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Optimization of osmotic dehydration parameters for sweet cherries (*Prunus avium*) using response surface methodology

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Osmotic dehydration of foods has recently gained attention as a processing method for obtaining better quality fruit products. The objective of this study was to investigate the effect of temperature of osmotic treatment (43, 50, 60, 70, and 77°C), concentration of osmotic solution (47, 50, 55, 60, and 63°Brix) and fruit-to-solution ratio (1:2, 1:3, 1:4, 1:5, and 1:6 w/w) on water loss (WL), solid gain (SG), and total antioxidant capacity (TAC) of osmotically dehydrated sweet cherries and to perform optimization of technological parameters by response surface methodology (RSM). The optimized criteria yielded high values of water loss, solid gain, and total antioxidant capacity.

Keywords: optimization, osmotic dehydration, sweet cherries, response surface methodology

Abbreviations:

OD – osmotic dehydration
WL – water loss
SG – solid gain
TAC – total antioxidant capacity
RSM – response surface methodology

INTRODUCTION

Cherries are one of the most popular early summer fruits. They comprise about 30 species, of which the most popular edible types are sweet cherry (*Prunus avium*), tart (sour) cherry (*Prunus cerasus*), and duke cherry. Cherries could be yellow, red, or yellow-red colored and are consumed fresh, dried, or processed. Sweet cherry fruits contain carbohydrates (12 – 26%), organic acids (0.3 – 0.8%), water-soluble vitamins (vitamin C, B vitamins) and fat-soluble vitamins (vitamins A, E and K), and some carotenoids [1, 2]. They are a good source of polyphenols (44 – 87 mg gallic acid equivalents/ 100 g) [1], anthocyanins (70 – 100 mg cyanidin 3-glucoside equivalents /100 g) [3], and flavonols (3 – 5 mg /100 g) [4]. The listed phytochemicals have been shown to exhibit high antioxidant capacity and to play a pivotal role in cell protection from reactive oxygen species (ROS) produced in the human body. Therefore, consumption of these fruits can reduce the risk of diseases such as cancer [5], arthritis, inflammation [6], and neurodegenerative diseases [7]. In the last few decades, sweet cherries are used for manufacturing health-promoting juices, syrups, jams, dried, and dietary foods, where preservation of antioxidants during cherry processing is of key importance.

Osmotic dehydration (OD) is one of the perspective pre-treatments for manufacturing dried fruit products, since it offers a number of benefits such as reducing the heat degradation of biologically active phytochemicals, color retention, reduction of fruit browning and decrease of the energy costs [8]. Osmotic dehydration often precedes processes such as air drying, freeze drying, or vacuum drying.

Osmotic dehydration is based on partial removal of water from plant tissues by immersion of foodstuff in a hypertonic water solution. It involves three simultaneous mass transfer flows (Fig. 1). The first one is water removal from the plant tissues into the osmotic solution, the second is the diffusion of osmotic agent from the osmotic solution into the plant cells and the third mass transfer flow is excretion of plant compounds (organic acids, mineral salts and vitamins) from the tissues to the osmotic solution. Although this third flow is not significant in the mass exchange, it is essential for the chemical composition and organoleptic qualities of the products [9].

Several factors are responsible for osmotic process efficiency, including type and concentration of the osmotic agent, temperature of the osmotic treatment, fruit : osmotic solution ratio and process duration.

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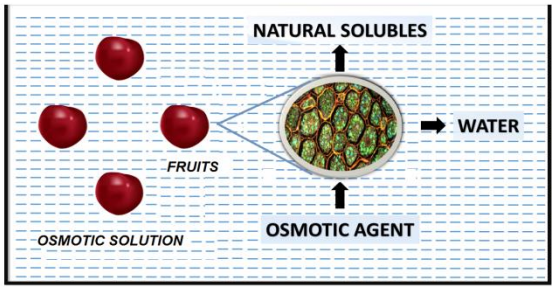

