

THE IMPACT OF CONVECTION DRYING TEMPERATURE ON CONTENT OF SELECTED PHYTOCHEMICALS IN DRIED WILD GARLIC LEAVES (*ALLIUM URSINUM* L.)

– Research paper –

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Abstract: Wild garlic is a well-known and highly valued herbal plant. It is used fresh, unprocessed, and in the form of dried plants. The paper presents the results of the impact of the applied convection drying conditions at 30, 40 and 60°C on the parameters of the drying process as well as selected properties and composition of dried garlic obtained from wild garlic. It has been unequivocally shown that the increase in drying temperature significantly affects the content of thermally labile compounds, especially low-molecular weight antioxidants, including polyphenols and vitamin C. The analysis of the results clearly indicates that the dried plants obtained at 30°C contain relatively the highest amounts of antioxidant components and show the most volatile compounds profile similar to fresh wild garlic. However, the analysis of the parameters of drying process revealed that the temperature of the process significantly affects the time needed for the completion of drying process. The recommended of the most favourable process conditions should be based on an extended analysis and optimization of the entire process, taking into account not only the quality parameters of the dried material, but other parameters such as energy inputs and labour costs.

Keywords: *Allium ursinum* L., drying; antioxidant activity; polyphenols

INTRODUCTION

Wild garlic (*Allium ursinum* L.) is a perennial plant found in Europe and Asia (Stearn 1980). The forests of the Sudetes and the Carpathians are the most frequent places of wild garlic occurrence in Poland (Oszmianski 2007). It grows in nutrient-rich, shady soils and in the vicinity of deciduous trees. It can be used all parts of wild garlic. Bulbs and leaves have been used in natural medicine for centuries. Additionally, this plant is very tasty and can be eaten raw or cooked (Janeczko et al. 2008). Due to the unique aroma of both fresh and dried leaves, garlic is used in many local cuisines in Europe (Radulovic 2015).

The most characteristic components of the wild gar-

lic plants are compounds including sulphur, which are responsible for the characteristic garlic smell and, additionally, are primarily responsible for the potential health benefits (Tomšik et al. 2015). Wild garlic has a destructive effect on bacteria, viruses and fungi (Iwanowa et al. 2009; Wu et al. 2009). In addition, it activates enzymes responsible for detoxification, and has been used, among others, in the treatment of various diseases, e.g. atherosclerosis and thrombosis. Its rich chemical composition includes vitamins A, C and E, selenium, iron (Stajner et al. 2008; Rouis-Soussi et al. 2014). Additionally, the plant contains flavonols - derivatives of kaempferol, which prevent heart disease and cancer (Schmitt et al. 2005; Oszmianski et al. 2013).

Throughout the year as a result of the variability of climatic conditions, the availability of fresh raw material of wild garlic is limited. This is due to the

Received: 05.02.2023

Accepted in revised form: 28.02.2023

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fact that it occurs in nature only in spring (Sobolewska et al. 2006). From the point of view of the quality parameters of the plant, it is important to use an appropriate storage method that allows the properties of the plant to be preserved throughout the year, until the next harvest (Tomšik et al. 2015). Drying is one of the proven methods that extends the life of the product, and thus allows the plant to be used in pharmacy or in the food industry for a longer period of time than in the case of fresh garlic. The selection of an appropriate temperature during the drying process of wild garlic allows to limit changes in the composition of heat-sensitive substances (Ben Haj Said et al. 2015).

The methods of food processing for preservation by means of food drying are divided into natural and forced. Natural drying methods use the energy of the sun to remove moisture from the food. The disadvantage of these methods is the dependence on weather conditions and the poor efficiency of the process (Toshniwal and Karale 2019). Forced drying methods have advantages over natural methods. According to Okoro and Madueme (2004), forced methods remove large amounts of moisture more efficiently, and also allow the control of applied factors such as drying air temperature and flow as well as drying time.

One of the verified methods of forced drying is convection drying. This method is based on the application of heat, which medium is a drying medium, responsible for removing moisture from the dried matrix. This method is characterized by relatively long drying times, but requires not very complicated apparatus (Brennan and Grandison 2006).

