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Osmotic dehydration of wild garlic in sucrose–salt solution

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Abstract. Due to its nutritional and nutraceutical qualities, wild garlic (*Allium ursinum* L.) has great potential for use in the food and pharmaceutical industries. The availability of this plant is limited to the spring season, and it is perishable immediately after harvest. Osmotic dehydration (OD) is verified as an effective pre-treatment to improve sustainability by reducing the water content of raw material with minimal negative effect on its nutritive and sensorial qualities. In this study, OD of wild garlic leaves in aqueous solution of sucrose and salt was analysed at three temperatures and after diverse immersion times. The effects of the processing time and temperature on the dry matter content, water loss, and solid gain were evaluated using response surface methodology and analysis of variance. Some components in the samples were determined before and after OD. The results showed that during OD, the dry matter content of wild garlic increased from 7.91 ± 1.08% to 51.51 ± 1.34%. The maximum achieved values for water loss (0.6189 ± 0.0146 g/g i.s.) and solid gain (0.2417 ± 0.0146 g/g i.s.) indicated a good dehydration level. In the osmotically dehydrated wild garlic, the amount of analysed minerals decreased, sodium and sugar increased, and the content of protein, cellulose, and fat did not change – compared to the fresh sample.

Keywords and phrases: *Allium ursinum*, osmotic solution, mass transfer kinetics, chemical and mineral content

1. Introduction

Wild garlic (*Allium ursinum* L.), a medicinal and dietary plant with a long tradition of use, grows spontaneously on fertile soils in shady, humid places and forests of Europe and northern Asia (*Ivanova et al.*, 2009; *Krivokapić et al.*, 2020). In Serbia, during the vegetation cycle of this plant – which starts in the early spring and ends at the beginning of the summer –, this species covers a large forest complex forming a dense population, expressed in hectares (*Djurdjević et al.*, 2004; *Tomšik et al.*, 2017). The leaves are becoming increasingly popular in culinary use as vegetable, salad, spice, or an ingredient in traditional dishes (*Wu et al.*, 2009; *Šobot et al.*, 2019). Sulphur-containing compounds (predominantly alliins and methilallins) and phenolic compounds (primarily kaempferol derivatives and phenolic acids) are related to the health benefits of wild garlic and the growing interest for exploitation of this plant in the food and pharmaceutical industries (*Pejatović et al.*, 2017; *Pavlović et al.*, 2017). The limiting factor is the short period of its availability (3–3.5 months) (*Schmitt et al.*, 2005). For medical purposes, this period is limited from April to the first half of May, since the highest content of some compounds with great therapeutic potential is present in fresh leaves before flowering (*Sobolewska et al.,* 2015). Also, following harvest, this plant has a very short shelf life. *Tomšik et al.* (2016) report that a bunch of wild garlic immersed in water becomes commercially unacceptable after more than 5 days of storage at room temperature due to the very intensive yellowing and wilting and the appearance of signs of rotting of the leaves.

Drying can extend the shelf life of wild garlic and ensure its availability throughout the year, which allows for the use of this plant in food and pharmaceutical industries. It is essential to find an adequate drying method with reduced time and energy consumption, which avoids the negative effects of temperature on thermosensitive compounds and enables the preservation of sensory, nutritional, and functional properties of plants (*Filipović et al.*, 2022). Osmotic dehydration (OD) provides a decrease in drying time as well as final moisture content, the reduction of nutritional and sensory losses of the food product – at low (or at room) temperature and with low energy consumption (*Champawat et al.*, 2019; *Leahu et al.*, 2020). In this process, the immersion of raw material in a hypertonic solution leads to the flow of water through cell membranes along with natural solutes from the intercellular space of the material into the surrounding solution, by means of osmosis (*Nićetin et al.*, 2021). Simultaneously, the plant material's impregnation by the solutes from osmotic solution occurs (*Sereno et al.*, 2001; *Lončar et al.*, 2022). The two most commonly used solutes in the preparation of hypertonic solutions are sucrose and sodium chloride, but many authors proved that the combination of these two solutes is the best choice in terms of effectiveness of OD (higher water losses with lower solid gain), convenience (increase in total solution concentration without reaching the saturation limits), and flavour (without significantly affecting osmodehydrated product taste) (*Akbarian et al.*, 2014; *Kvapil et al.*, 2020).

