Received: 14 October 2021 | Revised: 24 January 2022 | Accepted: 24 February 2022

DOI: 10.1111/jfpp.16512

Journal of Food Processing and Preservation | Institute of Food Science + Technology | ifst | WILEY

ORIGINAL ARTICLE

Synergetic dehydration method of osmotic treatment in molasses and successive lyophilization of peaches

Vladimir Filipović[1] | Jelena Filipović[2] | Biljana Lončar[1] | Violeta Knežević[1] | Milica Nićetin[1] | Ivana Filipović[3]

[1]Faculty of Technology Novi Sad, University of Novi Sad, Novi Sad, Serbia

[2]Insititute for Food Technology, University of Novi Sad, Novi Sad, Serbia

[3]SP Laboratory, Bečej, Serbia

Correspondence
Vladimir Filipović, Faculty of Technology Novi Sad, University of Novi Sad, Bulevar Cara Lazara 1, 21000 Novi Sad, Serbia.
Email: vladaf@uns.ac.rs

Funding information
Povincial Secretariat of Higher Education and Scientific Research, Autonomous Province of Vojvodina, Republic of Serbia, Grant/Award Number: 142-451-2289/2021-01/02

Abstract

Osmotic dehydration in molasses and successive lyophilization of peaches are investigated, in effort of obtaining new and improved product. Prepared samples were subjected to the osmotic dehydration in molasses of different time, concentration, and temperature, and then to the 5-hr lyophilization process. The results showed that all three osmodehydration parameters statistically significantly affected dry matter content and a_w of successively dehydrated samples, reaching peak values of 83.63% and 0.433 of dry matter content and a_w, respectively. Osmodehydration process contributed to upgrading overall dehydration effectiveness, while a_w values reduction in lyophilization stage contributed to the increased samples' microbiological stability. Mineral matter content was highly increased, up to 8.63, 248.30, 64.05, and 101.56 times higher, for K, Ca, Mg, and Fe, respectively, as a consequence of molasses application. Developed mathematical models of 12 responses of osmotic dehydration and lyophilization processes were statistically significant, well-describing synergetic performance of two successive dehydration methods.

Novelty impact statement: Osmotic dehydration, as a simple, low energy-demanding process, especially with molasses used as an osmotic solution, was applied in effort to reduce the extent of further dehydration phase. The osmotic dehydration phase also supplemented mineral matter content of the dehydrating peach samples with its high value nutritional content. Lyophilization, as energy demanding, yet high quality-producing technique, as a successive dehydration phase, provided peach samples of very high dry matter content and very low a_w values in reduced duration of application.

1 | INTRODUCTION

Water is the main factor that affects foods' chemical and microbiological stability, whereby lowering water activity values, the food products shelf-life can be increased and stability can be prolonged (Blanda et al., 2009; Moreno et al., 2013). The osmotic dehydration process consists of the immersion of the food material in a hypertonic solution, where water diffuses from the food toward the solution, due to the semi-permeability of the cell membranes of food tissue and, in the opposite way, the solute, used as osmotic solution agents, flows from the solution to the food, in minor extent (Da Costa Ribeiro et al., 2016). The driving force of the mass transfer in the process is the concentration difference between the osmotic solution and the interstitial fluid (Ciurzyńska et al., 2016). Sugar beet molasses has proven to be good choice as an osmotic solution, due to its technological effectiveness in mass transfer phenomena and its highly valued nutritive composition and low cost as a by-product of sugar industry (Filipović et al., 2017; Nićetin et al., 2021).

The osmotic dehydration process is simple, with low energy demands and the equipment used for this method is cheap. It is used

as one of the steps in food processing, where biological material is treated before finalization to the final product. For the further finalization processes, freezing, lyophilization, vacuum, and convective drying, can be used (Mandala et al., 2005; Shi & Xue, 2009).

Lyophilization is a valuable technique for producing high-quality dehydrated products with very high dry matter content. Low processing temperatures help to preserve nutrients, such as minerals, vitamins, and flavonoids (Igual et al., 2019). Lyophilization is considered better than air drying mainly due to less damage to the heat-sensitive compounds, while the product can be easily reconstituted with water (Fahloul et al., 2009). Application of this technology in the food industry has been limited to high added value products, since long processing times and high operation costs are needed for obtaining adequate quality lyophilized products. Considering process energy consumption, lyophilization requires almost the double the amount of energy for the removal of 1 kg of water from dehydrating material in comparison to conventional drying (Liu et al., 2008).

Peach has favorable nutritional content, free of sodium, fat, and cholesterol, with rich content of Vitamins A and C (Yadav et al., 2012); hence, preserving and possibly increasing its' nutritional content is of great importance.

The combination of these two dehydration methods (osmotic dehydration in molasses and successive lyophilization), where the final result provides enhanced nutritive composition of the dehydrated product, is not yet investigated.

The goal of this research is to investigate and model the effect of technological parameters on osmotic dehydration in molasses and successive lyophilization of peaches dehydration method performance, in effort of obtaining new and nutritionally improved peach products.

2 | MATERIAL AND METHODS

2.1 | Osmotic dehydration process of peaches

The initial dry matter content of the fresh peaches (*Prunus persica, var. nucipersica)* was 7.40% ± 0.08%. Before the osmotic treatment, peaches were washed with running water, dried with paper towels, peeled, and cut into cubes, of approximate dimensions of 1 × 1 × 1 cm.

Sugar beet molasses had initial dry matter content of 85.04%. Distilled water was used for the preparation of dilutions of the sugar beet molasses to the solution concentrations of 60%, 70%, and 80% of dry matter.

The osmotic dehydration process was performed in laboratory vessels under atmospheric pressure, at a constant temperature chamber (Memmert IN160, Germany). The temperature of the process varied between 20, 35, and 50°C.

The duration of the process was varied between 1, 3, and 5 hr.

The sample (peach cubes) to osmotic solution (sugar beet molasses solution) ratio of 1:5 (weight/weight) was used, to reduce excessive solution dilution. Peach samples were immersed in molasses and stirred every 15 min for the purpose of better molasses homogenization with the defunded water from the peach samples. After the end of the process, peach samples were taken out from molasses solutions to be lightly washed with water and gently blotted to remove excess water.

