# Osmotic dehydration as a preliminary technological process for the production of dried chokeberry (Aronia melanocarpa)

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Black chokeberry was subjected to osmotic dehydration in a solution containing concentrated sour cherry juice (60% w:w), concentrated apple juice (20% w:w), and inulin (20% w:w). The effects of osmotic treatment temperature, solution concentration, and fruit:solution ratio on water loss (WL) and weight reduction (WR) of chokeberry fruits were studied. Response surface methodology (RSM) was applied to assess the combinations of osmotic treatment temperature (43, 50, 60, 70, and 77°C), solution concentration (47, 50, 55, 60, and 63°Brix), and chokeberry:solution ratio (1:2, 1:3, 1:4, 1:5, and 1:6 w:w). Water loss values varied from 20.82 to 43.43% and the weight reduction values varied from 11.93 to 41.58%, respectively. Osmotic treatment temperature had the highest impact on water loss, and fruit weight reduction was mostly influenced by the osmotic solution concentration, with both effects being linear.

Keywords: black chokeberry, osmotic dehydration, water loss, weight reduction, response surface methodology

#### **INTRODUCTION**

Osmotic dehydration is a pre-treatment method for the partial removal of water from plant tissues by immersion of foodstuff in hypertonic water solution [1]. Osmotic dehydration involves three mass transfer processes: (1) water transfer from the plant product to the osmotic solution, (2) incursion of solutes from the solution into the foodstuff and (3) excretion of soluble solids from the plant tissues to the osmotic solution. The third transfer is quantitatively negligible compared to the first two types of transfer but it is essential for the composition of the product [2, 3]. The different flows in and out of the plant tissue are shown in Fig.1.



**Fig.1.** Mass transfer in fruit tissue during osmotic dehydration

The rate of osmotic dehydration and final indicators of dehydrated plant products is mainly dependent on the temperature of the osmotic

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solution, type and concentration of osmotic solution, and fruit to osmotic solution ratio [4, 5].

#### Temperature of the osmotic solution

The most important variable affecting the transfer during kinetics of mass osmotic dehydration is temperature [4]. Increasing the temperature of the osmotic solution results in an increase in the rate of water removal and sugar uptake [5]. Diffusion of flavour and odour compounds from the fruits to the solution is also increased at high temperature. According to the literature on osmotic treatment, temperatures around 50°C have been used for fruits and vegetables for the following reasons: 1) deterioration of flavour, texture, and thermosensitive compounds of the materials is limited at this temperature, 2) enzymatic browning and flavour deterioration of fruits start at 49°C, and 3) this temperature proved efficient to maintain the viscosity of the solution and ensure adequate infusion time without deterioration of fruit quality [6, 7].

### Type and concentration of osmotic agent

Phisut [5] reported that low molecular weight osmotic agents penetrate easier into the fruit cells compared to high molecular weight osmotic agents. The most commonly used osmotic agents are sucrose, glucose, fructose, sorbitol, glycerol, corn syrup and fructo-oligosaccharide.

During osmotic dehydration, the increase of solute concentrations results in higher water loss

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and solid gain rates [5]. The increased osmotic solution concentration resulted in higher water loss and weight reductions and increased drying rates [4, 8, 9].

# Fruit: osmotic solution ratio

The increase of fruit sample:osmotic solution ratio results in higher osmosis rate up to a certain level. However, it is essential to use an optimum ratio since large ratios offer practical difficulties in handling the processing of osmotic solution and fruit mixture. A ratio of 1:2 or 1:3 sample:osmotic solution is reported as optimal for practical purposes [10].

There are some major advantages of osmotic dehydration process in the food industry: quality aspect (improvement in terms of colour, flavour, and texture), product stability and retention of nutrients during storage, energy efficiency, packaging and distribution cost reduction, chemical treatment not required, product stability during storage [11-13].