Due to the lack of data in literature on the OD of wild garlic in sucrose–salt solution, the aim of this work was to examine the mass transfer kinetics and chemical composition of wild garlic leaves during OD in an aqueous solution of sucrose and salt.

2. Materials and methods

The samples for investigation (*Allium ursinum*) were harvested from a forest area near Novi Sad, Serbia (45º08'34.6"N; 19º36'55.0"E), in April 2018. Fresh leaves were separated, washed under running water, wiped, and cut into squares with a dimension of 1 x 1 cm. The osmotic solution was prepared using an electric propeller mixer, which completely dissolved 350 g of commercial sodium chloride and 1,200 g of commercial sucrose in 1 kg of distilled water. These quantities were chosen based on the maximum solubility of salt and sucrose in water at room temperature in order to obtain a maximum osmotic solution concentration of 60% w/w (these three components were mixed in the following ratio: sucrose 47.04%, NaCl 13.72%, and distilled water 39.2%). Previously weighted samples (5 g) were immersed in nine laboratory vessels filled with prepared osmotic solution (100 g), maintaining in each vessel the 1:20 wild garlic/osmotic solution ratio in order to minimize changes in the solution concentration, which could lead to a local reduction in the osmotic driving force during the process. The experiments were carried out at temperatures of 20°C, 35°C, and 50°C, kept constant in an incubator (In 160, Memmert, Schwabach, Germany), under atmospheric pressure, for 1, 2.5, and 4 hours resp., with manual mixing every 15 minutes for the better homogenization of the solution and defused water from the dehydrated samples of wild garlic, thus to enhance the mass transfer. The intensity, duration, and frequency of the mixing were the same for the samples under all temperatures and immersion times in order to make the results comparable. After the selected time intervals, samples were removed from the osmotic solution and rinsed, and the free water on the surfaces was carefully collected with paper towels and measured. The content of solids in fresh and osmodehydrated samples was determined gravimetrically by drying at 105°C for 24 hrs in an oven (Instrumentaria, Sutjeska, Serbia) until a constant weight was achieved in accordance with the AOAC method No. 925.10 (2).

In order to describe the effectiveness of the mass transfer during the OD process, specific kinetic parameters (dry matter content (DMC), water loss (WL), and solid gain (SG)) were calculated using equations described by *Filipović et al.* (2017):

$$\text{DMC} = \frac{m_d}{m_i} \; [100\%] \tag{1}$$

$$\text{WL} = \frac{m_i z_i - m_f z_f}{m_i} \left[\frac{\text{g}}{\text{g}_{\text{i.s.}}} \right] \tag{2}$$

$$\text{SG} = \frac{m_f s_f - m_i s_i}{m_i} \left[\frac{\text{g}}{\text{g}_{\text{i.s.}}} \right] \tag{3}$$

where m_d is the mass of dry matter for the final sample, m_i and m_f are the initial and final mass (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 solid (g total solid/g sample) respectively, and $g_{i.s.}$ is the mass (g) of the initial sample. These three key parameters (DMC, WL, and SG) for the characterization of mass transfer during OD were determined for all three selected temperatures and processing times and expressed as mean values of three repeated measurements with standard deviations.

