

Differentiation of Aroma Characteristics of Pine-Mushrooms (*Tricholoma matsutake* Sing.) of Different Grades Using Gas Chromatography–Olfactometry and Sensory Analysis

IN HEE CHO,[†] SOH MIN LEE,[†] SE YOUNG KIM,[†] HYUNG-KYOON CHOI,[‡]
 KWANG-OK KIM,[†] AND YOUNG-SUK KIM^{*,†}

Department of Food Science and Technology, Ewha Womans University, Seoul 120-750, South Korea, and College of Pharmacy, Chung-Ang University, Seoul 156-756, South Korea

Two independent approaches, gas chromatography–olfactometry and sensory analysis, were used to evaluate and compare the aroma characteristics of pine-mushrooms (*Tricholoma matsutake* Sing.) of four different grades. The aroma-active compounds responsible for the sensory attributes of pine-mushrooms were investigated based on the correlation between instrumental and sensory analyses through partial least-square regression. Piny, meaty, and floral attributes were strongly correlated with each other and were the most important descriptors for defining the pine-mushrooms of the highest grade, and they decreased as the grade decreased. Among 23 aroma-active compounds, (*E*)-2-decenal, α -terpineol, phenylethyl alcohol, and 2-methylbutanoic acid ethyl ester contributed most to these attributes. On the other hand, the major aroma characteristics of the pine-mushrooms of the lowest grade were wet soil-like, alcohol, metallic, moldy, and fermented, and they decreased as the grade increased. These aroma characteristics were strongly associated with 1-octen-3-one, 1-octen-3-ol, 3-octanol, 3-octanone, (*E*)-2-octen-1-ol, and methional.

KEYWORDS: Pine-mushroom (*Tricholoma matsutake* Sing.); instrument-sensory correlations; GC-O; quantitative descriptive analysis (QDA); partial least-square regression (PLSR)

INTRODUCTION

The aroma of a foodstuff is crucially important to its perceived quality (1). In general, aroma perception is determined by the properties and amounts of volatile components and their availability to the sensory system as a function of time (2). It has been shown in many foods that only a small number of volatiles activate the odorant receptors in the human nasal cavity (3–5), thereby contributing to the aroma perception during mastication. Such aroma-active compounds can be screened from the bulk of nonactive volatiles by combining the human olfactometry system with analytical techniques (6). Gas chromatography–olfactometry (GC-O) with aroma extract dilution analysis (AEDA) is the most frequently used method to estimate the sensory contribution of single aroma compounds. In AEDA, the assessors evaluate whether or not an aroma can be perceived and describe its aroma property. The result is expressed as the flavor dilution (FD) factor that corresponds to the maximum dilution at which the aroma is detected (7). In addition, information on the sensory attributes of a foodstuff can be provided by establishing their descriptive profiles with sensory descriptive analysis, such as a quantitative descriptive analysis (QDA) technique, which discriminates and describes both

qualitative and quantitative aspects of a foodstuff. The qualitative component comprises the descriptive terms, called “attributes”, which define the sensory profile of the samples, and the quantitative component measures the degree or intensity of each perceived attribute (8, 9).

Therefore, combining two approaches, GC-O and sensory evaluation, represents a powerful and comprehensive way to investigate the aroma characteristics of a foodstuff. Multivariate analysis can be used to extract interpretable and statistically reliable information from data sets obtained by separate approaches (10–15). In particular, partial least-square regression (PLSR) is one of the multivariate regression analysis techniques that can be used to understand the relationship between two data sets by predicting one data set (*Y*) from the other set (*X*). It not only provides solutions for both data sets but also attempts to find the best solution of one data set to explain the variation of the other data set (16).