The genus Aronia (Rosaceae family, Maloidea subfamily) includes two species A. melanocarpa, known as black chokeberry and A. arbutifolia as red chokeberry. Black chokeberry fruits contain carbohydrates (10-18% wet basis), pectins (0.6-0.7% wet basis), sorbitol and parasorboside [14, 15]. Analyses showed relatively high content of K and Zn, as well as some amounts of Na, Ca, Mg and Fe [16, 17]. Chokeberry fruits are very rich in bioactive compounds, mainly total polyphenols (3.44-7.49% of dry weight), anthocyanins (0.60-2.0% of dry weight) and flavonols (0.66-5.18% of dry weight) [18-20]. Due to the high anthocyanin content, chokeberry fruit is used as an ingredient of antioxidant health-promoting juices, teas, extracts for production of syrups and dietary supplements [21-23].

Response surface methodology (RSM) is a statistical tool used in process research for evaluation of the influence of different treatment parameters on product characteristics [24]. It has also been used to determine the optimal values of process parameters in various processes [25-27]. Several studies on optimization of plant products dehydration by RSM method have been reported [28-30].

Although the process of osmotic dehydration has been extensively studied, information about the mass transfer processes of osmotic dehydration of black chokeberry is hardly found in the literature. Therefore, the present study is focused on the water loss and weight reduction as a function of process variables (osmotic treatment temperature, solution concentration and chokeberry fruits:solution ratio).

# EXPERIMENTAL DETAILS

## Raw materials

Black chokeberry (*Aronia melanocarpa*) fruits with soluble solids of 24-27°Brix were supplied by the Agricultural and stockbreeding experimental station, 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 (oligofructose 87%, average degree of polymerization 8; sucrose, glucose, and fructose 12%) were purchased from Krichimfrukt Ltd. (Bulgaria), Agrobiotech Ltd. (Bulgaria), and Food consulting Ltd. (Bulgaria), respectively. The containers of concentrated sour cherry juice and concentrated apple juice were stored in a refrigerator at 3°C until used.

## Sample preparation and osmotic process

Black chokeberry was washed with tap water, and then subjected to splitting.

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) [31].

Osmotic dehydration of fruits was performed in a water bath (VEB MLW Prüfgeräte Werk, Medingen, Sitz Freital, Germany) to achieve the necessary solution temperature according to the experimental design (Table 1). The choice of the preferred process conditions was according to survey of other researchers on osmotic dehydration [3, 32]. The berries were dipped in osmotic solution of a specified concentration for 3 hours. The fruit:solution ratio was 1:2, 1:3, 1:4, 1:5, and 1:6 (w:w) according to the experimental design (Table 1). Further, the fruits were removed from the solutions, quickly rinsed with hot water and gently blotted with paper towel to remove surface solution.

Total dry matter and drained weight (final sample weight) of the osmotic dehydrated fruits were determined [33]. Further, the samples were analyzed for the main indicators - water loss and weight reduction. Weight reduction (WR) is defined as the net difference in weight between the initial weight of the fruit sample and the weight of the osmotically dehydrated fruit based on the initial sample weight. Water loss (WL) was defined as the net loss of water from the fresh fruits after osmotic dehydration based on the initial sample [34]. These parameters were calculated according to the following equations:

$$WR = \frac{M_o^o - M_f^o}{M_o^o} 100,\%$$
 (1)

$$WL = \frac{x_o^w M_o^o - x_f^w M_f^o}{M_o^o} 100,\%$$
 (2)

where  $M_o^o$  – the initial sample weight (g),  $M_f^o$  – the final sample weight (g),  $x_o^w$  – initial moisture content (%),  $x_f^w$  – final moisture content (%).

Experimental design and data analysis

Central composite rotatable design (CCRD) was used to prepare osmotic dehydration of black chokeberry, by response surface methodology with three variables at five levels. The actual factor values were chosen based on the literature review and the corresponding coded values (- 1.68, -1, 0, +1, +1.68) are presented in Table 1. The complete design consisted of 17 experimental runs with three replications of the center point. Experimental runs were carried out in random order.