The final dry matter content of fresh and osmotically dehydrated peaches in molasses was determined by drying the peach samples at 105°C for 24 hr in a heat chamber (Instrumentaria Sutjeska, Serbia) until a constant mass was achieved. All analytical measurements were carried out in accordance with AOAC. 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 25°C.

2.2 | Lyophilization

Osmotically dehydrated and fresh peach samples were frozen and stored at −30°C until lyophilization. Frozen samples were weighted and approximately 30 g of samples were placed in Freeze Dryer Christ ALPHA1-2 LD_{PLUS}, Osterode am Harz, Germany. Lyophilization parameters were set to pressure of the 1.6 Pa, condenser temperature of −57°C, and duration of the lyophilization of 5 hr. After the lyophilization process, samples were weighted and water activity (a_{wL}) was measured the same, as in case of osmotically dehydrated samples.

2.3 | Analysis of chemical and mineral matter content

Analysis of chemical content of fresh and treated (osmodehydrated and lyophilized) peach samples was performed according to the official methods of AACC: proteins (AACC, 2000a), sugar (AACC, 2000b), and ash (AACC, 2009).

The contents of potassium (K), calcium (Ca), magnesium (Mg), and iron (Fe) of fresh and treated (osmodehydrated and lyophilized) peach samples were performed according to ISO 6869:2000.

All analyses on tested samples were done in triplicates.

2.4 | Calculations

2.4.1 | Calculation of osmotic dehydration responses

In order to describe the effectiveness of the mass transfer of the osmotic dehydration process, dry matter content after osmotic dehydration (DMC) was calculated for different temperatures and processing times:

$$\mathrm{DMC} = \frac{m_f}{m_i} \bullet 100\,\% \tag{1}$$

where m_i and m_f are the initial and final mass (g) of the samples, respectively (Filipović et al., 2013).

2.4.2 | Calculation of Lyophilized Samples' Response

Dry matter content of lyophilized peach samples was determined according to:

$$DMC_L = \frac{\frac{DMC}{100} \bullet m_{PL}}{m_L} \bullet 100\,\% \quad (2)$$

where DMC_L is dry matter content of samples after lyophilization, m_{PL} is the mass of samples prior lyophilization, and m_L is mass of samples after lyophilization.

Water loss of osmodehydrated peach samples in the lyophilization stage (WL_L) was determined according to:

$$WL_L = (100 - DMC) - (100 - DMC_L) \quad (3)$$

2.4.3 | Response surface methodology

Second-order polynom was used for experimental data fitting. Twelve models of the following form were developed to relate 12 responses (Y) to three process variables (X):

$$Y_k = \beta_{k0} + \sum_{i=1}^{3} \beta_{ki} 0 X_i + \sum_{i=1}^{3} \beta_{kii} X_i^2 + \sum_{i=1}^{2} \sum_{j=i+1}^{3} \beta_{kij} X_i X_j, k = 1 - 12; \quad (4)$$

where were: β_{kij} regression coefficients; Y were either DMC_{OD} (Y_1), a_{wOD} (Y_2), DMC_L (Y_3) WL_L (Y_4), a_{wL} (Y_5), proteins (Y_6), sugar (Y_7), ash (Y_8), K (Y_9), Ca (Y_{10}), Mg (Y_{11}) or Fe (Y_{12}) and X represents process time (X_1), osmotic solution concentration (X_2), and process temperature (X_3).

The significance of the effect and interaction of individual factors, for every response, was determined by analysis of variance (ANOVA) and application of *post hoc* Tukey HSD test. For ANOVA and RSM analysis, StatSoft Statistica ver.12.0 software package is used.

3 | RESULTS

Table 1 shows DMC and a_w values of osmodehydrated and lyophilized samples at different applied osmotic dehydration process parameters. Maximal obtained DMC value of 50.81% was achieved after 5-hr osmotic dehydration process in molasses of maximal concentration (80%), at maximal process temperature of 50°C.

The minimal obtained a_w value was 0.864, achieved at the same set of technological parameters as in case of maximal obtained DMC value.

Fresh and osmotically dehydrated peach samples were subjected to the second dehydration stage—lyophilization process. The results of the same analysis (dry matter content and water activity), after the lyophilization stage (DMC_L and a_{wL}), are also presented in Table 1.

Maximal obtained DMC_L value of 83.63% was achieved in successive dehydration process of 5-hr osmotic dehydration stage in molasses of maximal concentration (80%), at a maximal process temperature of 50°C, and lyophilization stage (pressure of the 1.6 Pa, condenser temperature of −57°C, and duration of 5 hr).

In Table 2, values of chemical and mineral matter content of fresh and treated (osmodehydrated and lyophilized) peach samples, are shown.

The ANOVA calculation, presented in Table 3, showed the effects of the independent variables (osmodehydration process time, molasses' concentration, and osmodehydration process temperature) on all investigated responses (osmotic dehydration and lyophilization processes' responses, chemical and mineral matter content).

Table 4 shows regression coefficients of 12 s order polynom models of responses of osmotic dehydration and lyophilization processes, chemical and mineral matter content of peach samples. Statistical significance of individual coefficients is also marked.

4 | DISCUSSION

Analysis of DMC values (after first dehydration stage—osmotic dehydration process), Table 1, shows that with the increase of all three parameters' values (time, concentration, and temperature) DMC values of osmodehydrated peach samples statistically significantly increased. This trend is the same as in osmotic dehydration process of other biological materials—plant (Mišljenović et al., 2012, Nićetin et al., 2017; Knežević et al., 2019) and animal raw materials (Ćurčić et al., 2015; Filipović et al., 2013, 2017).

Values of a_w after the first dehydration stage, Table 1, show a similar trend of the effect of technological parameters, as in case of DMC values, except with the increase of all three parameters values, osmodehydrated peach samples a_w values statistically significantly decreased. Minimal obtained a_w value is in accordance with other obtained osmodehydrated plant material a_w values at similar applied technological parameters: 0.860—carrot cubes (Mišljenović et al., 2012) and 0.820—celery root (Nićetin et al., 2017).

Since the lyophilization process of the constant parameters was applied to all osmodehydrated peach samples, the differences between DMC_L values are similar to the ones in DMC values (after the first stage of dehydration), Table 1. The effects of different applied technological parameters of the osmodehydration process have the same tendencies in the lyophilized peach samples, as in osmodehydrated peach samples. The increase of all three osmodehydration technological parameters has also led to the statistically significant increase of DMC_L values of two-stage dehydrated peach samples.