Another method used to dry food products is microwave drying, which supplies energy through the use of electromagnetic radiation of the appropriate frequency. Microwave drying requires less time and temperature to remove moisture from food (Kahyaoglu et al. 2012).

Freeze-drying is an alternative method that competes with drying and consists in dehydrating substances by freezing them (-50°C), and then sublimating the resulting ice crystals. The last stage of the process is the final drying, taking place at a temperature of $40\text{--}50^{\circ}\text{C}$, which allows to obtain a finished product with an in range of 1-2% humidity (Abbasi and Azari 2009).

Vacuum drying (VD) is characterized by better kinetic parameters and, at the same time, a lower temperature of the preservation process, which allows to maintain the favorable parameters of the dried material. These properties improve the quality, keep the shape of the dried material close to the natural one and maintain favorable mechanical properties (Amellal and Benamara, 2008).

However, due to the simplicity and availability of installations intended for the drying process, convection drying is still the most frequently used method of preservation, therefore the aim of the work was to determine the effect of convection drying temperature on the kinetics of the process and the content of selected phytochemicals in the obtained dried material. It was hypothesized that the use of a drying agent with a lower temperature results in obtaining a higher quality of dried wild garlic leaves.

MATERIALS AND METHODS

Plant materials

The research material consisted of wild garlic leaves from the field cultivation. Aboveground biomass was harvested in April 2022. The dry matter content in the leaves was $11.32\% \pm 0.45$.

Methods of drying

The test samples, which consisted of wild garlic leaves weighing $500\text{ g} \pm 1.0\text{ g}$, were preserved by convection drying (CD). Drying was carried out in three technological repetitions. Three drying medium (air) temperatures were used: 30°C , 40°C and 60°C . The drying agent flow was 0.2 m s^{-1} . Drying was carried out until the final moisture content was obtained, with the obtained dried material parameters equal to $\pm 5.5\%$ moisture content. Subsequently, the obtained dried garlic was subjected to laboratory analyses to determine the

antioxidant potential, the content of vitamin C, as well as the content of polyphenols. The composition of the volatile fraction of the obtained dried materials was also determined.

Antioxidant activity, total polyphenolic and vitamin C content

Antioxidants from garlic leaves were extracted by shaking 0.5 g of dried and milled sample with 5 ml of 75 % methanol solution (v/v) and centrifuging at $10,000 \times g$ for 30 min. The obtained supernatant was subjected to DPPH antiradical activity and total phenolic compounds analysis. Antioxidant activity was determined using 2,2-diphenyl-1-picrylhydrazyl radicals (DPPH, Sigma-Aldrich, Darmstadt, Germany) according to the method presented by Oszmiański et al. (2017). The results were expressed as trolox equivalent of 1 g of the

product based on a calibration curve prepared with trolox standard (Sigma-Aldrich, Darmstadt, Germany) in the concentration range of 0.005-0.25 mg/ml. Total phenolic compounds level was assessed with Folin-Ciocalteu (Chempur, Piekary Śląskie, Poland) reagent as described by Matłok et al. (2020 c). The results were calculated based on a calibration curve prepared with gallic acid standards (Sigma-Aldrich, Darmstadt, Germany) in the concentration range of 0.004-0.125 mg/ml. For ascorbic acid level analysis, 1 g of milled sample was shaking with 20 ml of 2 % oxalic acid for 30 min at 150 rpm. The suspension was filtered and the clear extract was titrated with 0.25 mg/ml of 2,6-dichlorophenolindophenol (DCPIP, Sigma-Aldrich, Darmstadt, Germany) solution according to the protocol presented by Piechowiak et al. (2019). Each analysis was performed in three technical replicates.

Head Space – Solid Phase Microextraction HS-SPME

Dried wild garlic leaves obtained from the fresh plant were subjected to the solid phase microextraction analysis (HS-SPME) using 100 μ m polydimethylsiloxane (PDMS) fiber from Supelco Ltd. (Bellefonte, PA, USA) according to the method described by Matłok et al. (2020 a). The analytes were isolated using a Varian 450 GC gas chromatograph (Varian Instrument Group, Sunnyvale, CA, Canada) according to the methodology described by Matłok et al. (2020 b).