Pine-mushroom (*Tricholoma matsutake* Sing.) is the most valuable mushroom species worldwide, and it exhibits a characteristic and delicate flavor. Pine-mushrooms can be classified according to their appearance (17, 18), which is affected mostly by their ripening stages and cultivating conditions. The quality characteristics of pine-mushrooms, such as aroma, taste, texture, and color, vary with their grades. In particular, pine-mushrooms of higher grades have distinctive aroma characteristics as compared to those of lower grades. We recently investigated

* To whom correspondence should be addressed. Tel: +82 2 3277 3091. Fax: +82 2 3277 4213. E-mail: yskim10@ewha.ac.kr.

[†] Ewha Womans University.

[‡] Chung-Ang University.

Table 1. Definitions and References of the Descriptive Attributes for Pine-Mushrooms

attribute	definition	reference
sweet	fundamental taste elicited by sugars	sucrose (1.5% in water)
salty	fundamental taste elicited by salts	sodium chloride (0.4% in water)
sour	fundamental taste elicited by acids	citric acid (0.05% in water)
bitter	fundamental taste elicited by caffeine	caffeine (0.07% in water)
umami	fundamental meaty taste elicited by monosodium glutamate (MSG)	monosodium glutamate (0.15% in water)
piny	aroma associated with pine needle	pine needle tea (one tea bag infused in 100 mL of boiling water for 2 min)
floral	aroma associated with chrysanthemum	chrysanthemum tea (one tea bag infused in 200 mL of boiling water for 2 min)
alcohol	aroma associated with rum	white rum (0.125% in water)
meaty	aroma associated with cooked meat	boiled beef
moldy	aroma associated with typical mushroom	1-octen-3-one (1000 ppm in dichloromethane)
wet soil-like	aroma associated with damp soil	damp soil
fishy	aroma associated with fermented anchovy	fermented anchovy sauce (0.025% in water)
fermented	aroma associated with Sikhye (a traditional Korean fermented rice beverage)	Sikhye drink (50% in water)
metallic	aroma associated with metals	stainless steel spoon
astringent	mouth feeling associated with tannins	tannic acid (0.1% in water)

how the volatile compositions of pine-mushrooms vary with their grades by applying multivariate statistical methods to gas chromatography–mass spectrometry (GC-MS) data sets (18). We also revealed that 23 aroma-active compounds were key odorants of pine-mushrooms of the highest grade (19).

The aims of the present study were to (i) characterize and quantify the potent odorants in pine-mushrooms of different grades, (ii) describe and measure the intensity of each sensory attribute in pine-mushrooms according to their grades, (iii) ascertain how the results of instrumental and sensory analyses of pine-mushrooms vary with their grades, and (iv) identify the odorants responsible for aroma attributes of pine-mushrooms by correlating the GC-O and sensory data sets using PLSR.

MATERIALS AND METHODS

Chemicals. Dichloromethane ($\geq 99.9\%$ purity) was obtained from Fisher Scientific (Seoul, Korea). Sodium sulfate and *n*-alkane standards (C_8 – C_{22}) were purchased from Sigma-Aldrich (St. Louis, MO). Stock solutions of 23 authentic standard compounds were prepared in dichloromethane. Standards 1–9, 11–15, 17–21, and 23 were obtained from Sigma-Aldrich, while standards 10, 16, and 22 were purchased from Wako Pure Chemical Industries (Osaka, Japan).

Sample Materials. Pine-mushrooms of four grades cultivated in Inje-eup, Gangwon-do, South Korea, in 2004 were used. Raw pine-mushrooms were wrapped in low-density polyethylene film and stored at $-70\text{ }^\circ\text{C}$ until used.

Extraction of Volatiles. Frozen mushrooms were thawed at $4\text{ }^\circ\text{C}$ for 3 h and then sliced using a cutter (Shinomura, Sanjō, Niigata, Japan). The sliced mushrooms were transferred into a stainless steel container, frozen in liquid nitrogen, and then ground in a blender (Hanil Electric, Seoul, Korea). The ground mushrooms (100 g) were extracted with 200 mL of dichloromethane that was redistilled before use. The ground sample suspended in dichloromethane was magnetically stirred at 400 rpm for 30 min and then filtered through Whatman no. 41 filter paper (Maidstone, United Kingdom) under vacuum. Volatile components were then separated from the nonvolatiles using high vacuum sublimation (HVS) (18). The extract was dehydrated over anhydrous sodium sulfate, evaporated on a Vigreux column (50 cm length \times 3 cm i.d.) in a water bath at $45\text{ }^\circ\text{C}$, and then concentrated under a slow stream of nitrogen gas to obtain a final volume of 0.1 (for GC-MS and GC-O) or 1.0 mL (for fractionation by column chromatography).

