OPTIMIZATION OF THE OSMOTIC DEHYDRATION OF CARROT CUBES IN SUGAR BEET MOLASSES

by

Nevena M. MIŠLJENOVIĆ[a], Gordana B. KOPRIVICA[a], Lato L. PEZO[b*], Ljubinko B. LEVIĆ[a], Biljana Lj. ĆURČIĆ[a], Vladimir S. FILIPOVIĆ[a], Milica R. NIĆETIN[a]

[a] University of Novi Sad, Faculty of Technology, Bulevar Cara Lazara 1, 21000 Novi Sad, Serbia

[b] University of Belgrade, Instutute of General and Physical Chemistry, Studentski Trg 12 - 16, 11000 Belgrade, Serbia

[*]Corresponding author (L. Pezo): e-mail: latopezo@yahoo.co.uk

A Response Surface Methodology approach (RSM) was used to determine optimum conditions for the osmotic dehydration of carrot cubes in sugar beet molasses. Treatment times were set to 1, 3 and 5 h, at temperatures of 45, 55 and 65°C and molasses concentrations were 40, 60 and 80% (w/w). The used responses variables were: final dry matter content (DM), water loss (WL), solid gain (Sg), and water activity (a_w). A Box and Behnken's fractional factorial design (2 level-3 parameter) with 15 runs (1 block) was used for design of the experiment. DM, WL, Sg were significantly affected by all process variables (at 90-95% confidence level).

The optimum conditions were determined by superimposing the contour plots, with the following response limiting values: DM 50-60%, WL 0.7-0.8, Sg 0.08-0.09, and a_w 0.84-0.86. The optimum conditions generated were: treatment time of 4h, temperature of 60°C, sugar concentration of 66% (w/w).

Key words: *Osmotic dehydration; Carrot cubes; Sugar beet molasses; Response surface methodology; Optimization*

1. Introduction

The main purpose of post-harvest handling of agricultural products is to maintain its quality and the reduction of losses, due to organic decomposition. Processing of fruits, as well as preservation, are necessary for the production, drying is one of the oldest, also least expensive techniques developed for that purpose.

Many studies are concerned with recent developments of new drying methods and techniques, that are continually developing, and the ultimate aim is to keep the initial characteristics of final product (such as vitamins content, natural color and flavors, essential amino acids content). Osmotic dehydration is an environmentally acceptable, material gentle drying method, which received considerable attention because of the low processing temperature, base waste material and low energy requirement [1, 2].

Osmotic dehydration is a water removal process, based on soaking foods (fruit, vegetable, meat and fish) in a hypertonic solution. Water removal in liquid form, usage of mild temperatures and osmotic solution reusing are main advantages of osmotic dehydration process in comparison with other drying treatments [3, 4].

Several factors, such as concentration of osmotic solution, processing temperature and time, agitation, material to solution ratio and raw material characteristics have a high impact on the osmotic dehydration process [5, 6, 7, 8].

The type of osmotic solution also plays an important role in the process, with salt solutions used for vegetables and sugar solution and syrups used for fruits. [9, 1]. Recent research has shown that use of sugar beet molasses as hypertonic solution improves osmotic dehydration processes [10]. Sugar beet molasses is an excellent medium for osmotic dehydration, primarily due to the high dry matter (80%) and specific nutrient content. From nutrient point of view, an important advantage of sugar beet molasses, as hypertonic solution, is enrichment of the food material in minerals and vitamins, which penetrate from molasses to the plant tissue [11, 12].

Further studies were focused on the optimization of osmotic dehydration process, in order to develop rapid and effective removal of water using high molecular weight osmotic agents, as much as possible low temperatures and treatment times that could make the process efficient and practical [1, 2].

Response surface methodology (RSM) is an effective tool for optimizing a variety of food processes including osmotic dehydration [13, 14, 15]. The main advantage of RSM is reduced number of experimental runs that provide sufficient information for statistically valid results. The RSM equations describe effects of the test variables on the observed responses, determine test variables interrelationships and represent the combined effect of all test variables in the observed responses, enabling the experimenter to make efficient exploration of the process.

The objectives of here presented article were to investigate the effects of temperature, processing time and concentration on the mass transfer phenomena during osmotic dehydration of carrot cubes in sugar beet molasses solutions, to model *DM*, *WL*, *Sg* and a_w as a function of the process variables and to find the optimum osmotic dehydration conditions.

2. Materials and methods

2.1. Material

Carrot samples were purchased in a local market in Novi Sad (Serbia) and stored at 4°*C* until use. Initial moisture content, X_o, was 89.65 ± 0.48 %.

Sugar beet molasses was obtained from the sugar factory Pećinci, Serbia. Initial dry matter content in sugar beet molasses was 83.68%. For dilution of sugar beet molasses distilled water was used.