Statistical analysis

One-way analysis of variance (ANOVA, $\alpha = 0.05$) was performed with STATISTICA 13.1 (TIBCO Software Inc., Hillview Avenue, Palo Alto, CA, USA) to demonstrate statistically significant differences.

RESULTS AND DISCUSSION

The drying parameters

The drying kinetics of the plant material mainly depends on the applied temperature and the flow of the drying agent. As can be seen in figure 1, utilized 60°C, it is possible to achieve the assumed moisture content after 480 minutes. Decreasing the temperature significantly extends this time. For example, at 40°C drying time is 240 minutes longer but at 30°C it is 480 minutes more than in 60°C. However, decreasing the process temperature significantly reduces the energy demand and has a positive effect on the retention of thermally labile substances.

Antioxidant activities

Wild garlic as an addition to prepared dishes is used in two forms: fresh and dried. Dried plants are very functional due to their availability to consumers outside of their growing season. Research by Matłok et al. (2020) and Stępień et al. (2019) reveals data about the possibility of maintaining high quality parameters of the dried material and about the impact of the applied drying process on its quality parameters.

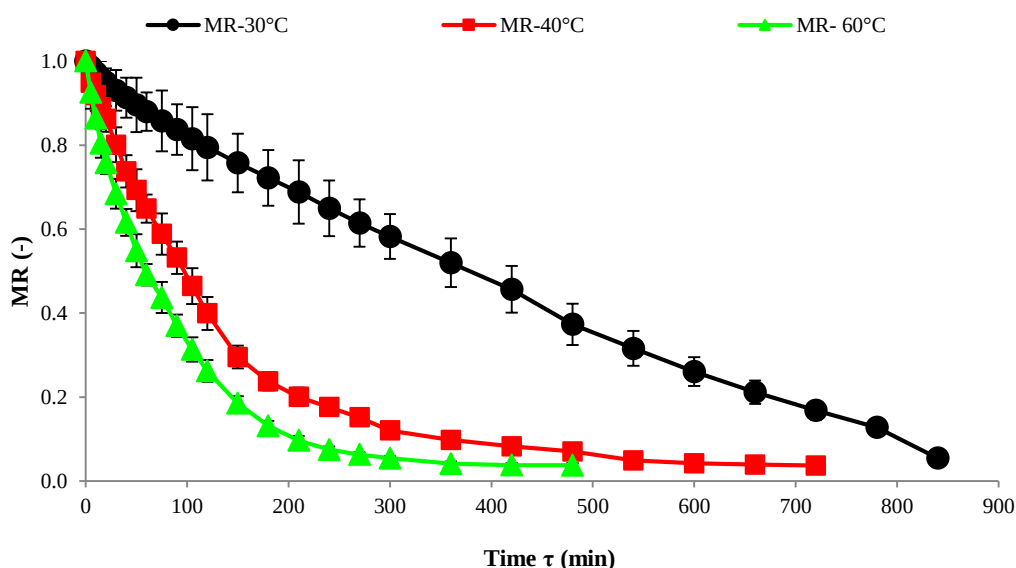


Figure 1. Water content decrease in *Allium ursinum* L. leaves during convective drying (CD) at 30, 40 and 60°C. Note: MR = moisture ratio, n=3; $\pm$ SD

It was noticed that at higher drying temperatures, the thermally labile compounds, mainly low-molecular-weight antioxidants, undergo decomposition (Figures 2 and 3). Similar observations were made in the research on wild garlic and it was shown that the factor that affects the quality of the raw material is the drying temperature. Convection drying at the temperature of 30°C, as expected, allowed to maintain a high level of the antioxidant potential of dried plants. It should be noted that at this temperature, the processed material required much longer drying time. It is important because the oxygen-containing atmosphere of the drying medium used during the drying process may also affect the degradation of antioxidant substances (Laguerre et al. 2007). The presented research has shown that the increased temperature is a much more important parameter of the drying process compared to the exposure time to the drying agent rich in atmospheric oxygen. This

was confirmed by the significantly lower values of the antioxidant potential determined as a result of the research, observed for the material dried at 40 °C and 60 °C. A strong correlation was also observed between the applied temperature and the antioxidant potential of the dried material, which is confirmed by the determined Pearson correlation coefficient at the level of -0.897