Analyzing lyophilization stage performance, it can be seen that a higher level of present water content in samples after the first osmodehydration stage had led to higher water removal in the second, lyophilization dehydration stage (WL_L values), Table 1. For example, the highest water removal in the lyophilization stage had occurred in

TABLE 1 Average values and standard deviations of osmotic dehydration process responses and lyophilized samples' responses

				Osmotic dehydration process responses		Osmodeyhdrated and lyophilized samples' responses		
Sample number	Time (h)	Concentration (d.m. %)	Temperature (°C)	DMC (%)	a_w	DMC_L (%)	WL_L (%)	a_{wL}
0	–	–	–	7.40 ± 0.11^{a}	0.940 ± 0.002^{o}	15.34 ± 0.11^{a}	7.94 ± 0.09^{a}	0.902 ± 0.002^{s}
1	1	60	20	15.88 ± 0.10^{b}	$0.927 \pm 0.007^{j-m}$	42.00 ± 1.12^{b}	26.12 ± 0.87^{b}	0.873 ± 0.004^{r}
2	3	60	20	23.40 ± 0.17^{f}	0.898 ± 0.002^{n}	50.39 ± 1.02^{c}	26.99 ± 0.57^{bc}	0.827 ± 0.005^{q}
3	5	60	20	30.92 ± 0.40^{i}	$0.893 \pm 0.003^{f-j}$	58.63 ± 1.18^{ef}	27.71 ± 0.67^{cd}	0.723 ± 0.005^{n}
4	1	70	20	17.81 ± 0.08^{cd}	0.909 ± 0.006^{m}	51.43 ± 1.32^{c}	33.62 ± 0.44^{jk}	0.803 ± 0.001^{p}
5	3	70	20	24.56 ± 0.27^{g}	$0.893 \pm 0.003^{g-k}$	56.97 ± 1.35^{e}	32.41 ± 0.73^{i}	0.741 ± 0.005^{o}
6	5	70	20	33.52 ± 0.11^{j}	$0.889 \pm 0.005^{e-h}$	62.64 ± 0.89^{ij}	29.12 ± 0.41^{fg}	0.641 ± 0.005^{i}
7	1	80	20	16.99 ± 0.18^{c}	0.905 ± 0.002^{lm}	59.46 ± 0.47^{fg}	42.47 ± 0.33^{r}	0.683 ± 0.007^{m}
8	3	80	20	25.22 ± 0.33^{g}	$0.899 \pm 0.007^{k-m}$	66.13 ± 1.44^{kL}	40.91 ± 0.75^{q}	0.590 ± 0.002^{g}
9	5	80	20	35.11 ± 0.40^{k}	$0.885 \pm 0.004^{d-g}$	70.10 ± 0.43^{m}	34.99 ± 0.41^{lm}	0.576 ± 0.002^{f}
10	1	60	35	16.93 ± 0.16^{c}	0.905 ± 0.003^{m}	53.58 ± 0.56^{d}	36.65 ± 0.29^{no}	0.795 ± 0.008^{p}
11	3	60	35	28.95 ± 0.19^{h}	$0.886 \pm 0.003^{e-h}$	$60.64 \pm 1.06^{g-i}$	31.69 ± 0.54^{hi}	0.664 ± 0.006^{k}
12	5	60	35	39.49 ± 0.41^{m}	$0.880 \pm 0.003^{c-f}$	67.42 ± 0.28^{L}	$27.93 \pm 0.39^{c-e}$	0.650 ± 0.001^{ij}
13	1	70	35	18.42 ± 0.21^{d}	$0.904 \pm 0.004^{k-m}$	$61.04 \pm 1.46^{g-i}$	42.63 ± 0.78^{r}	0.673 ± 0.002^{lm}
14	3	70	35	29.10 ± 0.30^{h}	$0.885 \pm 0.003^{d-g}$	66.02 ± 0.07^{kL}	36.92 ± 0.27^{o}	0.594 ± 0.004^{g}
15	5	70	35	39.97 ± 0.34^{m}	$0.880 \pm 0.004^{b-e}$	72.02 ± 0.83^{m}	32.04 ± 0.54^{hi}	0.546 ± 0.004^{d}
16	1	80	35	22.05 ± 0.20^{e}	$0.899 \pm 0.006^{i-m}$	$60.42 \pm 1.69^{f-h}$	38.38 ± 0.99^{p}	0.666 ± 0.007^{kL}
17	3	80	35	32.65 ± 0.13^{j}	$0.889 \pm 0.003^{e-i}$	71.08 ± 0.83^{m}	38.43 ± 0.49^{p}	0.560 ± 0.002^{e}
18	5	80	35	42.65 ± 0.63^{o}	0.872 ± 0.003^{abc}	77.13 ± 0.97^{n}	34.48 ± 0.76^{kL}	0.530 ± 0.002^{c}
19	1	60	50	21.42 ± 0.11^{e}	$0.897 \pm 0.005^{i-m}$	$57.17 \pm \pm 0.98^{e}$	$35.75 \pm 0.71^{m-o}$	0.733 ± 0.004^{no}
20	3	60	50	36.35 ± 0.47^{L}	$0.884 \pm 0.005^{c-f}$	65.38 ± 0.59^{k}	$29.03 \pm 0.51^{e-g}$	0.600 ± 0.002^{g}
21	5	60	50	41.90 ± 0.74^{no}	$0.874 \pm 0.003^{a-c}$	70.54 ± 0.20^{m}	$28.63 \pm 0.55^{d-f}$	0.570 ± 0.003^{f}
22	1	70	50	23.41 ± 0.26^{f}	$0.896 \pm 0.007^{h-l}$	61.65 ± 1.68^{hi}	38.24 ± 1.10^{p}	0.654 ± 0.002^{jk}
23	3	70	50	41.44 ± 0.63^{n}	$0.875 \pm 0.004^{b-d}$	72.65 ± 0.33^{m}	31.22 ± 0.44^{h}	0.551 ± 0.002^{de}
24	5	70	50	48.11 ± 0.23^{p}	0.869 ± 0.001^{ab}	78.13 ± 0.78^{n}	30.01 ± 0.49^{g}	0.513 ± 0.002^{b}
25	1	80	50	24.27 ± 0.25^{fg}	$0.890 \pm 0.006^{f-j}$	64.86 ± 1.58^{jk}	40.60 ± 1.10^{q}	0.611 ± 0.002^{h}
26	3	80	50	42.66 ± 0.36^{no}	$0.875 \pm 0.003^{b-d}$	78.40 ± 0.71^{n}	35.74 ± 0.41^{mn}	0.505 ± 0.002^{b}
27	5	80	50	50.81 ± 0.44^{q}	0.864 ± 0.004^{a}	83.63 ± 1.66^{o}	32.82 ± 0.98^{ij}	0.433 ± 0.005^{a}

[a–s]Different letters in superscript of the same table column indicate on the statistically significant difference between values, at level of significance of $p < 0.05$ (based on *post hoc* Tukey HSD test).