Fractionation by Column Chromatography. To identify odorants not detected by GC-MS, the HVS extracts (1.0 mL) were subjected to silica gel column chromatography. The mushroom extracts were loaded onto a cooled column (45 cm length \times 20 mm i.d.) filled with silica gel (35–70 mesh, 40 Å, Sigma-Aldrich). The concentrated volatile

extracts were separated into six fractions using a pentane/diethyl ether gradient (F1 extracts = 50/0 mL, F2 extracts = 40/10 mL, F3 extracts = 30/20 mL, F4 extracts = 20/30 mL, F5 extracts = 10/40 mL, and F6 extracts = 0/50 mL, respectively). Each fraction was further concentrated under a slow stream of nitrogen gas until it could be analyzed for the identification of unknowns in GC-MS analysis.

GC-MS. GC-MS analysis was performed using an HP 6890N gas chromatography-5973N mass selective detector (GC-MSD) (Hewlett-Packard, Palo Alto, CA) equipped with a DB-wax column (60 m length \times 0.25 mm i.d. \times 0.25 mm film thickness, J&W Scientific, Folsom, CA) and an HP 5890 series II GC/5972 MSD equipped with a DB-5ms column (30 m length \times 0.25 mm i.d. \times 0.25 mm film thickness). The carrier gas was helium at a constant flow rate of 0.8 mL/min. One microliter of mushroom extract was injected into the column using the splitless injection mode. The oven temperature was initially held at $40\text{ }^\circ\text{C}$ for 1 min, then raised to $200\text{ }^\circ\text{C}$ at a rate of $4\text{ }^\circ\text{C}/\text{min}$, and finally held at $200\text{ }^\circ\text{C}$ for 10 min. The temperatures of injector and detector were 200 and $250\text{ }^\circ\text{C}$, respectively. The mass detector was operated in electron impact mode with an ionization energy of 70 eV, a scanning range of 33–550 amu, and a scan rate of 1.4 scans/s.

GC-O. GC-O was conducted on a Varian CP-3800 GC (Varian, Walnut Creek, CA) equipped with a flame ionization detector (FID) and a sniffing port (ODO II, SGE, Ringwood, Australia) using a DB-wax column and a DB-5ms column. Effluent collected from the end of GC column was split equally between the FID and the sniffing port. The HVS extract was diluted stepwise with dichloromethane (1 + 1 by volume). An aliquot (1 μL) was injected into the capillary column. The oven temperature program was the same as that used for GC-MS. The temperatures of injector and detector were 200 and $250\text{ }^\circ\text{C}$, respectively. FD factors of the volatile components were determined by AEDA; the FD factor corresponded to the maximum dilution at which each component could be detected (20). Two experienced sniffers, each with >30 h training on GC-O, participated in AEDA. Then, the maximum value of them was provided as the FD factor of that compound.

Identification of Aroma-Active Compounds. For positive identifications, mass spectra, linear retention indices (RIs), and aroma properties of unknowns were compared with those of authentic standards. Tentative identifications were based on matching RIs and aroma properties of unknowns with those in the literature (21) or comparing the RIs and aroma properties of unknowns to those of authentic standards. Aroma properties of all authentic standards were determined by GC-O. The RI of each compound was calculated using *n*-alkanes C_8 – C_{22} as external references (22).