As indicated by numerous scientific reports, this negative effect of the drying process on the final chemical composition of the plant raw material can be eliminated by using more sophisticated drying methods, for example, drying using the combined method allows obtaining the highest quality dry basil at relatively high temperatures and in a short time (Calín-Sánchez et al. 2012) and thyme (Calín-Sánchez et al. 2013). However, the results obtained in the presented research are valuable because convection drying can be performed without the use of complicated equipment.

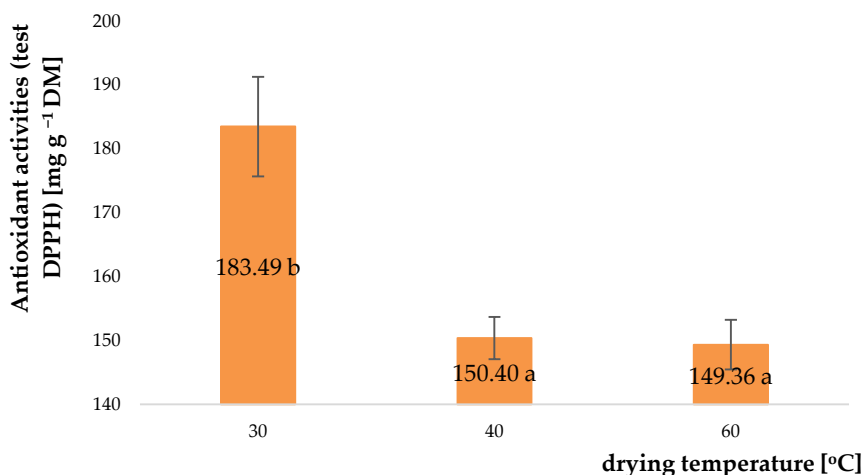


Figure 2. Antioxidant activities: DPPH test in dry leaves *Allium ursinum* L. Note: n=3; ± SD

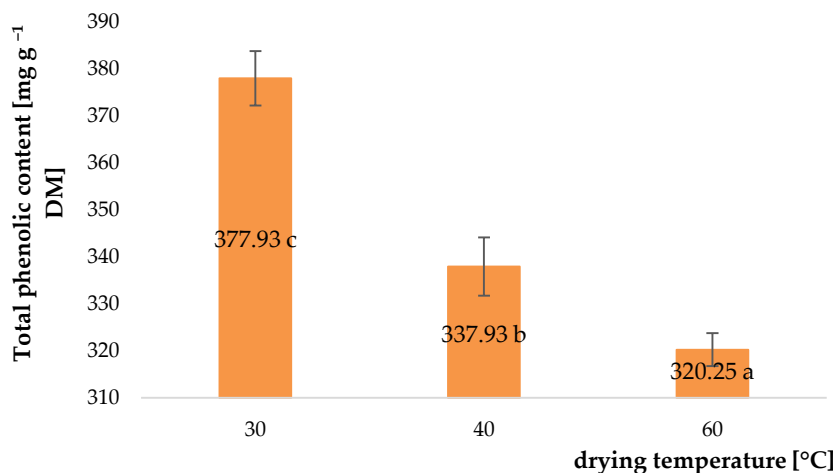


Figure 3. Total phenolic content in dry leaves *Allium Ursinum* L. Note: n=3; ± SD

Total phenolic content

Polyphenols are secondary metabolites characterized by a high potential of biological activity. Provided as food ingredients, they play an important role especially in the nutrition of people with special nutritional needs. A high supply of polyphenols and other antioxidant compounds in food is necessary during convalescence after illnesses and is often necessary during the treatment and prevention of especially cancerous diseases (Arsova-Sarafinavska and Dimovski 2013).