2.2. Osmotic dehydration

The carrot samples were washed thoroughly and peeled manually (using a stainless kitchen peeler). The peeled carrots were manually sliced into cubes, dimension 1x1x1 cm using a kitchen slicer. The amount of 100 g of sliced carrot cubes samples were prepared for each treatment. Different concentrations of sugar beet molasses (40.0%, 60.0% and 80.0% dry matter) were used as osmotic solution. The effect of temperature was also investigated and the experiments were conducted at temperatures of 45, 55 and 65ºC (tab. 1).

Table 1. Coded values of the treatment variables

	Treatment variables	Coded values		
		-1	0	+1
X_1	Temperature (^oC)	45	55	65
X_2	Time (h)	1	3	5
X_3	Concentration (%)	40	60	80

The carrot cubes were put in a glass jars with 1000 g of molasses solution with a material/solution ratio of 1:10 (w/w). The jars were placed in the heat chamber and process was performed without agitation. After each sampling time (1, 3 and 5 hours), which is determined according to the experimental design (tab. 2), the carrot cubes were taken out, washed with water and gently blotted with filter paper in order to remove the excessive water and weighed. Dry matter content of the fresh and treated samples was determined by drying the material at 105 ºC for 24h in a heat chamber (Instrumentaria Sutjeska, Serbia). Water activity (a_w) of the osmotic dehydrated samples was measured using a water activity measurement device (TESTO 650, Germany) with an accuracy of ±0.001 at 25ºC. Soluble solids content of the molasses solutions was measured using Abbe refractometer, Carl Zeis Jenna at 20 ºC.

Table 2. Experimental design and data for the response surface analysis

Run. No	X_1 Temp.	X_2 Time	X_3 Conc.	Y_1 *DM*	Y_2 *WL*	Y_3 *Sg*	Y_4 a_w
1	+1	-1	0	34.87	0.5841	0.0750	0.917
2	+1	0	+1	59.84	0.7916	0.0874	0.813
3	-1	-1	0	24.37	0.4323	0.0525	0.920
4	0	+1	-1	36.44	0.5368	0.0540	0.906
5	-1	0	-1	30.93	0.4953	0.0531	0.900
6	-1	+1	0	47.17	0.7042	0.0755	0.891
7	0	-1	+1	27.03	0.5356	0.0655	0.930
8	0	+1	+1	64.66	0.8217	0.1006	0.785
9	0	-1	-1	22.49	0.3147	0.0401	0.948
10	-1	0	+1	39.46	0.6686	0.0832	0.901
11	+1	+1	0	60.81	0.7818	0.0932	0.805
12	0	0	0	43.96	0.6740	0.0840	0.880

13	0	0	0	44.00	0.6710	0.0900	0.850
14	+1	0	-1	36.73	0.5896	0.0643	0.912
15	0	0	0	43.57	0.6820	0.0750	0.920

Evaluation of mass exchange between the solution and the sample during osmotic dehydration were made by using the parameters such as *DM*, *WL* and *Sg*. In order to account for initial weight differences between the samples *WL* and *Sg* were calculated acording to the following equations:

$$WL = \frac{m_i z_i - m_f z_f}{m_i} \quad \left[\frac{g}{g \;\; fresh\, sample} \right] \tag{1}$$

$$Sg = \frac{m_f s_f - m_i s_i}{m_i} \quad \left[\frac{g}{g \;\; fresh\, sample} \right] \tag{2}$$

where m_i and m_f are the initial and final weight (g) of the samples, respectively; z_i and z_f are the initial and final mass fraction of water (g water/ g sample), respectively; s_i and s_f are the initial and final mass fraction of total solids (g total solids/ g sample), respectively.

2.3. Response surface methodology

The RSM method was selected to estimate the main effect of the process variables on mass transfer variables and a_w, during the osmotic dehydration of carrot cubes. The accepted experimental design was taken from Box *et al*. [16]. The independent variables were temperature (X_1) of 45, 55 and 65°C; osmotic time (X_2) of 1, 3 and 5h; molasses concentration (X_3) of 40, 60 and 80% (by weight), and the dependent variables observed were the response: *DM* (Y_1), *WL* (Y_2), *Sg* (Y_3), and a_w (Y_4). Each of the variables and responses were coded, as shown in tab. 1. The variables osmotic temperature, treatment time, and solution concentration were coded as X_1, X_2, and X_3, respectively and the responses *DM*, *WL*, *Sg* and a_w as Y_1, Y_2, Y_3, and Y_4. The design included 15 experiments with 3 replications of the center point.