Panel Selection and Training for Sensory Evaluation. Eight subjects (female, 21–25 years of age), who previously participated in more than four descriptive analyses in the Department of Food Science and Technology at Ewha Womans University, were selected as the

Table 2. Aroma-Active Compounds of Pine-Mushrooms of Four Different Grades

no.	RI ^a on		aroma-active compound	aroma property ^b	FD factor ^c								ID ^d
	DB-wax	DB-5ms			DB-wax				DB-5ms				
					first	second	third	fourth	first	second	third	fourth	
1	1031	<800	2-methyl-3-buten-2-ol	herbaceous	1	1	1	0	2	2	1	0	MS/RI/odor
2	1073	895	2-methylbutanoic acid ethyl ester	floral and sweet	32	4	4	16	64	8	8	16	RI/odor
3	1097	807	hexanal	cut grasslike	2	2	1	2	2	2	1	2	MS/RI/odor
4	1195	1032	limonene	lemonlike	1	1	2	1	2	1	2	1	MS/RI/odor
5	1240	971	3-octanone	mushroomlike/buttery	2	1	4	4	4	4	4	8	MS/RI/odor
6	1259	<800	3-hydroxy-2-butanone	buttery	1	4	2	4	1	4	1	4	MS/RI/odor
7	1317	978	1-octen-3-one	mushroomlike	256	256	512	1024	256	256	1024	4096	MS/RI/odor
8	1360	865	1-hexanol	green	1	0	1	1	1	0	2	2	(F5) MS/RI/odor
9	1390	1104	nonanal	fatty and soapy	1	2	1	2	2	4	4	4	(F3) MS/RI/odor
10	1394	1007	3-octanol	mushroomlike/buttery	8	16	32	32	32	32	128	256	MS/RI/odor
11	1430	1052	(E)-2-octenal	sweet	1	2	1	2	2	4	2	4	MS/RI/odor
12	1435	1173	octanoic acid ethyl ester	green and sweet	1	0	1	0	1	1	2	1	MS/RI/odor
13	1451	1002	1-octen-3-ol	mushroomlike	8	4	16	32	8	4	8	32	MS/RI/odor
14	1480	914	methional	boiled potato-like	8	32	32	128	32	64	256	512	RI/odor
15	1484	1029	2-ethyl-1-hexanol	roselike	1	0	1	0	2	1	2	0	(F3–6) MS/RI/odor
16	1540	1105	linalool	citruslike	8	4	16	8	32	32	64	16	MS/RI/odor
17	1546	1074	1-octanol	mushroomlike/chemical	2	1	2	2	1	1	2	4	MS/RI/odor
18	1622	1071	(E)-2-octen-1-ol	mushroomlike	2	1	4	4	4	4	8	8	MS/RI/odor
19	1630	1260	(E)-2-decenal	orangeliike and fatty	2	1	1	0	2	1	1	1	(F3) MS/RI/odor
20	1648	1043	phenylacetaldehyde	floral and honey-like	1	1	0	2	2	2	0	4	MS/RI/odor
21	1652	1064	2-acetylthiazole	roasted	0	2	0	4	0	4	1	4	(F4) MS/RI/odor
22	1675	1198	α-terpineol	piny	2	0	0	0	2	0	0	0	(F3–5) MS/RI/odor
23	1810	1060	phenylethyl alcohol	floral and sweet	2	1	1	1	2	2	1	1	MS/RI/odor

^a RIs were determined using *n*-paraffins C₈–C₂₂ as external references. ^b Aroma properties perceived at the sniffing port. ^c FD factors by two panelists. An FD factor <1 means that the respective compound was not detected during sniffing of the undiluted extract. ^d Aroma-active compounds were identified on the basis of the following criteria: MS/RI/odor, mass spectrum, RI, and aroma properties perceived at the sniffing port were consistent with those of authentic standards; (Fn) MS/RI/odor, mass spectrum, RI, and aroma properties perceived at the sniffing port after fractionations by silica gel column chromatography using a pentane:diethyl ether gradient (*n* = 1, 50 mL of pentane; *n* = 2, 40 mL of pentane:10 mL of diethyl ether; *n* = 3, 30 mL of pentane:20 mL of diethyl ether; *n* = 4, 20 mL of pentane:30 mL of diethyl ether; *n* = 5, 10 mL of pentane:40 mL of diethyl ether; and *n* = 6, 50 mL of diethyl ether) were consistent with those of authentic standards.

panelists. The panelists were trained with pine-mushrooms of four grades used in this study. During the training session, they developed 15 descriptors, defined them, and selected reference samples corresponding to each attribute. Definitions and references of the generated descriptors are given in **Table 1**. The descriptive analysis procedure was developed by consensus among all of the panelists, and the training continued until panelists showed consistency.