Polyphenols are substances that provide a significant impact on the total antioxidant potential of the raw material in which they occur. They are also substances susceptible to oxidation and thermally labile. As shown by the research carried out, wild garlic subjected to the drying process in the temperature variants used in the research, was characterized by a significantly different content of total polyphenols. The raw material dried at 30 °C was characterized by the highest content, while the increase in temperature by 10 °C caused a 10% decrease in the total polyphenol content. Another increase (10°C) in temperature also resulted in a decrease in the content of this group of compounds by 15%. The analysis of the results showed that the total polyphenol content strongly depends on the temperature. The determined Pearson correlation coefficient ($R = -0.9760$) between the temperature and the content of polyphenols proves a strong relationship between these two parameters and confirms that, taking into account the content of this factor, the drying process should preferably be carried out at the lowest possible temperature.

A similar dependency in the studies was presented for the dried leaves of Rock Rose by Stępień et al. (2019), and for dried blueberry fruit Zielińska et al. (2016). The process of drying plants or fruits at high temperature causes irreversible losses in the quantitative composition of bioactive compounds (Arabhosseini et al. 2007, Zielińska et al. 2016).

Vitamin C content

Vitamin C is one of the most valuable ingredients that should be supplied to the body with food. Most of the food processing processes used lead to the decomposition of this labile compound (Vikram et al. 2005).

As in the case of other examined quality parameters, the vitamin C content in wild garlic leaves decreased as a result of an increase in the temperature of the drying process. An increase in the temperature of the drying process by 10°C causes a decrease in the vitamin C content by 13%, while an increase by 20°C increases this decrease to 28% (Figure 4). As in the case of the antioxidant potential and total polyphenols, the decrease in the content of this factor strongly depends on the drying temperature, which is confirmed by the correlation coefficient determining the level of linear dependence between random variables ($R = -0.9994$).

Similar relationships were observed during drying of other plants rich in vitamin C, including rosehips (Erenturk et al. 2005), tomatoes (Goula and Adamopoulos 2006) and other raw materials (Al-Zubaidy and Khalil 2007, Lin et al. (2011).

Chemical composition of HS-SPME

The edible parts of wild garlic are leaves with a characteristic smell and taste similar to garlic bulbs. In garlic tubers, sulfur compounds resulting from the breakdown of allin by allinase are dominant. A number of volatile substances are detected in garlic, mainly sulfur compounds (Süfer and Bozok 2019). Sulfur compounds of a similar structure also dominate in the profile of volatile compounds that have been isolated from wild garlic. The main sulfur compound identified is diallyl disulfide which is also found in *Allium Sativum*.

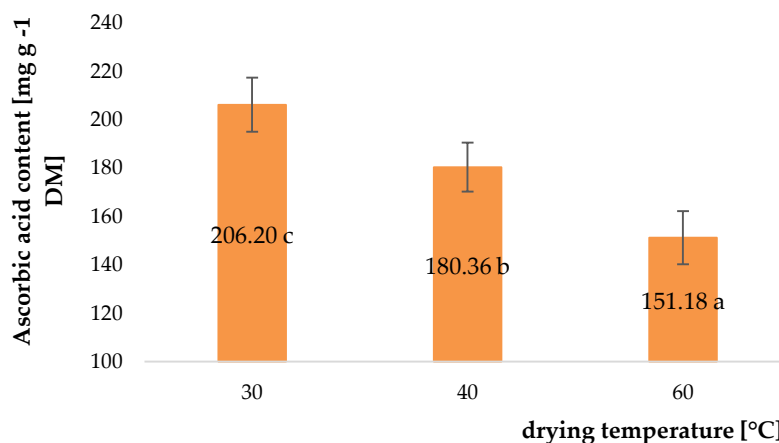


Figure 4. Vitamin C content in dry leaves *Allium Ursinum* L. Note: n=3; $\pm$ SD