Figure 1. Mass transfer in fruit tissue during osmotic dehydration

Improvement of the dehydration rate is very important, since osmotic drying is a rather slow process. The type of the osmotic agent is a very important factor that determines the rate of diffusion. The most commonly used osmotic agents for the preparation of osmotic solutions are sucrose, glucose, fructose, sorbitol, corn syrup, and fructo-oligosaccharides. A number of scientists have concluded that osmotic agents with low molecular weight pass easier into the fruit cells membrane compared to osmotic agents with high molecular weight [10, 11]. In osmotic treatments, the increase of the osmotic agent concentration leads to an increased rate of dehydration. The study of Falade *et al.* (2007) [12] on the osmotic dehydration of watermelon in solutions with 40 – 60°Brix confirmed that the increase in the concentration of the osmotic agent (sucrose) resulted in higher water loss and solid gain throughout the osmotic period. The temperature of osmotic dehydration is one of the most significant variables affecting the kinetics of mass transfer [13]. According to the concept of osmotic dehydration, the increase of the solution temperature results in increased water loss, weight reduction and uptake of solids. The effect of osmotic treatment temperature on the osmotic process efficiency is proved by a number of scientists [14, 15]. It was also found that the increase of fruit : osmotic solution ratio results in an increase in water loss, solid gain and rate of dehydration [16].

Response surface methodology (RSM) is a statistical tool used to compose experiments for evaluation of the effects of independent process variables on dependent variables, as well as to determine optimal process conditions. The advantages of RSM are: reduced number of experiments, evaluation of the relative significance of several affecting factors even in the presence of complex interactions, and improved statistical interpretation. Several reported studies on optimization of plant products dehydration by the RSM method show its efficacy [17].

Therefore, the objective of this study was to investigate the effect of independent process variables (temperature of osmotic treatment, concentration of osmotic solution, and fruit-to-solution ratio on dependent variables (water loss, solid gain, and total antioxidant capacity) of osmotically dehydrated sweet cherries and to perform optimization of the technological parameters by response surface methodology.

MATERIALS AND METHODS

Raw materials

Sweet cherry (*Prunus avium*) fruits with soluble solids of 15.40% were purchased from Degustos Ltd. (Bulgaria). The fresh fruits were sorted and stored in a refrigerator at 3°C until used. Osmotic agents - concentrated sour cherry juice, concentrated apple juice, and inulin (oligo-fructose - 87%; average degree of polymerization - 8; sum of sucrose, glucose, and fructose - 12%) were purchased from Krichimfrukt Ltd. (Bulgaria), Agrobiotech Ltd. (Bulgaria), and Food Consulting Ltd. (Bulgaria), respectively. The concentrated sour cherry juice and apple juice were stored in a refrigerator at 3°C until used.

Chemicals

1,1-Diphenyl-2-picrylhydrazyl radical (DPPH), (±)-6-hydroxy-2,5,7,8-tetramethyl-chromane-2-carboxylic acid (Trolox), Folin-Ciocalteu phenol reagent, and gallic acid Sigma-Aldrich (St. Louis, MO, USA) were used as analytical standards. Methanol, ethanol, concentrated hydrochloric acid (HCl) and sodium carbonate monohydrate ($Na_2CO_3 \cdot H_2O$) of analytical grade were purchased from Rai-Him, Bulgaria.

Methods

Sample preparation and osmotic process. The sweet cherries were washed with tap water and the stones were removed.

Osmotic solutions were prepared in five concentrations (47, 50, 55, 60 and 63°Brix) (Table 1) using concentrated sour cherry juice with 63°Brix (60% w:w), concentrated apple juice with 72°Brix (20% w:w), and inulin (20% w:w). The concentration of the osmotic solutions was monitored by an Abbe refractometer (VEB Carl Zeiss Jena, Germany).

Osmotic dehydration of cherries was performed in a water bath (VEB MLW Prüfgerätewerk, Medingen, Sitz Freital, Germany). The choice of the process conditions was based on a literature survey on osmotic dehydration. The fruits were kept in an osmotic solution with concentration from 47 to 63°Brix and temperature from 43 to 77°C for 4 hours according to the experimental design (Table 1). The

fruit : solution ratio was 1:2, 1:3, 1:4, 1:5, and 1:6 (w:w) (Table 1). Further, the osmotically dehydrated cherries were removed from the solutions, quickly rinsed with hot water (40°C) and gently dried with paper towel to remove surface moisture. Total dry matter and drained weight (final sample weight) of the osmotically dehydrated fruits were determined. The fruits were also analyzed for the mass transfer indicators (water loss, solid gain) and total antioxidant capacity.

Water loss (WL) is defined as the net loss of water from the fresh fruits after osmotic dehydration based on the initial sample.