A model was fitted to the response surface generated by the experiment.
The model used was function of the variables:

$$Y_k = f_k\left(temp., time, conc.\right) \tag{3}$$

The following second order polynomial (SOP) model was fitted to the data. Four models of the following form were developed to relate four responses (Y) such as DM , WL, Sg and a_w to four process variables (X):

$$Y_k = \beta_{k0} + \sum_{i=1}^{4} \beta_{ki} X_i + \sum_{i=1}^{4} \beta_{kii} X_i^2 + \sum_{i=1}^{4} \sum_{j=i+1}^{3} \beta_{kij} X_i X_j \tag{4}$$

where: β_{kn} are constant regression coefficients; Y, either DM (Y_1), WL (Y_2), Sg (Y_3) and a_w (Y_4); X_1, osmotic temperature; X_2 treatment time and X_3, solution concentration. The significant terms in the model were found analysis of variance (ANOVA) for each response.

2.4. Statistical analysis and verification of the experiments

Analysis of variance (ANOVA) and response surface regression method (RSM) were performed using StatSoft Statistica, for Windows, ver. 10 program. The model was obtained for each dependent variable (or response) where factors were rejected when their significance level was less than 90%. The same program was used for generation of graphs and contour plots.

The graphs of the responses with significant parameters were superimposed to determine optimum drying conditions, plotted on optimization graphic. After the optimum conditions were established, separate experiments were performed for model validations of the models.

3. Results and discussions

The study was conducted to determine the optimum osmotic dehydration conditions for sliced carrot cubes. The experimental data used for the analysis were derived using the Box and Behnken's fractional factorial (2 level-3 parameter) design, 1 block. Tab. 2 shows the response variables as a function of coded independent variables for the analysis.

The analysis of variance (ANOVA) tables exhibits the significant independent variables as well as interactions of these variables. In this article, ANOVA was conducted by StatSoft Statistica, ver. 10 to show the significant effects of independent variables to the responses and which of responses were significantly affected by the varying treatment combinations (tab. 3). *DM* was significantly affected by all process variables, temperature, treatment time and molasses concentration, at 95% confidence level. *WL* was also significantly affected by all process variables, at the same confidence level. Each of these responses was most affected by treatment time variable. Both *Sg* and a_w was significantly affected by temperature, treatment time and molasses concentration, but *Sg* reveal more sensitivity to the changes in molasses concentration. Water activity response was affected by temperature at 90% confidence level, as shown in tab. 3. Treatment time showed also greater influence on *Sg* and a_w.

Table 3. Analysis of variance for the overall effect of the three factors on the four responses

	Sum of squares			
Process Variables	*DM*	*WL*	*Sg*	a_w
Temperature (^oC)	316.513*	0,02493*	0,000386*	0,003403**
Time (h)	1258.013*	0,11951*	0,001017*	0,013448*
Concentration (%)	518.420*	0,09704*	0,001960*	0,007021*

*Significant at 95% confidence level, **Significant at 90% confidence level, ^{ns}Not significant

Tab. 4 shows the ANOVA calculation regarding response models developed when the experimental data was fitted to a response surface. The response surface used a second order polynomial in the form of eq. (4) in order to predict the function f_k (eq. (3)) for all dependent variables.

Table 4. Analysis of variance for the four responses

		Sum of Squares			
Source	df	Y_1	Y_2	Y_3	Y_4
Model	9	2362.41*	0.272951*	0.004055*	0.031026*
Linear	3	2092.95*	0.241496*	0.003363*	0.023872*
Quadratic	3	73.67*	0.028849*	0.000561**	0.000279 ^{ns}
Cross-product	3	195.79*	0.002606^{ns}	0.000130 ^{ns}	0.006875**
Lack of fit	3	1.69 ^{ns}	0.002333 ^{ns}	0.000129 ^{ns}	0.000496 ^{ns}
r^2		99.93	99.13	94.36	91.28

*Significant at 95% confidence level, **Significant at 90% confidence level, ^{ns}Not significant

The analysis revealed that the linear terms contributed substantially in all cases to generate a significant SOP model. The SOP models for all variables were found to be statistically significant and the response surfaces were fitted to these models. The linear terms of SOP model were found significant, at 95% confidence level, and their influence were found most important in model calculation. Quadratic and cross-product source influence *DM*, also at 95% confidence level. *WL* was significantly affected by quadratic term of SOP model, at 95% confidence level, while *Sg* was affected by this term at 90% confidence level. Also shown in tab. 4 is the residual variance where the lack of fit variation represents other contributions except for the first order terms. A significant lack of fit generally shows that the model failed to represent the data in the experimental domain at which points were not included in the regression [17]. All SOP models had insignificant lack of fit tests, which means that all the models represented the data satisfactorily.

