally showed a decreasing trend (Fig. 1j). This declining trend in SG at the end of the process could be attributed to the loss of some original solids in diced peppers.

As given in Table 2, cross product terms for all four responses were not significant except SA. Further analysis showed that the interaction between salt concentration and time significantly affected SA (Table 3). Therefore, SA vs. time plot (Fig. 2) was drawn at low (2.5 g/100 g) and high (7.5 g/100 g) salt concentrations to investigate the interaction between salt concentration and time while keeping the sorbitol concentration constant at 2.5 g/100 g. This plot showed that increase in SA with increasing time at high salt concentration was steeper as compared to the increase in SA at low salt concentration, indicating the existence of a significant interaction between salt concentration and time.

As a result of examination of the system by canonical analysis, all stationary points were determined as saddle (mini–max) points. Thus, graphical multi-response optimization techniques (Floros & Chinnan, 1988) and computer-generated contour plots were used rather than analytical optimization techniques. WL was considered as the most important response, and because temperature had no significant effect on any response, it was kept constant at 30 °C. A processing time of 240 min was chosen because the change in WL after that time (between 240 and 600 min) was not very significant. To locate the optimum processing conditions, contour plots for WL, SG, SA and SO were generated at 30 °C and 240 min of processing time (Fig. 3). WL changed from 17 to 26% with increasing salt and sorbitol concentrations from 2.5 to 7.5 g/100 g (Fig. 3a). At low sorbitol concentrations, SO by the tissue was only 1 g/100 ml extract, but this value reached 2.8 g/100 ml extract when the sorbitol concentration was increased to 7.5 g/100 g (Fig. 3b). High salt and sorbitol concentrations yielded maximum SG of 5.5% (Fig. 3c). Maximum SA took place at high salt and low sorbitol concentrations (Fig. 3d).

An optimum process represents conditions, which would result in maximum WL, possible minimum SG, SA $\leq$ 10 g/100 g dry pepper and SO $\leq$ 3 g/100 ml extract (Ozen et al., 2002). Based on these constraints and by superimposing the computer-generated contour plots for WL, SG, SA and SO (Fig. 3), an optimum region (shaded area) was obtained (Fig. 4). The predicted optimum conditions of salt 5.5 g/100 g and sorbitol 6 g/100 g at 30 °C for 240 min of processing time would result in WL of 23.3%, SG = 4.1%, SA = 8 g/100 g dry pepper and SO = 2.4 g/100 ml extract.

4. Conclusions

Response surface methodology and graphical optimization methods were effective in locating optimum processing conditions for an osmotic dehydration of diced green peppers. The processing temperature within the temperature range tested (20–40 °C) was not an important factor. The use of sorbitol could be useful from an organoleptic point of view, because it hindered the entrance of salt into the product. This could result in dehydrated peppers with low salt content. The OD of diced green peppers at optimum processing conditions of 30 °C for 240 min in an osmotic solution containing salt (5.5 g/100 g) and sorbitol (6 g/100 g) would reduce the original water content of the product by about 23.3%. Therefore,

the OD of green peppers could be effectively used as a pretreatment prior to freezing or air drying to reduce energy demands of these processes.

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