The determination of the composition of selected chemical and mineral components was conditioned by the attempt to monitor the changes caused by the mass transfer during the OD. The proximate composition of the analysed components of wild garlic sample before and after OD in the sucrose–salt solution was determined in accordance with standard AOAC methods (2000) for protein (method No. 950.36), fat (method No. 935.38), cellulose (method No. 973.18), starch content (method No. 996.11), reducing sugars (method No. 80-68), and ash (method No. 930.22). Each measurement was repeated six times. The mineral content – calcium (Ca), sodium (Na), zinc (Zn), copper (Cu), magnesium (Mg), and iron (Fe) – of samples was determined following standard methods described by AOAC (2000). Minerals were analysed by atomic absorption spectrophotometry (method No. 984.27) on a Varian Spectra AA 10 (Varian Techtron Pty Ltd., Mulgvare Victoria, Australia). Each measurement was performed in six replications.

In this study, StatSoft Statistica for Windows, ver. 10 program (Statistica, 2010) was applied for a full factorial experimental design, and the data were analysed using Response Surface Methodology (RSM) and the analysis of variance (ANOVA). The second-order polynomial (SOP) models were developed to relate the observed responses: DMC, WL, and SG with the two process variables: process time and temperature.

3. Results and discussions

In *Table 1*, mean values with standard deviations of the DMC, WL, and SG as responses of the wild garlic OD process are shown.

Table 1. Experimental results of dry matter content and kinetic parameters of wild garlic during osmotic dehydration in sucrose–salt solution

Sample	τ (h)	T (ºC)	DMC (%)	WL ($g/g_{i.s.}$)	SG ($g/g_{i.s.}$)
1.	0	20	7.91[a] ± 1.08	-	-
2.	1	20	17.84[b] ± 1.37	0.2538[a] ± 0.0112	0.0659[a]± 0.0011
3.	2.5	20	25.37[c] ± 2.03	0.3651[c] ± 0.0167	0.1098[bc] ± 0.0167
4.	4	20	30.36[d-f] ± 0.79	0.4086[d] ± 0.0058	0.1442[de] ± 0.0058
5.	1	35	20.80[b] ± 1.43	0.2978[b] ± 0.0113	0.0845[ab] ± 0.0113
6.	2.5	35	28.16[cd] ± 1.24	0.4100[d] ± 0.0088	0.1211[cd] ± 0.0088
7.	4	35	33.88[f] ± 1.19	0.4558[e] ± 0.0084	0.1592[e-g] ± 0.0084
8.	1	50	29.71[de] ± 0.09	0.4513[e] ± 0.0006	0.1194[cd] ± 0.0006
9.	2.5	50	41.25[g] ± 1.88	0.5783[f] ± 0.0110	0.1614[e-h] ± 0.0110
10.	4	50	51.51[h] ± 1.34	0.6189[gh] ± 0.0146	0.2417[j] ± 0.0146

Notes: [a–h] – the different letters in the superscript in the same column of the table indicate statistically significant difference between values at the level of significance of $p < 0.05$ (based on post-hoc Tukey HSD test); i.s. – initial sample.

The different applied parameters of the process temperature and time resulted in a statistically significant change in all analysed responses of the OD process: DMC, WL, and SG for osmotically dehydrated wild garlic leaves, at a significance level of $p < 0.05$ (*Table 1*). The increase in the content of dry matter in samples with the progress of the OD process is the result of water diffusion from wild garlic as well as of the impregnation of sucrose and salt from solution to the dehydrated material. Higher temperatures intensify the mass transfer during OD, increasing the permeability of cell membranes and reducing the osmotic solution viscosity, which facilitates the transport of water and solutes (*Tonon et al.*, 2007). The results reveal that DMC of wild garlic increases from 7.91 ± 1.08% (fresh sample) to 51.51 ± 1.34% (sample osmodehydrated 4 hrs at 50°C). According to the results, both kinetic parameters (WL and SG) studied as an indicator of the efficiency of OD rise with the increase of process time and temperature. The maximum values for WL (0.6189 ± 0.0146 g/g i.s.) and SG (0.2417 ± 0.0146 g/g i.s.) were achieved at the end of the 4 h process at the highest temperature of 50°C and indicated a good dehydration level. Similar results were also reported for the OD of celery leaves in aqueous solution of sucrose and salt – WL of 0.712 ± 0.006 g/g i.s. and SG of

0.240 ± 0.002 g/g i.s. (*Nićetin*, 2017) – and for nettle leaves osmodehydrated in the same solution – WL of 0.487 ± 0.001 g/g i.s. and SG of 0.260 ± 0.002 g/g i.s. (*Knežević et al.*, 2015). OD performed in the same solution but for animal material (pork meat) resulted in a similar value for SG (0.289 ± 0.012 g/g i.s), as observed by *Ćurčić et al.* (2014).