The coefficient of determination, r^2, is defined as the ratio of the explained variation to the total variation and is explained by its magnitude [18]. It is also the proportion of the variability in the response variable, which is accounted for by the regression analysis [19, 20, 21]. A high r^2 is indicative that the variation was accounted and that the data fitted satisfactorily to the proposed model (SOP in this case). The r^2 values for *DM* (0.999), *WL* (0.991), *Sg* (0.943) and a_w (0.913) were very satisfactory and show the good fitting of the model to experimental results. Tab. 5 shows the regression coefficients for the response SOP models of *DM*, *WL*, *Sg* and a_w used by eq. (4) for predicting the values at optimum conditions. The contour plots developed from the approximating function of *DM* are shown from fig. 1, while fig. 2-4 show the contour plots for *WL*, Sg and a_w, respectively. The contour plot for *DM* showed a saddle point configuration, and its value raised to the upper right corner of the plot, with the increase of all process variables, temperature, treatment time and molasses concentration, which were consistent with literature values. [1, 2].

Table 5. Regression coefficients (based on coded data) of the SOP models for the four responses

	Y_1	Y_2	Y_3	Y_4
β_0	55.47417*	-0.251961^{ns}	-0.171998^{ns}	0.327979^{ns}
β_1	-1.70792*	-0.012490^{ns}	0.002495^{ns}	0.014508^{ns}
β_2	-0.17625^{ns}	0.188732*	0.012909^{ns}	0.067188^{ns}
β_3	-0.10588^{ns}	0.019252*	0.003866*	0.006250^{ns}
β_{12}	0.03925*	-0.000927^{ns}	-0.000060^{ns}	-0.001038^{ns}
β_{13}	0.01823*	0.000036^{ns}	-0.000009^{ns}	-0.000125^{ns}
β_{23}	0.14800*	0.000400^{ns}	0.000132^{ns}	-0.000644^{ns}
β_{11}	0.00965*	0.000170^{ns}	-0.000010^{ns}	-0.000054^{ns}
β_{22}	-0.76542*	-0.016767*	-0.001988^{ns}	0.001333^{ns}
β_{33}	-0.00782*	-0.000141*	-0.000025*	0.000009^{ns}

*Significant at 95% confidence level, **Significant at 90% confidence level, ^{ns}Not significant

Contour plots of both *WL* and *Sg* showed that maximum value was a bit lower then upper right corner of the plot, tending to grow with temperature and processing time. On the other hand, a_w (fig. 4) decreased with all process parameters. For the osmotic dehydration of carrot cubes in this study, the optimum conditions would have to be similar to the operating conditions from literature and meet the desired product specifications. The desired responses for the optimum drying conditions: *DM* of at least 50%, not greater than 60%, *WL* in the range of 0.7-0.8, *Sg* not greater than 0.09 and a_w in the range of 0.84 to 0.86.

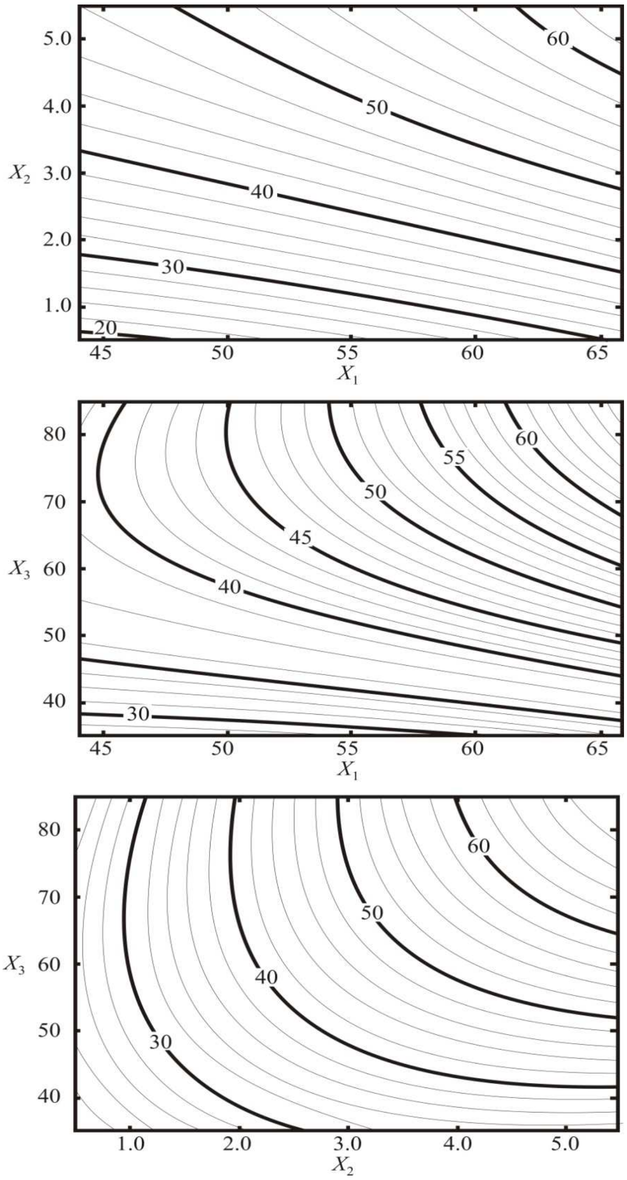


Figure 1. Contour plots for *DM* as function of temperature (x_1), time (x_2) and sugar concentration (x_3)

