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## **THE EFFECT OF LIOPHILIZATION ON THE AROMA COMPONENTS IN THE FRUIT BODIES OF EDIBLE MUSHROOMS**

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### **ABSTRACT**

The qualitative and quantitative evaluation of the content of volatile aroma compounds in the fruit bodies of two species of edible mushrooms: oyster (*Pleurotus ostreatus*) and nameko (*Pholiota nameko*) was performed by gas chromatography coupled with mass spectrophotometry. Both examined species of fungi differed from each other in the amounts of the main volatile substances. The most important components determining the aroma of these mushrooms were octen-1,3-ol and octanal. It was also shown that the retention of these substances was effected by the method of sample preparation for the analyses and the method of liophilization. The sublimatic drying enabled the retention of 50% of aroma compounds in oyster fruit bodies and 39.2% in nameko fruitbodies. Freezing of fruit bodies after harvesting enabled the retention of 63.5% of aroma compounds in oyster fruit bodies and 75% in nameko fruitbodies.

**Key words:** mushrooms, processing, aroma compounds

## INTRODUCTION

The important features that discriminate edible fungi from other food products are taste and flavour giving a total feeling of savouriness. This trait is identified with the presence of volatile compounds, mostly alcohols, aldehydes, ketones, esters and non-volatile substances such as free aminoacids and carbohydrates. The composition and level of these compounds change according to the species, state of maturity, and even growth conditions of mushrooms. The characteristic mushroom aroma is formed by about 150 substances. It was shown by Kompany and Rene [5, 6] that among volatile substances mostly contributing to the aroma are those that have 8 atoms of carbon and double bond at the first carbon atom and a functional group at the third carbon atom. In this group of compounds there are: 1-octen-ol, 1-octen-3-on, trans-2-octen-1-ol, 3-octanol, 1-octanol. They make about 90% of volatile compounds fraction. Moreover, in shiitake fruit bodies the cyclic sulfur containing compounds were found. The most important volatile compound found in a high amount in mushrooms but simultaneously having low point of perceptibility is 1-octen-3-ol. It makes 82.5% of volatile compounds fraction isolated from fruit bodies of *Agaricus bisporus*, 82.5% from *Boletus edulis*, 49.1% from *Tricholoma flavovirens* and 67.49% from *Xerocomus badius* [3, 5, 6, 10, 11].

In sensory evaluation, aroma compounds are the substantial element of food. These volatile, organic compounds mostly have low stability that is why they are easily evaporated and degraded during technological processes. The evaporation of the aroma compounds is especially intensive during the condensation and drying processes. The presence of water promotes the oxidation processes and the increase of temperature causes the undesired chemical reactions of the aroma components. It leads to the changes of the bouquet of aroma and its malformity, and results in the decrease of the quality of the product.

The freeze-drying allows for a high degree of the retention of volatile compounds, which can be explained by the microregion theory (Flink and Karel) or by selective diffusion (Rulkens and Thijssen). The freezing of product causes a high concentration of carbohydrates and volatile compounds in the intercrystalline space. The decrease of moisture leads to the association of the carbohydrates by the hydrogen bounds and the newly formed structure is permeable for the water and volatile compounds. When the water content in local microregions decreases, the volatile substances evaporate more and more slowly up to the limit of water content, when volatilization stops, though the water still evaporates [1, 2, 4, 7, 8, 9, 12].

The aim of this work was to establish the usefulness of sublimatic drying for the preservation of oyster and nameko mushrooms that can be further used as the components of vegetable-mushroom concentrates. The main testing criterion of the methods of dehydration and the evaluation of the product was the level of aromatic compounds in dry material.

## MATERIALS AND METHODS

The species of fungi used in the experiments were obtained from the collection of Department of Fruit and Vegetable Processing.

The oyster mushroom (*Pleurotus ostreatus*) belongs to Basidiomycetes class. It is a saprophyte growing on dead trunks, logs, and snags of wide leaf trees. It can also live on trees as a parasite, attacking an injured tissue. It has high possibilities of adaptation, therefore it can

live in almost every climate. Because of its fast growth, common occurrence and its taste value it has been gaining popularity and became the third mushroom in the world commercial production.