The second main ingredient is 2-Ethyl-6-methylpyrazine, which is also found in arabica coffee and field mint (www 1). It is a substance with a potato-like odor and a nutty flavor. These two substances shape the characteristic organoleptic properties of raw and dried wild garlic (www 2.). The table shows the VOC profile of fresh wild garlic and the resulting dried plant. As expected, substances with the lowest molecular weights, and thus the lowest values of the temperature of phase transformations, were desorbed during the drying process. Compared to the trans-2-hexenal compound present in fresh wild garlic, this compound was no longer identified in the dried product (Table 1). Low molecular weight compounds such as: 1- (methylsulfonyl) prop-1-ene; 1,3-Dithiane; dimethyl trisulfide are present in relatively large amounts in fresh material, while they are reduced as a result of the drying process and are present only at the level of traces in dried plant. It should be noted, that the profile of dry bear garlic plants aroma compounds is characterized by an increased content of substances with higher

molecular weights. This observation is related to a significant change in the flavor profile between fresh wild garlic and the obtained dry bear garlic plants, but the enrichment of the dried material with compounds with higher molecular weights makes the smell of such material much more durable, but with a lower intensity. Dried herbal materials are most often used as spices to enrich food products with a high water content, e.g. soups. These dishes are eaten heated, where the aromatic substances contained in the spices are released in a severe manner by transport with steam (www 3.). As a result of the analysis of changes in the volatile substances profile with the increase of the drying temperature, it can be observed that with the increase of the drying temperature, the profile of the aromatic compounds becomes enriched with substances of lower volatility. This is particularly evident in the example of butanoic acid, 2-methoxy-3-methyl-, methyl ester, which has also been identified in strawberry fruit (Eun-Ryong et al. 2000).

Table 1. Content of volatile fraction within fresh and dried wild garlic leaves

RT (min)	Peak share in the chromatogram (%)				Systematic substance name	Molecular weight	No CAS
	fresh	30°C	40°C	60°C			
5.21	15.79	-	-	-	trans-2-Hexenal	98	6728-26-3
6.97	2.35	trace	trace	trace	1-(methylsulfonyl)prop-1-en	120	5905-47-5
7.59	3.29	trace	trace	trace	1,3-Dithiane	120	505-23-7
8.23	trace	trace	trace	trace	dimethyl trisulfide	126	3658-80-8
10.56	28.48	30.32	28.62	28.42	2-ethyl-6-methyl pyrazine	136	13925-03-6
10.70	3.98	3.62	4.62	4.26	3,5-dimethyl-2-ethylpyrazine	136	13925-07-0
10.83	4.50	4.39	4.39	trace	2-allyl-(1,3)dithiane	146	63382-29-6
10.95	28.01	31.99	34.67	25.16	diallyldisulfide	146	2179-57-9
11.51	1.65	1.90	1.90	trace	undefined sulfur compound		
11.63	2.01	8.25	7.22	26.53	butanoic acid, 2-methoxy-3-methyl-, methyl ester	146	109989-32-4
12.25	5.43	9.32	9.32	6.34	2-mercapto-3,4-dimethyl-2,3- dihydrothiophene	146	137363-86-1
12.47	0.87	1.54	1.54	trace	3-vinyl-4H-1,2-dithiin	144	62488-53-3
12.64	trace	2.55	2.55	trace	di-tert-amyl disulfide	206	34965-30-5
14.19	trace	2.30	3.30	9.26	diallyl trisulfide	178	2050-87-5
16.69	trace	2.82	1.82	trace	4-(2,6,6-trimethyl-1-cyclohexenyl)-3- buten-2-one	192	14901-07-6

CONCLUSION

The paper presents the results concerning the impact of the applied convection drying conditions on the properties and composition of dried material obtained from wild garlic. It was clearly shown that the increase in the drying temperature of the

processed raw material significantly influences the content of thermally labile compounds, especially low-molecular antioxidants, including polyphenols and vitamin C. The analysis of the results clearly indicates that the dried material obtained at 30°C

contain relatively the highest amounts of antioxidant components and exhibit a volatile compound profile closest to unprocessed wild garlic. At the same time, the analysis of the parameters of the drying process indicate that the temperature of the process significantly affects the total necessary time of the drying process. An

unequivocal recommendation of the most favorable conditions for the drying process should be based in particular on the extended analysis and optimization of the entire process, taking into account not only the quality parameters of the dried fruit, but other parameters such as energy inputs and labor costs.

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