Table 2. ANOVA table of response values of the osmotic dehydration of wild garlic in sucrose–salt solution

Technolo-gical parameters	Term		Sum of squares		
		df+	DMC	WL	SG
Time	Linear	1	1483.085*	0.362417*	0.050002*
	Quadratic	1	89.544*	0.082539*	0.002231
Temperature	Linear	1	326.999*	0.051414*	0.005639*
	Quadratic	1	38.408	0.005052	0.000529
Cross product	Time x Temp.	1	107.724*	0.009007	0.002020
Error	Residual variance	6	52.311	0.023633	0.002262
	Total sum of squares	11	2051.315	0.521838	0.061641
	R^2		0.9745	0.95471	0.9633

* Statistically significant at a level of significance of $p < 0.05$; + df – degrees of freedom.

Table 2 shows the results of ANOVA of the RSM models that were developed on the basis of the experimental results shown in *Table 1*. Based on these results, the statistically significant effects of process parameters (time, temperature) as well as their interdependence on the responses of the mathematical model (dry matter content, water loss, solid gain) were analysed. The ANOVA test indicated that the values of the DMC, WL, and SG were statistically significantly ($p < 0.05$) influenced by both process parameters (linear terms of time and temperature), time being the most influential parameter. The quadratic term for time contributed statistically significantly to the formation of the SOP model for the prediction of DMC and WL, as a consequence of reducing mass transfer rates with the flow time of the process. The cross product of time and temperature was statistically significant ($p < 0.05$) and contributed to the forming of the SOP model only for DMC. Residual variances were not statistically significant, demonstrating that the applied model for DMC, WL, and SG was adequate for the OD of wild garlic leaves, with a high level of determination coefficient R^2 (0.97454, 0.95471, 0.9633). This indicated a good fitting of the SOP model with the obtained experimental values.

