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CHARACTERIZATION OF AROMATIC VOLATILE COMPOUNDS OF EIGHT WILD MUSHROOMS BY HEADSPACE GC-MSD

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Mushrooms are consumed in many countries because of their taste and aroma. The aroma is the most important organoleptic property in the consumption of mushrooms [1]. As a result of various investigations in the literature, approximately 175 different volatile compounds, including octane, octene, benzaldehyde, terpene, and sulfur compounds and their derivatives, have been identified in mushroom aroma [2, 3]. Researchers mostly focused on aliphatic compounds having eight carbons. Among them, 1-octen-3-ol is considered to be the major contributor to mushroom aroma among the aliphatic compounds [4–6].

In this study, we aimed to analyze the volatile compounds of eight wild mushrooms, i.e., *Agaricus bisporus*, *Chroogomphus rutilus*, *Clathrus ruber*, *Ganoderma adspersum*, *Lactarius deliciosus*, *Melanoleuca cognata*, *Pleurotus ostreatus*, and *Rhizopogon luteolus* collected from Anatolia, Turkey by the Headspace GC-MSD technique. To date, the volatile compounds of *A. bisporus* [7–10], *P. ostreatus* [11–13], and *C. rutilus* have been studied [14].

A. bisporus, *C. rutilus*, *C. ruber*, *L. deliciosus*, *M. cognata*, *P. ostreatus*, and *R. luteolus* were collected from Mugla, Turkey in December 2012, and their fungarium numbers were given as AT-1824, AT-1829, AT-1828, AT-1840, AT-1841, AT-1844, and AT-1831, respectively. These mushroom species are edible. *G. adspersum* was collected from Aydin, Turkey in December 2012, and its fungarium number was given as AT-1403. This mushroom is not edible but consumed as tea. All mushroom species were identified by Dr. Aziz Turkoglu. A voucher specimen has been deposited in the Fungarium of the Department of Biology, Mugla Sitki Kocman University.

Five grams of the collected mushrooms were sliced into small parts, placed into the 20 mL headspace vial, and mixed with a small amount of anhydrous Na_2SO_4. The vials were immediately sealed with an airtight silicone/polytetrafluoroethylene (PTFE) septum and then placed in the auto sampler tray for headspace sampling.

Headspace GC-MSD analyses were performed using an Agilent 7890 GC system coupled to an Agilent 5975C Triple Axis Detector MSD and equipped with an Agilent 7697A auto sampler. Samples were subjected to static headspace analysis for 20 min at 60°C. Headspace transfer line temperature was 120°C. Headspace gas was injected in 20:1 split ratio. Separation was performed on an HP-5MS column (30 m × 0.25 mm, 0.25 μm film thickness). The carrier gas was at a constant flow rate of 1.5 mL/min. The column oven temperature was initially set at 50°C for 1 min and then increased to 120°C at 8°C min^{-1} and held for 1 min at 120°C; then it was increased at 25°C min^{-1} to 300°C and held for 10 min. The temperature of the injector and ion source was 250°C. The mass detector was operated in electron impact mode with 70 eV ionization energy and scanning range of 10–600 amu. Retention indices were calculated by using the retention times of *n*-alkanes injected under the same chromatographic conditions. The volatile compounds were identified by comparison of their linear retention indices (relative to C_6–C_{26} alkanes on the HP-5MS column) and by mass spectral database search (NIST 2005 library).

The relative percentages (%) of volatile compounds in the studied mushroom species are given in Table 1. A total of 31 compounds was identified, including eight alcohols, seven aldehydes, two ketones, eleven terpenes, and three other compounds, using headspace GC-MSD.

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TABLE 1. Relative Percentage (%) of Aroma Compounds of Studied Mushroom Species