Nameko (*Pholiota nameko*) belongs to Basidiomycetes class and Strophariaceae family. It is the second most popular cultivated Asiatic mushroom, besides shiitake. It produces convex, round shaped pilei of orange-brown colour, 5-10 cm in diameter. Nameko grows on the trunks, logs and sawdust of coniferous trees such as fir (*Abies*), spruce (*Picea*), and pine (*Pinus*). It can be cultivated on the broad leaf trees sawdust medium from beech (*Fagus*) and oak (*Quercus*).

In the experiments the fruit bodies of oyster (B7) and nameko (B9) cultivated on the medium containing beech sawdust (80%) and wheat bran (20%) were used. The fruit bodies of both mushrooms were collected at the same phase of maturity. Before the use, the fruit bodies were stored in a cooler. Before drying, the fruit bodies were cut into 2-3 mm thick slices.

Two experimental combinations were compared: sublimatically dried fresh fruit bodies and frozen at  $-18^{\circ}\text{C}$ . Freeze-drying was performed in the freeze-drier LGA-05 type, at the pressure of 13.6 Pa and the temperature of the heating plate  $60^{\circ}\text{C}$ . The time of drying for oyster mushroom was 51 hours and for nameko 47.5 hours.

### **Extraction procedure**

To isolate volatile substances, homogenised sample (2 g) was placed in an extraction flask equipped with the reflux condenser and treated with dichloromethane (10 ml). Extraction was carried out in the water bath at  $60^{\circ}\text{C}$  for 2 hours. Cooled extract was filtered and analysed by means of GC-MS.

### **GC-MS analysis:**

Gas chromatograph coupled with mass spectrometer equipped with an ion trap (ITS-40, Finnigan MAT, USA) was used. The samples were injected into a DB-5 (I & W Scientific, USA) fused silica capillary column (30 m  $\times$  0.25 mm i.d.; 0.25  $\mu\text{m}$  film thickness) by splitless injection. The temperature of sample injector was  $300^{\circ}\text{C}$ . The temperature program applied: isotherm  $35^{\circ}\text{C}$  (2 min), temperature slope  $4^{\circ}\text{C}/\text{min}$  to  $300^{\circ}\text{C}$  and final isotherm  $300^{\circ}\text{C}$  (10 min). Helium as carrier gas with the flow of 1.5 ml/min was used. MS conditions were as follows: EI (70 eV), monifold temperature  $220^{\circ}\text{C}$ , mass range 35-400 a.u.m.

### **Analyte identification**

To identify compounds in the extracts, their MS spectra were compared with the spectra gathered in the NIST library.

Identification was confirmed by retention indexes. Retention index defines the analyte peak location on the chromatogram with regard to the n-alkanes:

$$I_x = (t_{R_x} - t_{R_n}) \div (t_{R_{n+1}} - t_{R_n}) \times 100 + 100n;$$

Where:  $t_{R_x}$  – the analyte retention time,  $t_{R_n}$  – the retention time of the n-alkane with n-carbon atoms,  $t_{R_{n+1}}$  – the retention time of alkane with n+1 carbon atoms and  $t_{R_n} \leq t_{R_x} \leq t_{R_{n+1}}$ .

## RESULTS

The results of qualitative and quantitative analyses of the volatile compounds in the oyster mushroom and nameko fruit bodies showed interesting relations between the analysed species of mushrooms and between the methods of mushroom preservation.

The analyses made by gas-chromatography coupled with mass spectrometry (GC-MS) method showed that the most important aroma compound of mushrooms is octen-1,3-ol ([table 1](#)). It makes more than 63% of volatile fraction of frozen oyster fruit bodies and 75% of frozen fruit bodies of nameko ([table 2](#)). The volatile fraction of freeze-dried fruit bodies contained 53% and 80% of this compound, respectively, ([table 3](#)). It gives the characteristic mushroom aroma and is characterized by the low level of sensory perceptibility (0.01 ppm).

In the liophilized samples of oyster mushroom the aroma compounds were preserved more than in 28% and for nameko samples more than 30% ([table 4](#)).