Solid gain (SG) is defined as the net uptake of sugar by the osmotically dehydrated fruits based on the initial sample weight.

WL and *SG* were calculated according to the following equations [18]:

$$WL = \frac{x_o^w M_o^o - x_f^w M_f^o}{M_o^o} 100\,,\% \quad + \qquad (1)$$

$$SG = \frac{x_f^{st} M_f^o - x_o^{st} M_o^o}{M_o^o} 100,\% \qquad (2)$$

where: x_o^w – initial moisture content (%), x_f^w – final moisture content (%), M_o^o – initial sample weight (kg), M_f^o – final sample weight (kg), x_o^{st} – initial solids content (%), x_f^{st} – final solids content (%).

The moisture content in the fruits and the concentrated juices was determined according to BCS EN 12143:2000 and BCS EN 12145:2000.

Extract preparation. The osmotically dehydrated sweet cherries (5.00 g) were mixed with acidified methanol HCl (1000 ml MeOH with 2.3 ml conc. HCl) in a 50 ml volumetric flask. After 12 hours in a refrigerator at 3°C, the extracts were filtered through filter paper and transferred into flasks.

Total antioxidant capacity (*TAC*) was determined by the DPPH assay (free radical scavenging activity).

The ability of the fruit extract to interact with free radicals (scavenger again DPPH•) was determined by the colorimetric method of Brand-Williams *et al.* (1995) [19]. A 2250 µl aliquot of DPPH - ethanol solution (2.4 mg DPPH in 100 cm^3 ethanol) was mixed with 250 µL of methanol extract. The samples were incubated in a dark cabinet at room temperature. The change in absorbance after 15 minutes was measured at 515 nm by a spectrophotometer (UV-Vis Thermo Fisher Scientific, Madison, WI, USA). The standard curve for the method was created with ethanol solutions of Trolox in a concentration range between 100 and 1000 µmol per 100 ml. The total antioxidant capacity (TAC) was expressed as µmol Trolox equivalent per 100 g of samples on dry weight basis (dw). All determinations were performed in triplicate (n = 3).

Experimental design and statistical analysis. Response surface methodology (RSM) was used to investigate the main effects of the process variables (osmotic treatment temperature, solution concentration and fruit : solution ratio) on WL, SG, and TAC during osmotic dehydration of sweet cherries and to find the optimum parameters of dehydration. The experimental design adopted was a central composite rotatable design with three factors and five levels for each factor [20]. Selection of the actual factor values was based on the literature. The independent variable values and their corresponding levels are presented in Table 1.

The complete design consisted of 17 experimental runs with three replications of the central point. The generalized second-order polynomial model used in the response surface analysis was the following:

$$Y = b_o + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n} b_{ii} x_i^2 + \sum_{i=1}^{n} \sum_{j=1}^{n} b_{ij} x_i x_j \qquad (3)$$

where: Y is the dependent variable (response), x_i and x_j are the independent variables (factors), β_o, β_i, β_{ii}, β_{ij} are the regression coefficients for intercept, linear, quadratic, and interaction terms. Experimental design data were analysed through the analysis of variance (Anova) and the *F*-test at $P < 0.1$, using the statistical software SYSTAT (SPSS Inc., Chicago, USA, version 7.1) and Excel (Microsoft Office, 97, 2003).

RESULTS AND DISCUSSION

The average values for WL, SG, and TAC of the osmotic dehydrated sweet cherries are presented in Table 2.

Regression analyses of the water loss, solid gain, and total antioxidant capacity of osmotically dehydrated sweet cherries indicated that all second-order polynomial models correlated well with the measured data and were statistically significant ($p < 0.05$).

Table 1. Central composite rotatable design in coded form and natural units of independent variables

Run №	Osmotic treatment temperature - $\mathbf{X_1}$ (°C)	Solution concentration $\mathbf{X_2}$ (°Brix)	Fruit : solution ratio $\mathbf{X_3}$ (w/w)
1.	50 (-)	50 (-)	1:3 (-)
2.	70 (+)	50 (-)	1:3 (-)
3.	50 (-)	60 (+)	1:3 (-)
4.	70 (+)	60 (+)	1:3 (-)
5.	50 (-)	50 (-)	1:5 (+)
6.	70 (+)	50 (-)	1:5 (+)
7.	50 (-)	60 (+)	1:5 (+)
8.	70 (+)	60 (+)	1:5 (+)
9.	43 (-1.68)	55 (0)	1:4 (0)
10.	77 (+1.68)	55 (0)	1:4 (0)
11.	60 (0)	47 (-1.68)	1:4 (0)
12.	60 (0)	63 (+1.68)	1:4 (0)
13.	60 (0)	55 (0)	1:2 (-1.68)
14.	60 (0)	55 (0)	1:6 (+1.68)
15.	60 (0)	55 (0)	1:4 (0)
16.	60 (0)	55 (0)	1:4 (0)
17.	60 (0)	55 (0)	**1:4 (0)