 Table 1. Response surface central composite design

 with three factors (variables) at five levels

№	Osmotic treatment temperature	Solution concentration	Fruit : solution ratio
	X <sub>1</sub> (°C)	X <sub>2</sub> (°Brix)	X <sub>3</sub> (w/w)
1	50 (-1)	50 (-1)	1:3 (-1)
2	70 (+1)	50 (-1)	1:3 (-1)
3	50 (-1)	60 (+1)	1:3 (-1)
4	70 (+1)	60 (+1)	1:3 (-1)
5	50 (-1)	50 (-1)	1:5 (+1)
6	70 (+1)	50 (-1)	1:5 (+1)
7	50 (-1)	60 (+1)	1:5 (+1)
8	70 (+1)	60 (+1)	1:5 (+1)
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 generalized second-order polynomial model used in the response surface analysis was the following:

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

where *y* is the dependent variable (response),  $x_i$ and  $x_j$  are the independent variables (factors),  $\beta o$ ,  $\beta_i$ ,  $\beta_{ii}$ ,  $\beta_{ij}$  are the regression coefficients for intercept, linear, quadratic, and interaction terms. SYSTAT statistical software (SPSS Inc., Chicago, USA, version 7.1) and Excel (Microsoft Office, 97-2003) were used to analyse the data.

# RESULTS AND DISCUSSION

The average weight reduction (WR) and water loss (WL) values of the osmotically dehydrated black chokeberry are presented in Table 2. WR varied from 11.93 to 41.58% and WL values varied from 20.82 to 43.43%, respectively.

**Table 2.** Weight reduction and water loss values of the osmotically dehydrated black chokeberry

N⁰	Weight reduction	Water loss Y <sub>2</sub> (%)	
	Y <sub>1</sub> (%)		
1	11.93	20.82	
2	20.55	28.84	
3	17.27	25.80	
4	28.88	36.80	
5	20.63	29.13	
6	28.25	36.01	
7	34.97	33.22	
8	41.58	43.43	
9	15.30	24.61	
10	28.22	37.27	
11	15.35	28.70	
12	38.17	41.74	
13	16.64	29.98	
14	18.87	30.43	
15	17.47	25.51	
16	16.87	26.10	
17	17.11	25.61	

**Table 3.** Regression coefficients and analysis of variance for weight reduction of black chokeberry

N⁰	Regression coefficients	Sum of squares	F- value	P-value
Constant	581.471			
A:X <sub>1</sub>	-2.347	231.179	12.29	0.0099*
B: X <sub>2</sub>	-18.523	465.337	24.74	0.0016*
C:X <sub>3</sub>	-18.294	187.111	9.95	0.0161*
AA	0.023	60.766	3.23	0.1153
AB	0.005	0.490	0.03	0.8763
AC	-0.075	4.500	0.24	0.6397
BB	0.164	188.530	10.02	0.0158*
BC	0.350	24.500	1.30	0.2913
CC	0.906	9.247	0.49	0.5058

\*Significant at P < 0.05

Statistical analysis of variance (ANOVA) for WR and WL shows that 4 effects have p-values less than 0.05, indicating that they are significantly different from zero at the 95.0% confidence level (Table 3 and Table 4). For the indicator weight reduction the R-squared is 0.89; the standard error of the estimate is 4.34, and the mean absolute error is 2.08. For water loss of osmotic-dehydrated fruits R-squared is 0.93; the standard error of the estimate is 2.62, and the mean absolute error is 1.33.

**Table 4.** Regression coefficients and analysis of variance for water loss of black chokeberry

N⁰	Regression coefficients	Sum of squares	F- value	P-value
Constant	460.836			
$A:X_1$	-2.357	241.266	35.15	0.0006*
B: X <sub>2</sub>	-14.137	157.515	24.74	0.0020*
C:X <sub>3</sub>	-5.661	67.167	9.95	0.0167*
AA	0.017	31.494	3.23	0.0694
AB	0.016	4.977	0.03	0.4227
AC	-0.024	0.466	0.24	0.8020
BB	0.127	114.333	10.02	0.0047*
BC	0.036	0.265	1.30	0.8525
CC	1.412	22.462	0.49	0.1134

\*Significant at P < 0.05

Each of the estimated effects and interactions are shown in the standardized diagrams (Fig.2 and Fig.3). The linear effect due to the concentration of osmotic solution had the highest impact on the weight reduction. The temperature of osmotic treatment also had significant effect on weight reduction (P<0.05). Similar results were observed by Fernandes et al. [35] at osmotic dehydration of papaya in corn syrup and sugar.