TABLE 2 Average values and standard deviations of chemical and mineral matter content of osmodehydrated and lyophilized samples

				Chemical content			Mineral matter content			
Sample number	Time (h)	Concentration (d.m. %)	Temperature (°C)	Proteins (% d.m.)	Sugar (% d.m.)	Ash (% d.m.)	K (mg/100 g d.m.)	Ca (mg/100 g d.m.)	Mg (mg/100 g d.m.)	Fe (mg/100 g d.m.)
0	-	–	–	8.27 ± 0.04^{a}	76.23 ± 0.20^{h}	3.64 ± 0.02^{a}	183.00 ± 0.75^{a}	0.80 ± 0.01^{a}	3.70 ± 0.02^{a}	0.05 ± 0.00^{a}
1	1	60	20	8.27 ± 0.04^{a}	$75.75 \pm 0.43^{g,h}$	3.79 ± 0.01^{b}	451.33 ± 1.69^{b}	38.85 ± 0.16^{b}	48.53 ± 0.13^{b}	0.92 ± 0.00^{b}
2	3	60	20	8.26 ± 0.06^{a}	$75.1 \pm 0.09^{d-g}$	$3.99 \pm 0.01^{e,f}$	821.14 ± 2.87^{j}	91.26 ± 0.39^{h}	110.36 ± 0.34^{h}	2.14 ± 0.00^{j}
3	5	60	20	8.26 ± 0.03^{a}	$74.85 \pm 0.24^{b-g}$	$4.08 \pm 0.02^{h,i}$	964.89 ± 3.15^{n}	111.64 ± 0.61^{L}	134.38 ± 0.63^{L}	2.60 ± 0.02^{n}
4	1	70	20	8.27 ± 0.06^{a}	$75.45 \pm 0.19^{f-h}$	3.89 ± 0.01^{c}	611.43 ± 4.20^{d}	61.53 ± 0.43^{c}	75.32 ± 0.21^{c}	1.45 ± 0.01^{d}
5	3	70	20	8.26 ± 0.04^{a}	$75.22 \pm 0.22^{d-h}$	$3.96 \pm 0.01^{d,e}$	754.56 ± 1.55^{h}	81.81 ± 0.16^{f}	99.23 ± 0.23^{f}	1.90 ± 0.00^{h}
6	5	70	20	8.26 ± 0.05^{a}	$74.84 \pm 0.20^{b-g}$	$4.08 \pm 0.01^{h,i}$	967.34 ± 4.03^{n}	111.94 ± 0.32^{L}	$134.78 \pm 0.58^{L,m}$	2.62 ± 0.00^{n}
7	1	80	20	8.26 ± 0.05^{a}	$75.32 \pm 0.17^{e-h}$	3.94 ± 0.02^{d}	709.83 ± 2.97^{g}	75.44 ± 0.29^{e}	91.74 ± 0.06^{e}	1.78 ± 0.01^{g}
8	3	80	20	8.26 ± 0.01^{a}	$75.03 \pm 0.11^{c-g}$	$4.03 \pm 0.01^{f,g}$	872.72 ± 4.77^{k}	98.53 ± 0.26^{i}	118.93 ± 0.54^{i}	2.28 ± 0.01^{k}
9	5	80	20	8.26 ± 0.04^{a}	$74.61 \pm 0.49^{a-f}$	$4.16 \pm 0.02^{k,l}$	1116.51 ± 3.95^{q}	133.10 ± 0.41^{o}	159.73 ± 0.65^{p}	3.07 ± 0.01^{q}
10	1	60	35	8.27 ± 0.03^{a}	$75.49 \pm 0.20^{f-h}$	3.88 ± 0.02^{c}	612.67 ± 1.04^{c}	61.71 ± 0.13^{c}	75.50 ± 0.31^{c}	1.41 ± 0.00^{c}
11	3	60	35	8.26 ± 0.03^{a}	$75.24 \pm 0.17^{d-h}$	$3.96 \pm 0.03^{d,e}$	752.10 ± 3.63^{h}	81.47 ± 0.24^{f}	98.80 ± 0.47^{f}	1.90 ± 0.01^{h}
12	5	60	35	8.26 ± 0.03^{a}	$74.85 \pm 0.31^{b-g}$	$4.09 \pm 0.02^{i,j}$	976.83 ± 4.85^{n}	113.32 ± 0.58^{L}	136.37 ± 0.62^{m}	2.62 ± 0.01^{n}
13	1	70	35	8.27 ± 0.02^{a}	$75.40 \pm 0.36^{e-h}$	3.89 ± 0.01^{c}	652.63 ± 1.31^{e}	67.39 ± 0.18^{d}	82.14 ± 0.35^{d}	1.56 ± 0.00^{e}
14	3	70	35	8.26 ± 0.03^{a}	$74.97 \pm 0.37^{b-g}$	$4.04 \pm 0.02^{g,h}$	894.24 ± 1.52^{L}	101.64 ± 0.27^{j}	122.57 ± 0.55^{j}	2.34 ± 0.00^{L}
15	5	70	35	8.26 ± 0.01^{a}	$74.77 \pm 0.18^{a-g}$	$4.13 \pm 0.01^{j,k}$	1023.11 ± 3.57^{o}	119.87 ± 0.40^{m}	144.12 ± 0.66^{n}	2.79 ± 0.01^{o}
16	1	80	35	8.26 ± 0.06^{a}	$75.32 \pm 0.24^{e-h}$	3.94 ± 0.01^{d}	702.11 ± 2.38^{f}	74.31 ± 0.19^{e}	90.44 ± 0.55^{e}	1.73 ± 0.01^{f}
17	3	80	35	8.26 ± 0.02^{a}	$74.95 \pm 0.20^{b-g}$	$4.04 \pm 0.01^{g,h}$	918.56 ± 1.55^{m}	105.00 ± 0.37^{k}	126.62 ± 0.83^{k}	2.44 ± 0.01^{m}
18	5	80	35	8.26 ± 0.01^{a}	$74.59 \pm 0.25^{a-f}$	$4.15 \pm 0.01^{k,l}$	1128.74 ± 6.31^{r}	134.84 ± 0.75^{p}	161.74 ± 0.25^{q}	3.14 ± 0.01^{r}
19	1	60	50	8.26 ± 0.02^{a}	$75.21 \pm 0.44^{d-h}$	$3.96 \pm 0.01^{d,e}$	768.78 ± 5.29^{i}	83.83 ± 0.25^{g}	101.61 ± 0.39^{g}	1.96 ± 0.01^{i}
20	3	60	50	8.26 ± 0.02^{a}	$74.54 \pm 0.10^{a-f}$	4.18 ± 0.01^{L}	1153.46 ± 4.85^{p}	138.34 ± 0.44^{q}	165.87 ± 0.60^{r}	3.24 ± 0.02^{s}
21	5	60	50	8.26 ± 0.07^{a}	$74.05 \pm 0.58^{a-c}$	4.33 ± 0.03^{o}	1435.93 ± 6.94^{s}	178.37 ± 0.56^{t}	213.10 ± 0.58^{u}	4.14 ± 0.02^{v}
22	1	70	50	8.26 ± 0.03^{a}	$74.95 \pm 0.24^{b-g}$	$4.06 \pm 0.02^{g-i}$	918.55 ± 3.08^{m}	105.05 ± 0.41^{k}	126.62 ± 0.57^{k}	2.41 ± 0.00^{m}
23	3	70	50	8.26 ± 0.02^{a}	$74.25 \pm 0.38^{a-d}$	4.28 ± 0.01^{n}	1326.94 ± 5.75^{r}	162.94 ± 0.63^{s}	194.87 ± 0.91^{t}	3.78 ± 0.01^{u}
24	5	70	50	8.26 ± 0.04^{a}	73.95 ± 0.34^{ab}	4.37 ± 0.01^{o}	1499.18 ± 8.19^{t}	187.34 ± 0.08^{u}	223.64 ± 0.80^{v}	4.34 ± 0.03^{w}
25	1	80	50	8.26 ± 0.03^{a}	$74.68 \pm 0.29^{a-f}$	$4.14 \pm 0.02^{k,l}$	1075.94 ± 3.87^{p}	127.31 ± 0.53^{n}	152.94 ± 0.75^{o}	2.96 ± 0.02^{p}
26	3	80	50	8.26 ± 0.06^{a}	$74.40 \pm 0.23^{a-e}$	4.23 ± 0.02^{m}	1225.99 ± 3.93^{q}	148.61 ± 0.62^{r}	178.01 ± 0.87^{s}	3.45 ± 0.02^{t}
27	5	80	50	8.25 ± 0.05^{a}	73.78 ± 0.19^{a}	4.42 ± 0.02^{p}	1578.76 ± 3.16^{u}	198.64 ± 1.43^{v}	236.97 ± 0.75^{w}	4.57 ± 0.01^{x}