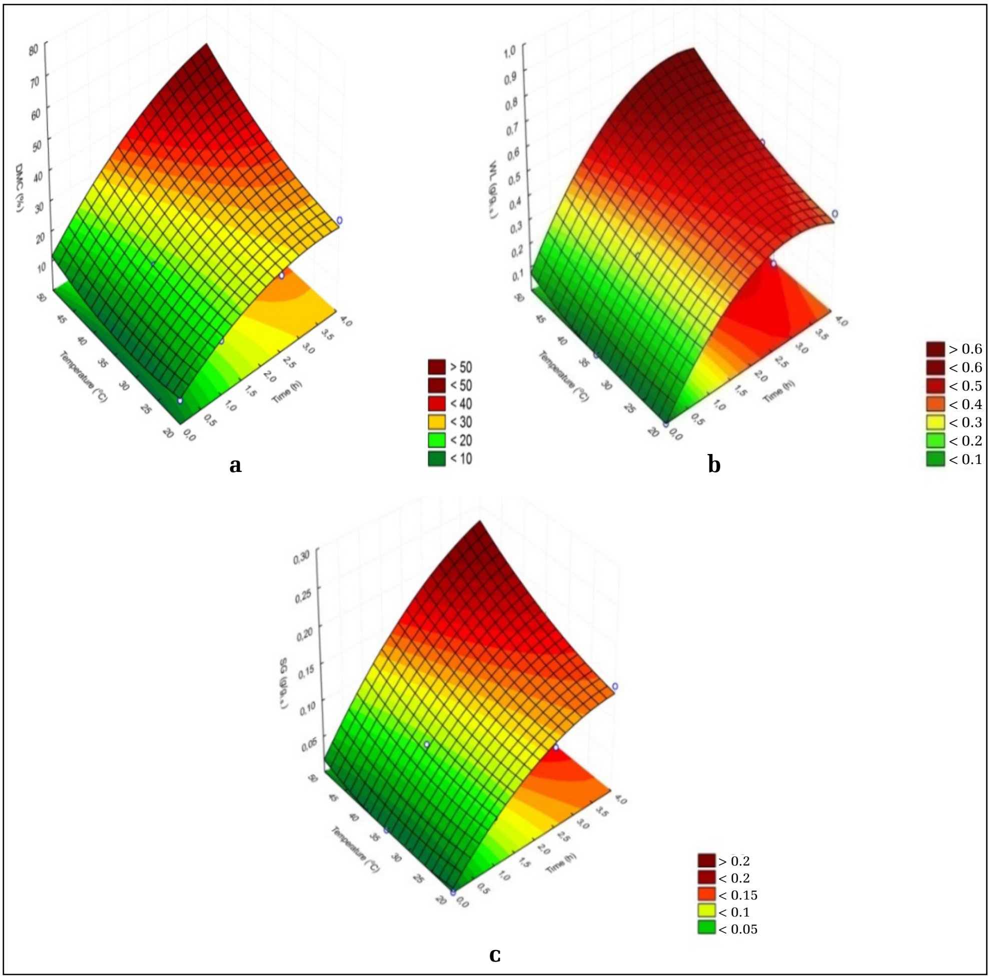

Figure 1. Change in dry matter content (a), water loss (b), and solid gain (c) depending on temperature and time during the osmotic dehydration of wild garlic

Figures 1 (a, b, c) provide graphical representations of developed mathematical models for all responses of the process of OD of wild garlic in aqueous osmotic solution of sucrose and salt, from which the dependence of changes in the observed response on changes in time and temperature can be monitored. It can be seen from the figures that the increase in the value of both process parameters (time and temperature) led to an increase in the values of DMC, WL, and SG. The graphs can also quantify the greater influence of time change as compared to temperature change on the change of all responses during the OD process of wild garlic, which is in accordance with the results of the ANOVA test (*Table 2*).

The uptake of dissolved substances from osmotic solution and the leaching of food constituents (such as vitamins and minerals) result in the alternation of the composition of the original product, which could adversely influence the nutritional profile and organoleptic attributes (*Akbarian et al.*, 2014). High values of SG in wild garlic are undesirable since the increased uptake of sucrose is associated with the occurrence of diabetes and dental problems and that of salt with hypertension (*Kaur et al.*, 2022). In order to achieve the greatest possible water loss (0.409 ± 0.006 g/g i.s) with the lowest sugar and salt impregnation (0.144 ± 0.006 g/g i.s), the sample dehydrated at 20°C after 4 hours was selected for examining the influence of OD in sucrose–salt solution on the chemical composition of wild garlic.

The amounts of chemical compounds were expressed as the percentage of dry matter of wild garlic. Of the total 7.91% of dry matter in fresh wild garlic, it was determined that proteins participate with 33.97%, cellulose with 32.12%, lipids with 0.11%, and ash with 9.80%. Similar to the presented results, *Piatkowska et al.* (2015) reported that the content of dry matter in wild garlic was 7.9%, of which 17.72% protein content, 32.9% fibre, 7% fat, and 11.26% minerals as ash. In the case of osmotically dehydrated samples, the total percentage of dry matter is reduced by 14.4% of the dry matter (SG) that was adopted from the osmotic solution. Based on the results in *Table 3*, it is obvious that the content of protein, cellulose, and fat in comparison with the dry matter in the fresh samples remained unchanged. On the other hand, as a consequence of sugar uptake through mass transfer during OD, osmodehydrated wild garlic contained 10% of total sugars. Also, the ash content in the osmotically dehydrated sample increased by about 4% as a result of salt (sodium) intake.