The contour plots (fig. 1-4) were superimposed to ascertain the optimum osmotic dehydration conditions for carrot cubes. Fig. 5 shows the superimposed graph of the dehydration conditions. An optimum operating area was derived and crosshatched and point A was deduced by approximating the optimum position in obtained area on each graph. Moving point A to the left of the obtained area, by decreasing osmotic temperature, would lead to the increase of time coordinate, and also molasses concentration, and translating this point to the right, would result in process temperature enhancement, while decreasing of processing time and molasses concentration. Optimization of the dehydration process is performed to ensure rapid processing conditions yielding an acceptable product quality and a high throughput capacity. The optimum osmotic drying conditions for carrot cubes are as follows: soaking time of 4 h, sugar solution concentration and temperature of 66% and 60°C. Deviating of point A (Fig. 5) to the right and upper corner will increase *DM* which is the most important response variable in osmotic dehydration as shown by the ANOVA, tab. 3, which will yield the optimum responses of osmotic system.

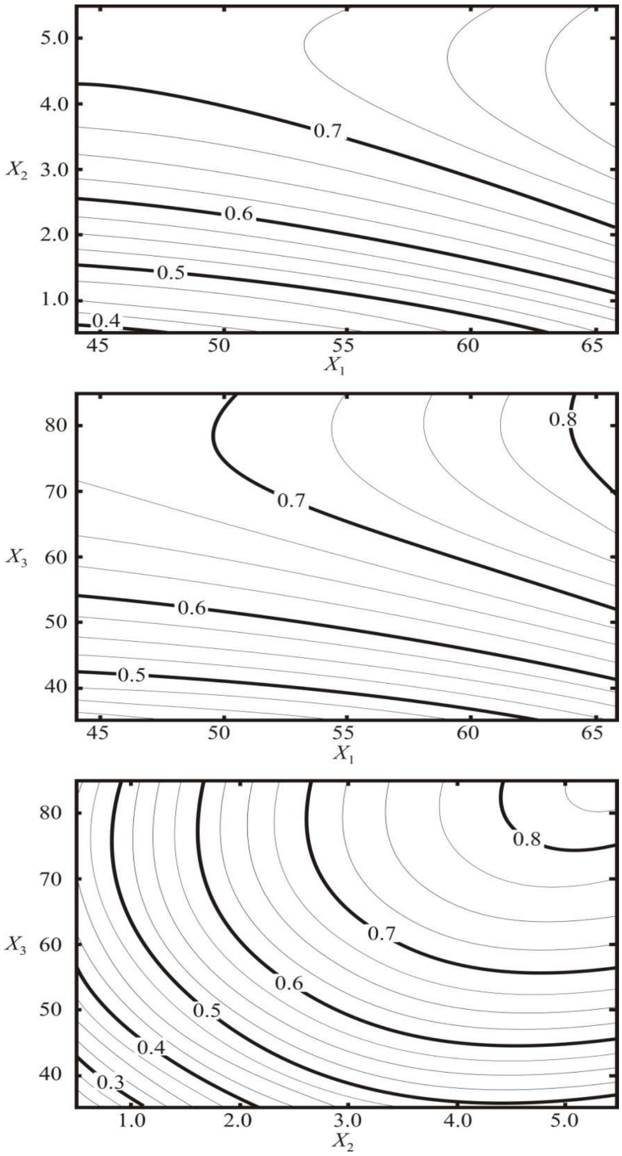


Figure 2. Contour plots for *WL* as function of temperature (x_1), time (x_2) and sugar concentration (x_3)