Sample Preparation and Evaluation Procedure. Pine-mushrooms of similar size were selected for each experiment. The outer layers were peeled off before dicing each mushroom into about 0.5 cm × 0.5 cm cubes. Each sample comprised 10 g of mushrooms in a porcelain container (8 cm i.d. × 4.5 cm height) and was coded with a three-digit random number. A descriptive analysis of pine-mushrooms was performed using a QDA method (23) with partial adoption of spectrum descriptive analysis method (24). All four samples were presented simultaneously with random order and examined in individual sensory booths at room temperature (22 ± 2 °C) under the dim red light. The pine-mushrooms were evaluated according to a protocol established during training periods. The aroma attributes of the mushrooms were evaluated by opening the lid of the container half way and sniffing three times. The taste and mouth-feel attributes of the mushrooms were assessed by tasting approximately 2 g of each sample, which was applied to the tongue using an odorless melamine spoon (Sokoware,

Sakhon, Thailand). The intensity for each attribute was rated on a 15-point category scale, ranging from “weak” (score of 1) to “strong” (score of 15). Panelists rinsed their mouths five times with water filtered using a ceramic filter system (Faiey Industrial Ceramics, London, United Kingdom). Each session took approximately 20 min, and each descriptive analysis was conducted in four replications.

Statistical Analysis. Analysis of variance (ANOVA) was performed using the general linear model procedure to determine significant differences in the sensory attributes of pine-mushrooms according to their grades. Duncan’s multiple range test was conducted when the samples exhibited significance between samples, with the level of significance set at *P* < 0.05. Both ANOVA and Duncan’s multiple range test were performed with SPSS (version 10.1, Chicago, IL). PLSR was applied to clarify the correlation between instrumental and sensory data sets. The task of PLSR was performed with SIMCA-P software (Umetrics, Umeå, Sweden).

RESULTS AND DISCUSSION

Aroma-Active Compounds in Pine-Mushrooms of Different Grades. **Table 2** lists aroma-active compounds identified in pine-mushrooms according to the grades, their aroma properties, the RI values, and the FD factors for each of the

Table 3. Mean Values of Sensory Attributes in Pine-Mushrooms of Four Different Grades

	first	second	third	fourth
sweet	7.72 a ^a	5.28 b	5.22 b	3.69 c
salty	4.09 b	6.66 a	6.13 a	6.56 a
sour	3.59 c	5.75 b	6.13 b	6.84 a
bitter	3.03 d	4.69 c	5.66 b	6.87 a
umami	5.00 d	7.84 a	6.56 b	5.84 c
piny	8.31 a	6.88 b	4.87 c	3.69 d
floral	8.38 a	6.66 b	5.13 c	4.34 d
alcohol	3.97 d	5.19 c	5.97 b	7.41 a
meaty	8.06 a	6.78 b	5.16 c	3.88 d
moldy	2.91 d	4.25 c	5.47 b	7.66 a
wet soil-like	2.81 d	4.34 c	5.34 b	7.50 a
fishy	3.03 c	6.38 ab	5.72 b	6.47 a
fermented	3.22 d	4.47 c	6.03 b	7.47 a
metallic	2.88 d	4.25 c	5.16 b	6.38 a
astringent	3.13 d	4.34 c	5.28 b	6.94 a

^a There are significant differences ($P < 0.05$) among pine-mushrooms using Duncan's multiple range comparison test between the samples with a different letter in a row.