The resulting models, after removing the non-significant terms, were evaluated in terms of uncoded factors and are presented below:

$$WL = 564.81 - 6.74X_1 - 16.33X_2 + 0.07X_1^2 + 0.18X_2^2 - 0.64X_2X_3, \%\quad (R^2 = 0.97) \qquad (4)$$

$$SG = 20.13 - 1.38X_1 + 0.87X_2 + 0.02X_1X_2 + 1.03X_3^2, \%\quad (R^2 = 0.93) \qquad (5)$$

$$TAC = 74452.7 + 2313.57X_1 - 4089.52X_2 - 15.94X_1^2, \ \mu mol\ TE/100g\quad (R^2 = 0.93) \qquad (6)$$

It was found that the osmotic treatment temperature and solution concentration were the most significant factors affecting the water loss, solids gain, and total antioxidant capacity. The effects of the independent variables (osmotic treatment temperature, solution concentration and fruit: solution ratio) on the dependent variables (WL, SG, and TAC) are indicated by the response surfaces plots (Figs. 2, 3, and 4) developed from the equation models shown above.

The criterion for estimation of the optimal osmotic dehydration conditions of sweet cherries was the achievement of high values of water loss (WL > 35%), solid gain (SG > 7.5%), and total antioxidant capacity (TAC > 21000 μmol TE/100 g).

Optimization was carried out by the superposition of several contour surfaces of competing responses. The response surface plots were generated for interaction of two independent variables (osmotic treatment temperature and solution concentration), while the value of the third variable (fruit : solution ratio) remained constant (at the central value). The best conditions that correspond to the shaded area obtained by superimposing contour plots of water loss, solid gain and total antioxidant capacity, are shown in Figure 5.

Table 2. Water loss, solids gain, and total antioxidant capacity of osmotically dehydrated sweet cherries

№	Water loss **Y_1** (%)	Solids gain **Y_2** (%)	Total antioxidant capacity **Y_3** (μmol TE / 100 g dw)
1	28.35	6.84	18982.5
2	58.89	8.83	16804.5
3	44.48	7.14	24304.6
4	74.25	15.62	19832.2
5	31.55	6.16	18540.1
6	56.79	9.54	18201.2
7	33.37	6.57	26811.5
8	60.89	12.63	23750.2
9	30.29	7.28	19198.6
10	72.49	12.07	14894.2
11	37.33	6.34	23773.3
12	51.66	9.11	25325.3
13	35.06	12.19	19417.4
14	35.63	11.25	23890.1
15	34.23	8.47	23174.7
16	35.11	8.32	21470.8
17	34.47	8.43	19723.7

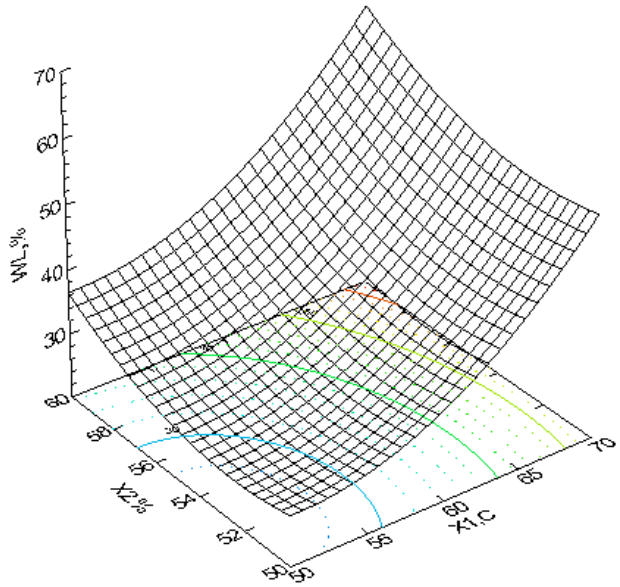

Figure 2. WL (%) depending on X_1 (ºC) and X_2 (°Brix) at fruit : solution ratio 1:4 (w:w).