$^{a-x}$Different letters in superscript of the same table column indicate on the statistically significant difference between values, at level of significance of $p < 0.05$ (based on *post hoc* Tukey HSD test).

TABLE 3 The ANOVA calculation for the osmotic dehydration and lyophilization of peach

Technological parameters	Term	df[a]	Sum of squares											
			Osmotic dehydration process responses		Osmodehydrated and lyophilized samples' responses			Chemical content			Mineral matter content			
			DMC	a_w	DMC_L	WL_L	a_{wL}	Proteins	Sugar	Ash	K	Ca	Mg	Fe
Time	Linear	1	1908.19*	0.002838*	918.904*	178.7421*	0.095193*	0.000133*	2.944356*	0.299538*	974417*	19,578.25*	27,221.44*	10.44245*
	Quadratic	1	15.60*	0.000091*	10.225	0.5660	0.003098*	0.000000	0.003919	0.000258	1110	22.32	30.90	0.01402
Concentration	Linear	1	76.71*	0.000242*	617.944*	259.2058*	0.091164*	0.000022*	0.320000*	0.034497*	107653*	2155.84*	3005.71*	1.12001*
	Quadratic	1	0.469	0.000009	1.233	0.1812	0.000456	0.000001	0.000474	0.000143	16	0.37	0.45	0.00002
Temperature	Linear	1	635.40*	0.001682*	730.340*	3.3035	0.092021*	0.000108*	2.247200*	0.233928*	766231*	15,390.18*	21,398.98*	8.12045*
	Quadratic	1	3.316	0.000009	15.092	32.5574*	0.001360	0.000024*	0.496896*	0.050539*	159121*	3195.96*	4446.84*	1.76042*
Cross product	Time x Conc.	1	4.301	0.000007	0.427	2.0184	0.000114	0.000001	0.010800	0.002187	3631	72.13	101.44	0.04813
	Time x Temp.	1	43.51	0.000000	8.545	13.4917	0.000331	0.000009*	0.058800*	0.005720*	18759*	377.44*	523.25*	0.20803*
	Conc. x Temp.	1	9.98*	0.000001	9.821	39.6059*	0.004033*	0.000003	0.003333	0.000192	308	6.31	8.69	0.00241
Error	Residual variance	17	58.79	0.000276	60.794	89.0408	0.010797	0.000025	0.186341	0.018290	63,523	1277.42	1773.90	0.65634
	Total sum of squares	26	2756.28	0.005154	918.904	618.7126	0.298568	0.000326	6.272119	0.645293	2,094,770	42,076.22	58,511.60	22.37227
r^2			.979	.947	.974	.856	.964	.924	.970	.972	.970	.969	.970	.971

[a]Degrees of freedom.

*Statistically significant at level of $p < .05$.