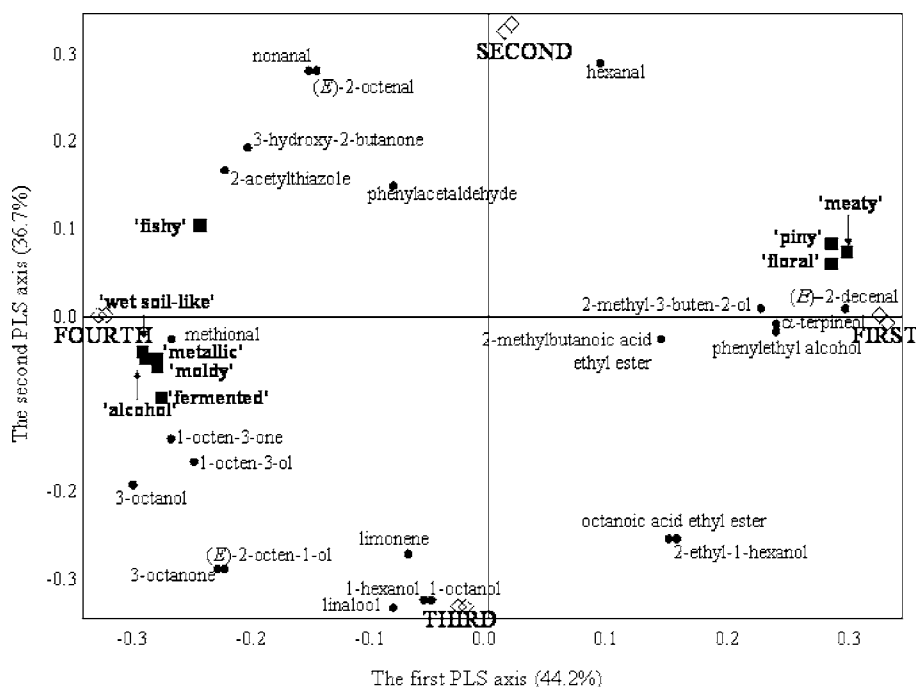
two capillary columns of different polarities, such as the DB-wax and DB-5ms columns. A total of 23 aroma-active compounds were identified in pine-mushrooms of four grades. Among them, 15 compounds (nos. **1**, **3–7**, **10–13**, **16–18**, **20**, and **23**) were positively identified by comparing their mass spectra, RI values, and aroma properties with those of authentic standards. Numbers **2** and **14** were detected by GC-O and confirmed by comparing their RIs and aroma properties with those of standards using GC-O. Enrichment by column chromatography was necessary for the identification of nos. **8**, **9**, **15**, **19**, **21**, and **22**.

1-Octen-3-one (**7**), described as mushroomlike, was the most potent aroma-active compound in the pine-mushrooms of all grades (i.e., with the highest FD factor), followed by methional (**14**, boiled potato-like), 3-octanol (**10**, mushroomlike/buttery), 1-octen-3-ol (**13**, mushroomlike), linalool (**16**, citruslike), and 2-methylbutanoic acid ethyl ester (**2**, floral and sweet). It is notable that 2-methylbutanoic acid ethyl ester was found with

the second highest FD factor in pine-mushrooms of first grade. 2-Methylbutanoic acid ethyl ester has a pleasant odor note with a detection threshold of 0.06–0.24 ng/L in air (25) and is a well-known aroma-active compound of apples (26) and other fruits (27, 28).