Compound	RI*	1	2	3	4	5	6	7	8
Pentanal	730	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.	1.49	N.d.
3-Pentanol	755	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.	4.42	N.d.
Hexanal	803	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.	4.53	N.d.
1-Heptanol	881	N.d.	N.d.	N.d.	N.d.	N.d.	2.94	N.d.	N.d.
1,3-Diethylbenzene	890	0.10	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.
2-Cyclohexen-1-one	917	N.d.	N.d.	N.d.	N.d.	N.d.	2.32	N.d.	N.d.
α-Pinene	941	N.d.	15.33	N.d.	N.d.	N.d.	5.60	N.d.	N.d.
Camphene	957	N.d.	N.d.	N.d.	N.d.	N.d.	4.50	N.d.	N.d.
Benzaldehyde	962	22.66	N.d.	N.d.	N.d.	0.41	11.11	2.20	2.88
1-Octanol	983	N.d.	N.d.	N.d.	N.d.	N.d.	2.52	0.45	N.d.
1-Octen-3-ol	985	9.10	28.58	82.62	79.30	86.46	3.32	17.22	67.92
β-Pinene	988	N.d.	4.95	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.
3-Octanone	991	53.92	35.44	7.06	5.21	5.81	1.91	42.24	1.76
3-Octanol	992	5.62	8.85	N.d.	N.d.	3.24	N.d.	13.36	N.d.
2-Pentyl furan	998	N.d.	N.d.	N.d.	2.08	N.d.	12.57	N.d.	N.d.
Octanal	1005	N.d.	N.d.	1.31	0.86	N.d.	6.93	0.58	N.d.
(+)-2-Carene	1023	N.d.	N.d.	N.d.	N.d.	N.d.	0.73	N.d.	N.d.
o-Cymene	1027	N.d.	3.37	N.d.	N.d.	N.d.	19.92	0.30	N.d.
Limonene	1033	N.d.	3.46	N.d.	N.d.	N.d.	2.22	N.d.	N.d.
1,8-Cineole	1039	N.d.	N.d.	1.73	N.d.	N.d.	4.32	N.d.	N.d.
2-Octen-1-ol	1043	0.36	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.	26.04
Benzene acetaldehyde	1049	5.68	N.d.	N.d.	10.07	1.26	3.71	0.44	0.71
2*E*-Octenal	1061	0.45	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.
γ-Terpinene	1075	N.d.	N.d.	N.d.	N.d.	N.d.	3.72	N.d.	N.d.
m-Cresol	1091	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.	10.97	N.d.
Nonanal	1108	0.46	N.d.	5.08	2.44	2.00	11.61	0.70	0.62
Phenyl ethanol	1120	1.07	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.
(–)-β-Fenchol	1142	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.	0.25	N.d.
Camphor	1151	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.	0.30	N.d.
Borneol	1174	N.d.	N.d.	N.d.	N.d.	N.d.	N.d.	0.33	N.d.
2*Z*-Dodecenol	1465	N.d.	N.d.	1.10	N.d.	N.d.	N.d.	N.d.	N.d.
Total, %		99.42	99.98	98.90	99.96	99.18	99.95	99.78	99.93
Alcohols		16.15	37.43	83.72	79.30	89.70	8.78	35.45	93.96
Aldehydes		29.25	0.00	6.39	13.37	3.67	33.36	9.94	4.21
Ketones		53.92	35.44	7.06	5.21	5.81	4.23	42.24	1.76
Terpenes		0.00	27.11	1.73	0.00	0.00	41.01	1.18	0.00
Others		0.10	0.00	0.00	2.08	0.00	12.57	10.97	0.00

1 – *A. bisporus*; 2 – *C. rutilus*; 3 – *C. rubber*; 4 – *G. adspersum*; 5 – *L. deliciosus*; 6 – *M. cognata*; 7 – *P. ostreatus*; 8 – *R. luteolus.* N.d.: not detected. *Retention index was based on HP-5MS column.

1-Octen-3-ol and 3-octanone were found to be major compounds in the mushroom species. These two compounds were detected in all the studied mushrooms. 1-Octen-3-ol was the major aroma compound of *C. ruber* (82.62%), *G. adspersum* (79.30%), *L. deliciosus* (86.46%), and *R. luteolus* (67.92%), while 3-octanone was the major aroma compound of *A. bisporus* (53.92%), *C. rutilus* (35.44%), and *P. ostreatus* (42.24%). In addition, *o*-cymene was the major compound in *M. cognata* (19.92%).

As shown in Table 1, the highest percentage of total alcohol was found in *R. luteolus* (93.96%), followed by *L. deliciosus* (89.70%) and *C. ruber* (83.72%). All identified aldehyde compounds were detected in the highest amount in *M. cognata* (33.36%) and *A. bisporus* (29.25%), while no aldehyde compounds were found in *C. rutilus*. Total ketone amount of *A. bisporus* (53.92%) was the highest, followed by *P. ostreatus* (42.24%) and *C. rutilus* (35.44%). The total amounts of terpenes were found to be lower than those of aliphatic eight-carbon alcohols, ketones, and aldehydes.

In conclusion, volatile compounds of *C. ruber*, *G. adspersum*, *L. deliciosus*, *M. cognata,* and *R. luteolus* mushroom species were investigated for the first time in this study.

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