TABLE 4 Second-order polynom regression coefficients for 12 responses of osmotic dehydration and lyophilization processes, chemical and mineral matter content of peach samples

	Y_1 (DMC)	Y_2 (a_w)	Y_3 (DMC_L)	Y_4 (WL_L)	Y_5 (a_{wL})	Y_6 (Proteins)	Y_7 (Sugar)	Y_8 (Ash)	Y_9 (K)	Y_{10} (Ca)	Y_{11} (Mg)	Y_{12} (Fe)
β_0	2.091081	1.039205*	−43.5699	−45.6610	2.257937*	8.241146*	76.12997*	3.500392*	303.3948	17.58031	23.96728	0.577824
β_1	3.249876	−0.014542*	3.8860	0.6362	−0.075111*	0.000881	−0.26389	0.096111*	151.4943	21.42111	25.32069	0.521389*
β_{11}	−0.403138*	0.000972*	−0.3264	0.0768	0.005681*	−0.000041	0.00639	−0.001639	−3.4008	−0.48222	−0.56736	−0.012083
β_2	0.295309	−0.002249	1.4035	1.1082	−0.024068	0.000456	−0.02639	0.012314	11.4678	1.63908	1.91404	0.033472
β_{22}	−0.002796	0.000012	−0.0045	−0.0017	0.000087	−0.000003	0.00009	−0.000049	−0.0165	−0.00247	−0.00274	−0.000017
β_3	−0.451250	−0.001125	1.2559*	1.7071*	−0.017481*	0.000784*	0.08075*	−0.025003*	−43.2287*	−6.13007*	−7.22841*	−0.143519*
β_{33}	0.003304	0.000005	−0.0070	−0.0104*	0.000067	−0.000009*	−0.00128*	0.000408*	0.7238*	0.10258*	0.12100*	0.002407*
β_{12}	0.029935	0.000037	0.0094	−0.0205	0.000154	−0.000014	0.00150	−0.000675	−0.8697	−0.12258	−0.14537	−0.003167
β_{13}	0.063474*	−0.000006	0.0281	−0.0353	−0.000175	−0.000029*	−0.00233*	0.000728*	1.3179*	0.18694*	0.22011*	0.004389*
β_{23}	0.006080	0.000002	−0.0060	−0.0121*	0.000122*	−0.000003	−0.00011	0.000027	0.0338	0.00483	0.00567	0.000094

*Statistically significant at level of $p < .05$.

lyophilization of sample osmodehydrated during 1 hr, in molasses of 70% concentration, at 35°C (WL_L = 42.63%), where DMC value was only 18.42%, reaching 61.04% after lyophilization stage. With the increase of osmodehydration stage duration, the water removal in the lyophilization stage (indicated with WL_L values, or increase of DMC_L values) has statistically significantly decreased. This dehydration rate reduction of the lyophilization stage can be explained by the different accessibility of present water in samples (Mathlouthi, 2011) dehydrated at different osmodehydration parameters. In osmodehydration processes, where lower DMC values were obtained, more free or less-bound water was preserved in osmodehydration samples, where in successive lyophilization stage, this water was easily removed. In osmodehydration processes, where higher DMC values were obtained, only strong-bound water, harder to remove, remained for the lyophilization stage to be removed; hence, the lower dehydration rates of the lyophilization stage were determined.

Dehydration effectiveness contribution of osmodehydration process, as a pretreatment to lyophilization, can be best seen by analyzing the level of obtained DMC of the peach samples, subjected only to lyophilization stage. Obtained DMC values of single-stage lyophilized samples were up to 5.45 times lower than in the dehydration processes with osmotic dehydration. This result can also be viewed from the perspective of time and energy consumption reduction in the lyophilization process, since obtaining the same level of moisture content in a single-stage lyophilization process, would require much longer time and energy consumption.

Similar to DMC_L values, a_{wL} values were statistically significantly affected by all three varied osmodehydrated process parameters, Table 1. Trends of the effects are the same as in case of a_w values, with the increase of all three osmodehydration parameters' values, lyophilized peach samples' a_{wL} values statistically significantly decreased, reaching minimal value of 0.433. This obtained a_w value provides exceptional microbiological stability, since it is below growth limiting a_w value for all microorganisms (Tortora et al., 2013).

The lyophilization stage of dehydration had led to much more significant peach samples a_w values reduction than the osmotic dehydration stage. Peach samples' a_w values had lowered for up to 0.431 units in the lyophilization stage, where maximum of a_w values reduction in the osmodehydration process was for 0.076 units.

From the presented results of protein content, Table 2, it can be seen that there was no statistically significant difference of fresh and treated peach samples, indicating that the protein content of fresh peaches was preserved throughout the successive dehydration processes.

From the results of the sugar content, it can be seen that by applying the highest values of osmodehydration technological parameters, sugar content has statistically significantly decreased in comparison to fresh peach samples. These results indicate that high initial sugar content of fresh peach samples (Colarič et al., 2004) was lowered for up to 3.21% after dehydration treatments. Two mass transfers of the osmodehydration process (Filipović et al., 2014) can lead to this sugar content balance. Water loss from the osmodehydrating peach material can cause dissolved sugar leakage, while solid gain from molasses, as an osmotic solution, can replace the part of the lost sugar content, considering molasses' high sugar content (Šarić et al., 2016).

Ash content has statistically significantly increased in treated peach samples in comparison to the fresh peach, Table 2, also as a result of osmotic dehydration stage of dehydration. Secondary mass transfer of osmodehydration process (Filipović et al., 2014) has incorporated, via solid gain, molasses high ash content (Šarić et al., 2016), in dry matter of dehydrated peach samples, increasing it up to 21.43% in comparison to the untreated peach samples.

The changes of dehydrated peach samples' mineral matter content are much more profound than of the chemical content as can be seen from Table 2. All four mineral matter content responses were statistically significantly affected by all three osmodehydration process parameters. The increase of time, concentration, and temperature had led to a statistically significant increase of K, Ca, Mg, and Fe content in dehydrated (osmodehydrated and lyophilized) peach samples. Maximal values of all peach samples' mineral matter content responses were obtained after 5-hr osmotic dehydration process in molasses of maximal concentration (80%), at a maximal process temperature of 50°C, and successive 5-hr lyophilization. Values were up to 8.63, 248.30, 64.05, and 101.56 times higher, in dehydrated than in fresh (untreated) peach samples, for K, Ca, Mg, and Fe, respectively.