In a previous study, the major aroma-active compounds of pine-mushrooms of first grade were related to 1-octen-3-one, 3-octanol, 1-octen-3-ol, (*E*)-2-octen-1-ol, 3-octanone, and 1-octanol and were described as mushroomlike, mushroomlike/buttery, and mushroomlike/chemical (19). In addition, 2-methylbutanoic acid ethyl ester, linalool, methional, hexanal, phenylethyl alcohol, (*E*)-2-decenal, α -terpineol, 2-methyl-3-buten-2-ol, limonene, nonanal, (*E*)-2-octenal, 2-ethyl-1-hexanol, phenylacetaldehyde, 1-hexanol, and octanoic acid ethyl ester contributed to the characteristic odor of pine-mushrooms (19). In this study, the FD factors of 1-octen-3-one, 3-octanol, and methional increased on both columns with the grades of the pine-mushrooms, with the reverse being true for the FD factors of 2-methyl-3-buten-2-ol (**1**, herbaceous), (*E*)-2-decenal (**19**, orangeliike), α -terpineol (**22**, piny), and phenylethyl alcohol (**23**, floral and honeylike).

Sensory Evaluation of Pine-Mushrooms of Different Grades. A total of 15 sensory attributes on pine-mushrooms were described by eight trained panelists. They were sweet, salty, sour, bitter, umami, piny, floral, alcohol, meaty, moldy, wet soil-like, fishy, fermented, metallic, and astringent. The mean intensity values of sensory attributes of pine-mushrooms of the four grades are listed in **Table 3**. The intensities of all of the attributes differed significantly ($P < 0.05$) among pine-mushrooms of four different grades. The intensities of sweet, piny, floral, and meaty attributes were strongest in pine-mushrooms of the highest grade, and they decreased as the grade decreased. In contrast, the intensities of sour, bitter, alcohol, moldy, wet soil-like, fermented, metallic, and astringent attributes were highest in pine-mushrooms of the lowest grade and decreased as the grade increased. It is interesting to note that the intensity of umami taste was strongest and weakest in pine-mushrooms of second and first grades, respectively. The salty taste was weakest in pine-mushrooms of first grade but did not

**Figure 1.** Relationship between aroma-active compounds (●) and sensory attributes (■) of pine-mushrooms in PLSR plot.

differ significantly among mushrooms of other grades. The intensities of fishy odor were highest and lowest in pine-mushrooms of fourth and first grades, respectively. The major sensory attributes of the pine-mushrooms of first grade were sweet, piny, floral, and meaty, whereas they were umami, fishy, and salty were for pine-mushrooms of second grade. The pine-mushrooms of fourth grade possessed relatively stronger intensities of moldy, fermented, wet soil-like, metallic, alcohol, bitter, sour, salty, and fishy attributes, which were weakest in mushrooms of first grade.

Relationship between GC-O and Sensory Data Sets. Multiple attempts have been made to correlate the information obtained from sensory studies and the molecules exhibiting the typical taste of edible mushrooms (29–31). However, there is no study on the odorants responsible for aroma characteristics of mushrooms. Multivariate analysis can be used to extract interpretable and statistically reliable information from data sets obtained by separate approaches. PLSR has been frequently used to draw linkages between instrumental and sensory analyses. In this study, the aroma-active compounds responsible for the sensory attributes of pine-mushrooms were investigated based on the correlation between instrumental and sensory analyses through PLSR.

The first and second PLSR components were calculated by cross-validation for a PLSR analysis model based on 23 aroma-active compounds (X variable) and nine aroma attributes (Y variable) excluding tastes of sweet, salty, sour, bitter, and umami and a mouth feeling of astringent. Two PLS components explained 44.2 and 36.7% of the total variance (Figure 1). The first PLS component generated three clusters, such as first-, second-, and third-, and fourth-grade pine-mushrooms. In addition, pine-mushrooms of second grade were separated from those of third grade in the second PLS component. The first PLS component was mainly defined by the aroma descriptors showing a contrast between piny, meaty, and floral attributes on the positive dimension and wet soil-like, alcohol, metallic, moldy, fermented, and fishy attributes on the negative dimension. As compared with other samples, pine-mushrooms of first grade were evaluated as possessing strong piny, meaty, and floral attributes. In contrast, wet soil-like, alcohol, metallic, moldy, and fermented attributes were also strongly correlated with each other and showed high loadings on the opposite direction from piny, meaty, and floral attributes. These negative attributes were pronounced in pine-mushrooms of fourth grade. In addition, the aroma-active compounds responsible for sensory attributes of pine-mushrooms of different grades were investigated by correlating the instrumental and sensory analyses. (E)-2-Decenal, α -terpineol, phenylethyl alcohol, and 2-methylbutanoic acid ethyl ester, which have fruity, floral, and piny odor notes, were correlated with the sensory attributes of piny, meaty, and floral odors, whereas 1-octen-3-one, 1-octen-3-ol, 3-octanol, 3-octanone, and (E)-2-octen-1-ol, which were described mainly as the typical mushroom odor note, were related to fermented, moldy, wet soil-like, alcohol, and metallic aroma characteristics.