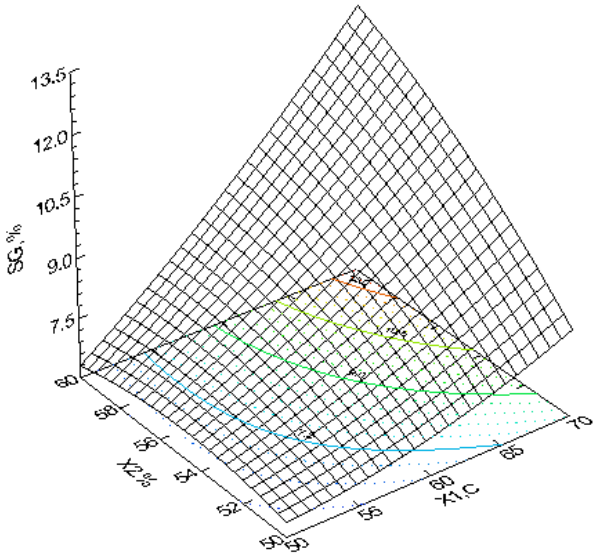

Figure 3. SG (%) depending on X_1 (ºC) and X_2 (°Brix) at fruit : solution ratio 1:4 (w:w).

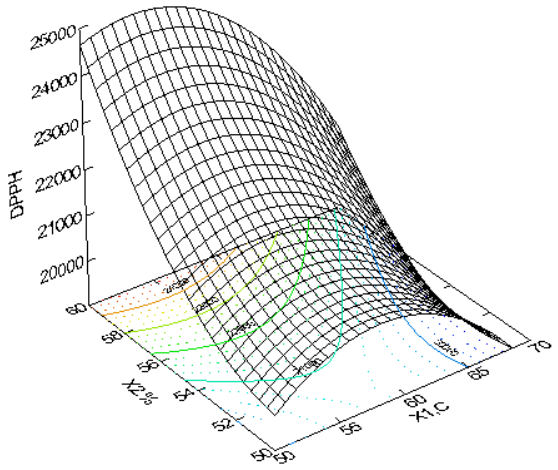

Figure 4. TAC (μmol TE/100 g) depending on X_1 (ºC) and X_2 (°Brix) at fruit : solution ratio 1:4 (w:w).

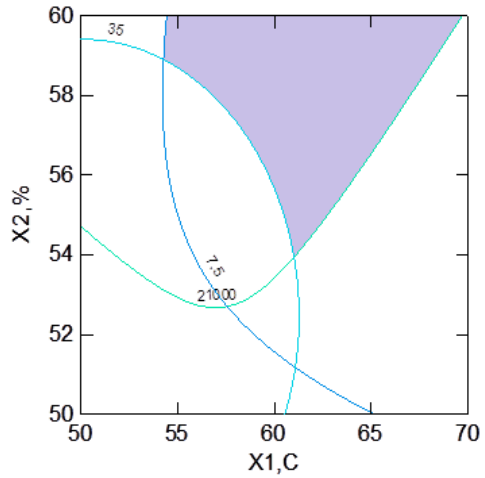

Figure 5. Superposition area of the responses as an effect of the treatment temperature (X_1) and solution concentration (X_2) on the osmotic dehydration of sweet chokeberry.

CONCLUSION

Response surface methodology was used for a quantitative study on the effects of process variables on water loss, solid gain, and total antioxidant capacity of osmotic-dehydrated cherry fruits. Optimization was carried out by the superposition of several contour surfaces of competing responses. The criterion to determine the optimal osmotic dehydration conditions of sweet cherries was the achievement of high values of water loss (WL > 35%), solid gain (SG > 7.5%), and total antioxidant capacity (TAC > 21000 μmol TE/100 g). Results from the present study showed that the osmotic treatment temperature and solution concentration had a significant effect on the mass transfer (water loss and solid gain) and the total antioxidant capacity. The increase of the osmotic treatment temperature resulted in degradation of the biologically active components and, respectively, in decreased total antioxidant capacity. The application of response surface methodology proved to be very efficient for the optimization of osmotic dehydration of sweet cherries.

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