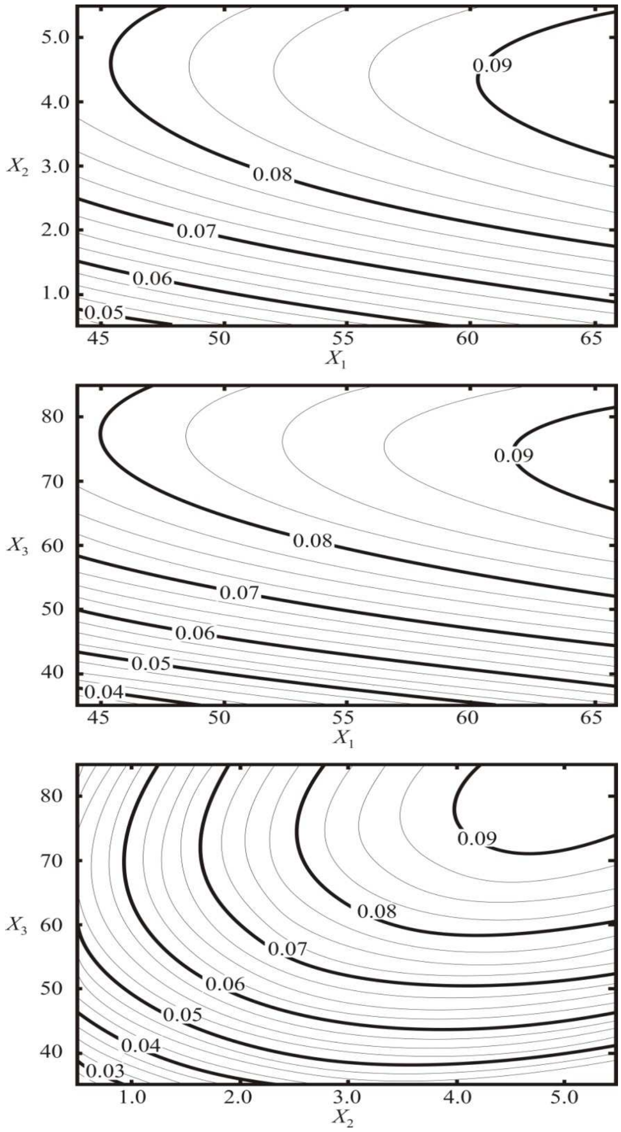


Figure 3. Contour plots for *Sg* as function of temperature (x_1), time (x_2) and sugar concentration (x_3)

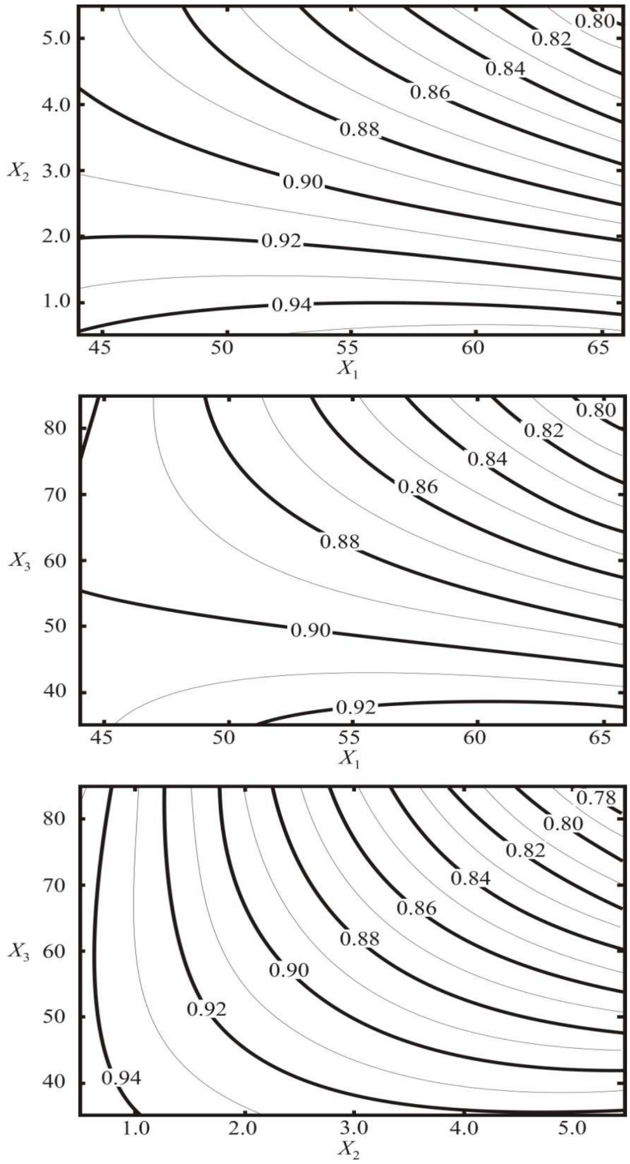


Figure 4. Contour plots for a_w as function of temperature (x_1), time (x_2) and sugar concentration (x_3)