**Fig.2.** Estimated effects of regression model coefficients on the weight reduction of black chokeberry





**Fig.3.** Estimated effects of regression model coefficients on the WL of black chokeberry

In agreement with Fig.3, temperature of the osmotic solution had the most significant effect on water loss, followed by the concentration of the osmotic solution and fruit to osmotic solution ratio (P<0.05). İspir and Toğrul [36], Kumar and Devi [37] also confirmed that an increase in temperature resulted in an increase in the rate of water loss.

Based on the results from ANOVA after removing the insignificant effects, the following regression equations were obtained:

$$WR = 581.47 - 2.35X_1 - 18.52X_2 - 18.29X_3 + 0.16X_2^2, \%$$
(4)

$$WL = 460.84 - 2.36X_1 - 14.14X_2 - 5.66X_3 + 0.13X_2^2, \%$$
(5)

The effect of osmotic dehydration conditions on the weight reduction and water loss of black chokeberry are shown on Fig.4 and Fig.5. The figures show the combined effect of two factors (temperature and concentration of osmotic solution) on the response of dependent variables.



**Fig.4.** WR (%) depending on  $X_1$  (°C) and  $X_2$  (°Brix) at fruit:solution ratio 1:4 (w:w)

The increase in solution concentration and temperature resulted in higher water loss (Fig.5) and weight reduction (Fig,4) values throughout the osmosis period. WR and WL increase about 1.8 and 1.5 times respectively (see Table 2) with raising the osmotic treatment temperature from 43 to 77°C (at solution concentration 55°Brix and fruit:solution ratio 1:4 (w/w). Similar results were reported by many authors [36, 38-41]. Higher temperatures seem to promote faster water loss through swelling and plasticizing of cell membranes as well as the better water transfer characteristics on the product surface due to lower viscosity of the osmotic

medium [5, 38]. Fruit sample has a porous structure so that high temperature releases the trapped air from the tissue resulting in more effective to the removal of water by osmotic pressure. This enhances the removal of water and uptake of solids [5].



**Fig.5.** Water loss (%) depending on  $X_1$  (°C) and  $X_2$  (°Brix) at fruit:solution ratio 1:4 (w:w)

Our results show that WR and WL increase about 2.5 and 1.5 times respectively (see Table 2) with raising the solution concentration from 47 to 63°Brix (at osmotic treatment temperature 60°C and fruit:solution ratio 1:4 (w/w). The increase in solution concentration resulted in an increase in the osmotic pressure gradients [36, 41] and, hence, higher water loss (Fig.5) and weight reduction (Fig.4) values throughout the osmosis period were obtained. These results indicate that by choosing a higher concentration, some benefits in terms of faster water loss could be achieved.

# CONCLUSION

Combination of different osmotic agents was chosen to achieve the purpose of the study. The optimal area of combinations of osmotic agents was presented in our previous study [42]. Rich in biologically active substances fruit juices are used (concentrated sour cherry juice, concentrated apple juice) and inulin. The main advantages of fructans including inulin are: highly soluble, low degree of polymerization (8 fructose units), technological properties that are closely related to those of sugar and glucose syrups. In addition, inulin is a prebiotic food ingredient.

The choice of the preferred process conditions was according to survey of other researchers on osmotic dehydration. Response surface methodology was used for a quantitative study on the effects of the osmotic treatment temperature, solution concentration, and fruits:solution ratio on weight reduction and water loss of osmotically dehydrated chokeberry fruits. Results show that all three main effects are statistically significant for both responses during the osmotic dehydration of fruits with osmotic solution contain cherry juice (60% w:w), concentrated apple juice (20% w:w), and inulin (20% w:w).

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