This high increase of mineral matter content of dehydrated peach samples can be attributed to the osmotic dehydration stage of the process, especially to the secondary mass transfer, which supplements dehydrating material with osmotic solutions' (molasses) dry matter components (Yadav & Singh, 2014). Molasses, as an osmotic solution, besides its good technological effectiveness, provides enrichment of dehydrating material with its favorable nutritive composition (Nićetin et al., 2017; Šarić et al., 2016), which can be seen from the results of dehydrated peach samples mineral matter composition.

The ANOVA calculation, presented in Table 3, showed that the second-order polynom models for all responses were found to be statistically significant and the response surfaces were fitted to these models.

Osmodehydration process time had shown to be the most influential independent variable, then osmodehydration process temperature and the least influential independent variable had shown to be molasses' concentration, on all tested responses, except for WL_L. In case of WL_L, the influential hierarchy was as follows: concentration, time, and temperature.

Linear terms of time, concentration, and temperature statistically significantly contributed to all tested responses models forming, except for linear terms of temperature for WL_L response.

The quadratic term of time was statistically significant for responses of DMC, a_w, and a_{wL}, while the quadratic term of temperature was statistically significant for responses of WL_L and all responses of chemical and mineral content. For cross products, the statistically significant terms were time × temperature for all responses of chemical and mineral content, and concentration × temperature for DMC, WL_L, and a_{wL} responses.

The residual variance is also shown in Table 3, where the lack of fit represents other contributions of higher order terms. A statistically 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 (Madamba, 2002). In this research, all second-order polynom models had an 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. It is also the proportion of the variability in the response variable that is accounted for by the regression analysis. A high r^2 is indicative that the variation was accounted for and that the data fitted satisfactorily to the proposed model (Nićetin et al., 2017).

The r^2 values ranged from .856 (for WL_L) to .979 (for DMC), showing good fit of experimental results to all calculated models.

Regression coefficients, presented in Table 4, can be used for completing quadratic Equation (4), which describe mathematical models of different peach dehydration responses. Solving these equations with input values of independent variables (osmodehydration process time, molasses' concentration, and osmodehydration process temperature) values of desired responses (osmotic dehydration and lyophilization processes responses, chemical and mineral matter content) can be calculated. In that way, values of investigated responses can be predicted in the ranges of values of independent variables for which mathematical models were developed.

From the presented results, it can be concluded that all three osmotic dehydration parameters statistically significantly affected DMC and a_w of successively dehydrated peach samples.

Osmodehydration process, as a pretreatment to lyophilization, contributed to upgrading overall dehydration effectiveness, by increasing obtained DMC values of successive dehydration process, reducing time and energy consumption of high energy demanding single-stage lyophilization process.

Exceptional peaches samples' a_w values reduction in lyophilization stage contributed to the synergetic dehydration method with samples' microbiological stability, obtaining a dehydrated product of only 0.433 of a_w value.

The chemical content of dehydrated peach samples was preserved, while mineral matter content was highly increased, as a direct consequence of molasses application, as an osmotic solution, in the osmodehydration stage. In this manner new, nutritive improved, microbiologically safe peach product is produced.

By applying response surface methodology, mathematical models of 12 responses of osmotic dehydration and lyophilization processes, chemical and mineral matter content of peach samples were developed, where testing all developed models showed statistical significance. Mathematical models described the synergistic performance of two successive dehydration methods in well manner. Predicted and observed responses had good correlation, allowing good prediction of values of investigated responses based on the ranges of applied technological parameters, as independent variables.

ACKNOWLEDGMENTS

Presented results are part of the research project titled: "Production and implementation of innovative product from domestic peach with improved sensory and nutritive properties", funded by the Provincial Secretariat of Higher Education and Scientific Research, Autonomous Province of Vojvodina, Republic of Serbia, contract number: 142-451-2289/2021-01/02.

CONFLICT OF INTEREST

The authors have declared no conflicts of interest for this article.

AUTHOR CONTRIBUTIONS

Vladimir S Filipović: Conceptualization; funding acquisition; investigation; methodology; writing – review and editing. **Jelena S Filipović:** Investigation; supervision; writing – original draft; writing – review and editing. **Biljana Loncar:** Investigation; writing – original draft; writing – review and editing. **Violeta M. Knežević:** Investigation; project administration; writing – review and editing. **Milica R. Nicetin:** Investigation; writing – review and editing. **Ivana Filipović:** Formal analysis; investigation; writing – review and editing.

DATA AVAILABILITY STATEMENT

Data openly available in a public repository that issues datasets with DOIs.

ORCID

Vladimir Filipović https://orcid.org/0000-0002-2276-7371
Ivana Filipović https://orcid.org/0000-0002-0980-1889

REFERENCES

American Association of Cereal Chemists (AACC). (2000a). *Method No. 46–13, Crude protein-Micro-Kjeldahl method*. Approved methods of the AACC (10th ed.). AACC International.

American Association of Cereal Chemists (AACC). (2000b). *Method No. 08–01, Ash-Basic Method*. Approved methods of the AACC (10th ed.). AACC International.

American Association of Cereal Chemists (AACC). (2009). *Method No. 80–68.01, Determination of Reducing Sugars—Schoorl Method*. Approved methods of the AACC (11th ed.). AACC International.

Blanda, G., Cerretani, L., Cardinali, A., Barbieri, S., Bendini, A., & Lercker, G. (2009). Osmotic dehydrofreezing of strawberries: Polyphenolic content volatile profile and consumer acceptance. *LWT-Journal of Food Science and Technology*, *42*, 30–36. https://doi.org/10.1016/j.lwt.2008.07.002

Ciurzyńska, A., Kowalska, H., Czajkowska, K., & Lenart, A. (2016). Osmotic dehydration in production of sustainable and healthy food. *Trends in Food Science & Technology*, *50*, 186–192. https://doi.org/10.1016/j.tifs.2016.01.017

Colarič, M., Štampar, F., & Hudina, M. (2004). Contents of sugars and organic acids in the cultivars of peach (Prunuspersica L.) and nectarine (Prunuspersica var. nucipersica Schneid.). *Acta agriculturae slovenica*, *83*, 53–61. http://aas.bf.uni-lj.si/junij2004/07colaric.pdf

Ćurčić, B., Pezo, L., Filipović, V., Nićetin, M., & Knežević, V. (2015). Osmotic treatment of fish in two different solutions-artificial neural network model. *Journal of Food Processing and Preservation*, *39*, 671–680. https://doi.org/10.1111/jfpp.12275