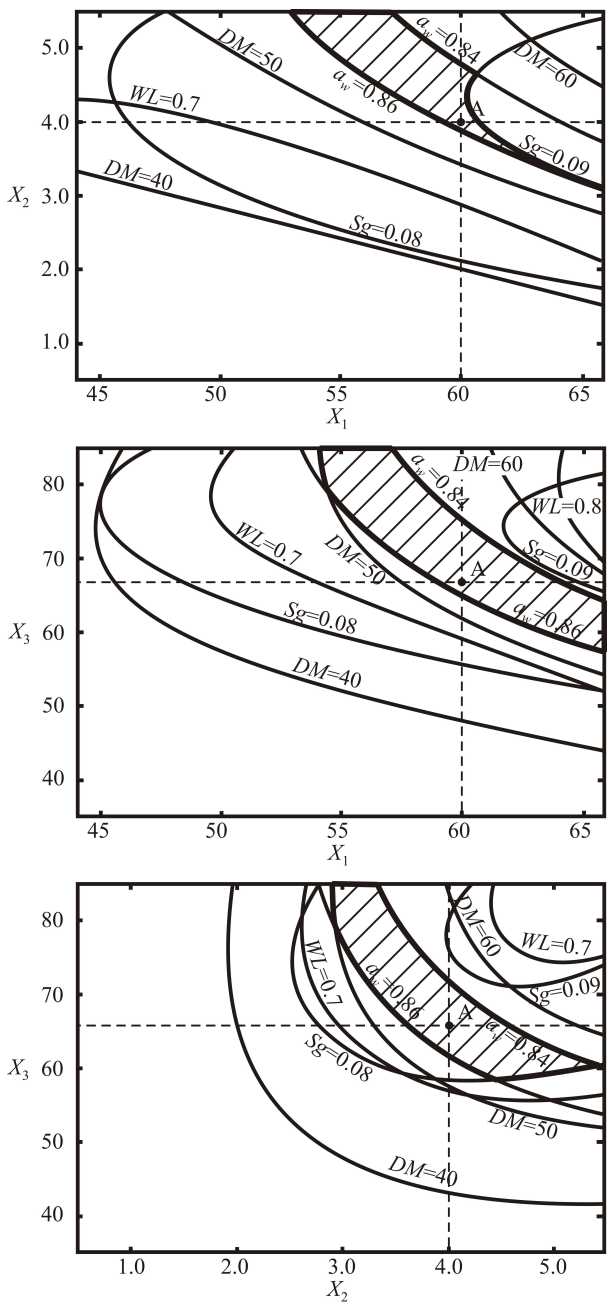
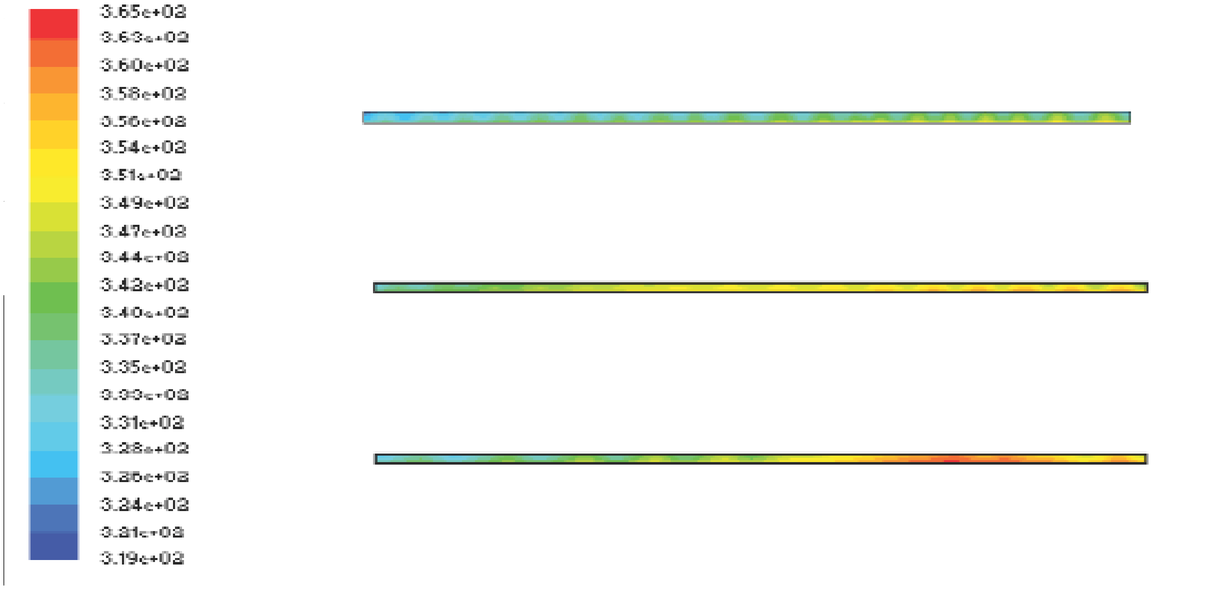


Figure 5. Optimum regions obtained after superimposing the contour plots of the responses

To determine the adequacy of the SOP models, independent experiments were performed at optimum conditions for validation [18]. Tab. 6 shows the model validation results. As shown in



4. Conclusions

The experimental data used for the optimization study were obtained using a Box and Behnken's fractional factorial design (2 level-3 parameter), 15 runs. The RSM algorithm was used to optimize the osmotic dehydration of carrot cubes utilizing *DM*, *WL*, *Sg*, and a_w as responses. SOP models for all system responses were statistically significant while predicted and observed responses correspond very well. The optimum dehydration process parameters were found by superimposition of the contour plots of all responses, for treatment time of 4 h, molasses beet solution concentration of 66% and temperature of 60°C were obtained for carrot cubes dehydration process. Predicted values of responses at optimum conditions were: *DM* of 50%, *WL* 0.74, *Sg* 0.09 and a_w 0.86.