In the investigation by *Vučić et al.* (2018), the content of the examined macroelements in the samples of wild garlic from different locations was in the range of 317–335 mg/kg for magnesium (Mg), 31–33 mg/kg for sodium (Na), and 1,532–1,559 mg/kg for calcium (Ca), and the content of the examined microelement was in the range of 13.9–15.6 mg/kg for iron (Fe), 2.3–2.6 for zinc (Zn), and 1.6–1.9 mg/kg for copper (Cu). This is in accordance with the presented results, but the determined amounts of Mg, Zn, and Cu were higher in this study. When the composition of mineral components was expressed in the same way as of the chemical components (percentages reduced by 14.4% given by the dry matter content of fresh wild garlic), the amounts of Mg, Zn, Cu, and Fe were reduced by about 30% and that of Ca by about 40%. This finding is in accordance with that reported by *Cvetković et al.* (2019), who find the content decreased of Fe, Cu, Mg, and Ca at 30–60% after the osmotic dehydration of cabbage in sucrose–salt solution. The reduction of mineral components is a result of the diffusion of part of the cellular juices from the wild garlic tissue into the osmotic solution. On the other hand, an increase in sodium and total sugars reflects the penetration of the solutes from the osmotic solution during the OD.

Table 3. Changes in chemical and mineral components in wild garlic after osmotic dehydration in sucrose–salt solution

Chemical component	Fresh wild garlic	Osmodehydrated wild garlic (% d. m.)	Mineral matter	Fresh wild garlic	Osmodehydrated wild garlic (mg/kg)
Protein	33.97 ± 1.27[a]	29.01 ± 0.74[b]	Zn	5.31 ± 0.37[a]	2.81 ± 0.11[b]
Starch	0.00 ± 0.00[a]	0.00 ± 0.00[a]	Cu	1.63 ± 0.10[a]	0.86 ± 0.08[b]
Total sugars	0.00 ± 0.00[a]	10.08 ± 0.26[b]	Mg	911.77 ± 89.19[a]	501.77 ± 70.44[b]
Cellulose	32.12 ± 1.81[a]	27.03 ± 0.41[b]	Na	32.87 ± 2.01[a]	4356 ± 1.20[b]
Fat	0.11 ± 0.01[a]	0.09 ± 0.01[b]	Ca	1432.01 ± 93.91[a]	770.46 ± 94.58[b]
Ash	9.80 ± 0.51[a]	12.95 ± 1.83[b]	Fe	18.71 ± 2.01[a]	10.03 ± 0.99[a]

Note: [a–b] – the different letters in the superscript in the same column of the table indicate statistically significant difference between values at the level of significance of $p < 0.05$ (based on post-hoc Tukey HSD test).

Having an extended shelf life, the obtained partially dehydrated wild garlic leaves could be used for direct consumption or in combination with various food products. Osmotically dehydrated wild garlic in a solution of sucrose and salt is suitable as an ingredient in food formulations such as yogurt, sauces, bakery products, and snacks for the nutritional and sensory improvement of these products. *Šobot et al.* (2019) proposed a new product, a biscuit with osmotically dehydrated wild garlic in molasses. Unlike osmodehydrated wild garlic, adding fresh leaves can adversely affect the texture of food products. Also, the increased content of dry matter in osmodehydrated wild garlic allows for greater quantities to be added.

4. Conclusions

Based on the above-discussed, it can be concluded that:

– An increase in processing temperature and duration of osmotic dehydration resulted in improved mass transfer during the process. Accordingly, the maximum values obtained for dry matter content, water loss, and solid gain were achieved at the end of 4 hours of the process at the highest temperature (50°C), indicating a good level of dehydration.

– Statistical analysis confirmed that the values of investigated kinetic parameters were influenced by both process parameters (time and temperature), with time being the most influential parameter.

– The content of mineral components in osmodehydrated wild garlic decreased, whereas sodium and sucrose content increased as a consequence of mass transfer during the osmotic dehydration in the sucrose–salt solution.

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