Da Costa Ribeiro, A. S., Aguiar-Oliveira, E., & Maldonado, R. R. (2016). Optimization of osmotic dehydration of pear followed

by conventional drying and their sensory quality. *LWT-Food Science and Technology*, *72*, 407–415. https://doi.org/10.1016/j.lwt.2016.04.062

Fahloul, D., Lahbari, M., Benmoussa, H., & Mezdour, S. (2009). Effect of osmotic dehydration on the freeze drying kinetics of apricots. *Journal of Food, Agriculture & Environment*, *7*, 117–121. https://doi.org/10.1234/42009.1556

Filipović, I., Ćurčić, B., Filipović, V., Nićetin, M., Filipović, J., & Knežević, V. (2017). The effects of technological parameters on chicken meat osmotic dehydration process efficiency. *Journal of Food Processing and Preservation*, *41*, 1–7. https://doi.org/10.1111/jfpp.13116

Filipović, V., Lević, L. J., Ćurčić, B., Nićetin, M., Pezo, L., & Mišljenović, N. (2013). Optimisation of mass transfer kinetics during osmotic dehydration of pork meat cubes in complex osmotic solution. *Chemical Industry and Chemical Engineering Quarterly*, *20*, 305–314. https://doi.org/10.2298/CICEQ120511012

Filipović, V., Lončar, B., Nićetin, M., Knežević, V., Filipović, I., & Pezo, L. (2014). Modeling Counter-Current Osmotic Dehydration Process of Pork Meat in Molasses. *Journal of Food Process Engineering*, *37*, 533–542. https://doi.org/10.1111/jfpe.12114

Igual, M., Cebadera, L., Ma Cámara, R., Agudelo, C., Martínez-Navarrete, N., & Cámara, M. (2019). Novel ingredients based on grapefruit freeze-dried formulations: nutritional and bioactive value. *Food*, *8*, 506–528. https://doi.org/10.3390/foods8100506

ISO 6869: 2000 *Animal feeding stuffs—Determination of the contents of calcium, copper, iron, magnesium, manganese, potassium, sodium and zinc—Method using atomic absorption spectrometry.*

Knežević, V., Pezo, L., Lončar, B., Filipović, V., Nićetin, M., Gorjanović, S., & Šuput, D. (2019). Antioxidant capacity of nettle leaves during osmotic treatment. *Periodica Polytechnica Chemical Engineering.*, *63*, 491–498. https://doi.org/10.3311/PPch.12688

Liu, Y., Zhao, Y., & Feng, X. (2008). Energy analysis for a freeze-drying process. *Appllied Thermal Engineering*, *28*, 675–690. https://doi.org/10.1016/j.applthermaleng.2007.06.004

Madamba, P. (2002). The response surface methodology: An application to optimize dehydration operations of selected agricultural crops. *LWT - Food Science and Technology*, *35*, 584–592. https://doi.org/10.1006/fstl.914

Mandala, G., Anagnostaras, F., & Oikonomou, K. (2005). Influence of osmotic dehydration conditions on apple air-drying kinetics and their quality characteristics. *Journal of Food Engineering*, *69*, 307–316. https://doi.org/10.1016/j.jfoodeng.2004.08.021

Mathlouthi, M. (2011). Water content, water activity, water structure and the stability of foodstuffs. *Food Control*, *12*, 409–417. https://doi.org/10.1016/S0956-7135(01)00032-9

Mišljenović, N., Koprivica, G., Pezo, L., Lević, L., Ćurčić, B., Filipović, V., & Nićetin, M. (2012). Optimization of the osmotic dehydration of carrot cubes in sugar beet molasses. *Thermal Science*, *16*, 43–52. https://doi.org/10.2298/TSCI110808129M

Moreno, J., Simpson, R., Pizarro, N., Pavez, C., Dorvil, F., & Petzold, G. (2013). Influence of ohmin heating/osmotic dehydration treatments on polyphenoloxidase inactivation, physical properties and microbial stability of apples (cv. Granny Smith). *Innovative Food Science and Emerging Technologies*, *20*, 198–207. https://doi.org/10.1016/j.ifset.2013.06.006

Nićetin, M., Pezo, L., Lončar, B., Filipović, V., Šuput, D., Knežević, V., & Filipović, J. (2017). The possibility to increase antioxidant activity of celery root during osmotic treatment. *Journal of Serbian Chemical Society*, *82*, 253–265. https://doi.org/10.2298/JSC161020015N

Nićetin, M., Pezo, L., Filipović, V., Lončar, B., Filipović, J., Šuput, D., & Knežević, V. (2021). The effects of solution type temperature and time on antioxidant capacity of osmotically dried celery leaves. *Thermal Science*, *25*, 1759–1770. https://doi.org/10.2298/TSCI191101184N

Shi, J., & Xue, S. J. (2009). Application and development of osmotic dehydration technology in food processing. In C. Ratti (Ed.), *Advances in food dehydration* (pp. 190–231). Taylor & Francis Group.

Šarić, L., Filipčev, B., Šimurina, O., Plavšić, D., Šarić, B., Lazarević, J., & Milovanović, I. (2016). Sugar beet molasses: Properties and applications in osmotic dehydration of fruits and vegetables. *Food and Feed Research*, *43*, 135–144. https://doi.org/10.5937/FFR1602135%C5%A0

Tortora, G., Funke, B., & Case, C. (2013). *Microbiology an introduction (11th ed, 153–158, 153–158)*. Pearson Education.

Yadav, A. K., & Singh, S. V. (2014). Osmotic dehydration of fruits and vegetables: a review. *Journal of Food Scince and Technology*, *51*, 1654–1673. https://doi.org/10.1007/s13197-012-0659-2

Yadav, B., Yadav, R., & Jatain, M. (2012). Optimization of osmotic dehydration conditions of peach slices in sucrose solution using response surface methodology. *Journal of Food Science and Technology*, *49*, 547–555. https://doi.org/10.1007/s13197-011-0298-z

How to cite this article: Filipović, V., Filipović, J., Lončar, B., Knežević, V., Nićetin, M. & Filipović, I. (2022). Synergetic dehydration method of osmotic treatment in molasses and successive lyophilization of peaches. *Journal of Food Processing and Preservation*, 46, e16512. https://doi.org/10.1111/jfpp.16512