ACKNOWLEDGEMENT: The authors acknowledge for the financial support the Ministry of Education and Science of the Republic of Serbia, TR – 31055, 2011-2014

5. Literature

[1] Panagiotou N. M., Karanthanos V. T., Maroulis Z. B., Effect of osmotic agent on osmotic dehydration of fruits, *Drying Technology, 17* (1999), pp. 175–189

[2] Waliszewiski K. N. *et al.*, Color parameter changes in banana slices during osmotic dehydration , *Drying Technology, 17* (1999), pp. 955-960

[3] Della Rosa M., Giroux F., Osmotic treatments (OT) and problems related to the solution management, *Journal of Food Engineering*, 49 (2001), pp. 223-236

[4] Torreggiani D., Osmotic dehydration in fruit and vegetable processing, *Food Research International*, *26* (1993), pp. 59-68

[5] Uddin M. B., Ainsworth P., İbanoğlu Ş., Evaluation of mass exchange during osmotic dehydration of carrots using response surface methodology, *Journal of Food Engineering, 65* (2004), pp. 473-477

[6] Ozen B. F. *et al.*, Processing factors affecting the osmotic dehydration of diced green peppers, *International Journal of Food Science and Technology, 37* (2002), pp. 497-502

[7] Mavroudis N. E., Gekas V., Sjoholm I., Osmotic dehydration of apples – effects of agitation and raw material characteristics, *Journal of Food Engineering, 35* (1998), pp. 191-209

[8] Lazarides H. N.: Reasons and possibilities to control solids uptake during osmotic treatment of fruits and vegetables, in: *Osmotic Dehydration & Vacuum Impregnation* (Ed. Fito P., Chiralt A., Barat J. M., Spiess W. E. L., Behsnilian D.), Technomic Publishing Company Inc., Lancaster, 2001, Chapter 4

[9] Lewicki P. P., Linart A., Osmotic Dehydration of Fruits and Vegetables, in: *Handbook of Industrial Drying 3rd edition* (Ed. By Mujumdar AS.) Taylor & Francis Group, LLC, Philadelphia, 2006, Chapter 28

[10] Mišljenović N., Koprivica G., Lević Lj., Comparison of the kinetics of osmotic drying apples in sugar beet molasses and sucrose solutions, *Journal of Processing of Energy and Agriculture, 14* (2010), pp. 32-35

[11] Koprivica G., Mišljenović N., Lević Lj., Kuljanin T., Influence of nutrients present in sugar beet molasses and saccharose solution on the quality of osmodehydrated carrot, *Journal of Processing of Energy and Agriculture, 13* (2009), pp. 178-180

[12] Filipčev B. *et al*., Quality characteristics and antioxidant properties of breads supplemented with sugar beet molasses-based ingredients, *International Journal of Food Properties*, *13* (2010), pp**.** 1035-1053

[13] Azoubel P. M., Murr F. E. X., Optimization of the osmotic dehydration of cashew apple (*Anacardium occidentale* L.) in sugar solutions, *Food Science and Technology International*, *9* (2003), pp. 427-433

[14] Ozdemir M. *et al.*, Optimization of the osmotic dehydration of diced green peppers by response surface methodology, *LWT-Food Science and Technology, 41* (2008), pp**.** 2044-2050

[15] Singh B. *et al*., Optimisation of osmotic dehydration process of carrot cubes in mixtures of sucrose and sodium chloride solutions, *Food Chemistry*, *123* (2010), , pp. 590-600

[16] Box G. E. P., Behnken D. W., Some new three level designs for the study of quantitative variables, *Technometrics, 2* (1960), pp**.**455–475

[17] Montgomery D. C., Design and Analysis of Experiments, 2nd edition, John Wiley and Sons Inc., New York, USA, 1984.

[18] Madamba P. S.,The response surface methodology: an application to optimize dehydration operations of selected agricultural crops, *LWT-Food Science and Technology, 35* (2002), pp. 584-592

[19] McLaren, C. G *et al*., Experimental Design and Data Analysis for Agricultural Research, Book of abstracts, *1 IRRI*, Los Banos, Laguna, Philippines, 1997

[20] Kosoy B. V., Microchannels in macro thermal management solutions, *Thermal science*, *10* (2006), 1, pp. 81-98

[21] Zheleva I., Kambourova V., Identification of heat and mass transfer processes in bread during baking, *Thermal science*, *9* (2005) 2, pp. 73-86