**Table 1. Aroma compounds content (peak areas) in *Pleurotus ostreatus* and *Pholiota nameko* fruit bodies**

No	Scan	Compound name	Pleurotus fruit bodies		Pholiota fruit bodies	
			Frozen	Liophilized	Frozen	Liophilized
1.	728	Octen-1,3-ol	6411	1491	1400	480
2	739	3-octanon	539	277	27	10
3.	748	Penthyl-fural	238	20	198	17
4.	768	3-octanal	2649	990	140	15
5.	782	Octanal	114	47	102	40
6.	794	4,5-dimethyl-4-hexen-3-on	151	11	0	0

**Table 2. Percentage of volatile compounds in the analyzed fractions for the frozen fruit bodies (%)**

No	Scan	Compound name	Pleurotus	Pholiota
1.	728	Octen-1,3-ol	63.47	75.00
2.	739	3-octanon	5.32	1.45
3.	748	Penthyl-fural	2.38	10.60
4.	768	3-octanal	26.24	7.50
5.	782	Octanal	1.14	5.45
6.	794	4,5-dimethyl-4-hexen-3-on	1.45	0.0

**Table 3. Percentage of volatile compounds in the analyzed fractions for the freeze-dried fruit bodies (%)**

No	Scan	Compound name	Pleurotus	Pholiota
1.	728	Octen-1,3-ol	52.57	80.00
2.	739	3-octanon	9.77	1.67
3.	748	Penthyl-fural	0.70	2.85

4.	768	3-octanal	34.91	8.38
5.	782	Octanal	1.66	6.70
6.	794	4,5-dimethyl-4-hexen-3-on	0.39	0.0

**Table 4. Retention of volatile compounds in the liophilized fruit bodies of *Pleurotus ostreatus* and *Pholiota nameko* (%)**

No	Scan	Compound name	Pleurotus	Pholiota
1.	728	Octen-1,3-ol	23.25	34.30
2.	739	3-octanon	51.39	37.03
3.	748	Penthyl-fural	8.40	8.60
4.	768	3-octanal	37.30	35.70
5.	782	Octanal	41.20	39.20
6.	794	4,5-dimethyl-4-hexen-3-on	7.28	0.0

The experiments showed the differences in the quality of aroma compounds between two analysed species of edible mushrooms. A very strong influence of biological features of the species growing on the same medium could be noticed. The fruit bodies of nameko maintained the volatile substances on a much higher level than oyster fruit bodies ([table 4](#)).

Drying processes cause strong volatilization of aroma. At high temperatures aroma compounds are the subject of undesirable chemical transformations. It causes changes of the aroma bouquet and its malformations and as a result it reduces the quality of the product. Therefore, the liophilization carried out in low temperatures is a good method that enables the retention of volatile components.

## DISCUSSION

The chromatographic analysis of the most important volatile compounds content and their retention level confirmed the effects obtained by other authors [5, 6, 10]. Liophilization turned out to be the technological process that allows for high retention of these compounds important for the quality of dried mushrooms.

The volatile compounds content depended on the mushrooms species. The second important observation was a noticeable influence of the method of processing. The frozen fruit bodies maintained the volatile substances on a higher level than the samples dried sublimatically. The reduction of biochemical metabolism by freezing had a distinct influence on the retention of high amounts of the substances influencing the quality of the product. From the practical point of view it can be noticed that although freezing of mushroom fruit bodies allows for maintaining higher aroma compounds content, freeze-dried mushrooms are more suitable for further processing.

Comparing these two species of mushrooms, another interesting conclusion can be drawn. Nameko, which contains approximately five times fewer volatile compounds than oyster mushroom, is characterized by higher retention after processing ([table 4](#)).

## CONCLUSIONS

1. The time of sublimatical drying was different for the examined mushrooms, and it was 51 hours for oyster mushroom and 47 hours for nameko.
2. The quantitative analysis of the volatile compounds in the fruit bodies showed that independently of the species, the main component deciding about their aroma is octen-1,3-ol.
3. The retention of octen-1,3-ol in liophilized products was substantial and made about 28% for oyster mushroom and 30% for nameko.
4. The amount of the components remaining in the liophilized product was dependent on the method of pretreatment of the raw material.
5. The liophilized fruit bodies of oyster and nameko are good materials for vegetable concentrates, pastes and powders.
6. There is a need for further studies on the retention of volatile substances in mushrooms.

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