Aroma perception is a complex process, which makes the direct correlation of volatiles with sensory attributes difficult. Multivariate statistical methods can be effective at revealing the relationship between data from instrumental and sensory analyses. In our present study, aroma-active compounds and sensory attributes were analyzed to evaluate and understand the aroma characteristics of pine-mushrooms of different grades. The most noteworthy result is that the flavor quality of pine-mushrooms clearly differs with their grade.

ABBREVIATIONS USED

GC-O, gas chromatography–olfactometry; AEDA, aroma extract dilution analysis; FD, flavor dilution; QDA, quantitative descriptive analysis; PLSR, partial least-square regression analysis; GC-MS, gas chromatography–mass spectrometry; HVS, high vacuum sublimation; RI, retention indices; ANOVA, analysis of variance.

LITERATURE CITED

- (1) Taylor, A. J.; Linforth, R. S. T. Techniques for measuring volatile release in vivo during consumption of food. In *Flavor Release*; Roberts, D. D., Taylor, A. J., Eds.; ACS Symposium Series 763; American Chemical Society: Washington, DC, 2000; pp 8–21.
- (2) Pionnier, E.; Nicklaus, S.; Chabanet, C.; Mioche, L.; Taylor, A. J.; Le Qu  r  , J. L.; Salles, C. Flavor perception of a model cheese: Relationships with oral and physico-chemical parameters. *Food Qual. Pref.* **2004**, *15*, 834–852.
- (3) Grosch, W. Aroma compounds. *Food Science and Technology, Handbook of Food Analysis*; Marcel Dekker: New York, 2004; pp 717–746.
- (4) Schuh, C.; Schieberle, P. Characterization of the key aroma compounds in the beverage prepared from Darjeeling black tea: quantitative differences between tea leaves and infusion. *J. Agric. Food Chem.* **2006**, *54*, 916–924.
- (5) Frauendorfer, F.; Schieberle, P. Identification of the key aroma compounds in cocoa powder based on molecular sensory correlation. *J. Agric. Food Chem.* **2006**, *54*, 5521–5529.
- (6) Fritsch, H. T.; Schieberle, P. Identification based on quantitative measurements and aroma recombination of the character impact odorants in a Bavarian pilsner-type beer. *J. Agric. Food Chem.* **2005**, *53*, 7544–7551.
- (7) Grosch, W. Determination of potent odorants in foods by aroma extract dilution analysis (AEDA) and calculation of odour activity values (OAVs). *Flavour Fragrance J.* **1994**, *9*, 147–158.
- (8) Meilgaard, M.; Civille, G. V.; Carr, B. T. *Sensory Evaluation Techniques*; CRC Press: Boca Raton, FL, 1987.
- (9) McTigue, M. C.; Koehler, H. H.; Silbernagel, M. J. Comparison of four sensory evaluation methods of assessing cooked dry bean flavor. *J. Food Sci.* **1989**, *54*, 1278–1283.
- (10) Engel, E.; Baty, C.; le Corre, D.; Souchon, I.; Martin, N. Flavor-active compounds potentially implicated in cooked cauliflower acceptance. *J. Agric. Food Chem.* **2002**, *50*, 6459–6467.
- (11) le Fur, Y.; Mercurio, V.; Moio, L.; Blanquet, J.; Meunier, J. M. A new approach to examine the relationships between sensory and gas chromatography–olfactometry data using generalized procrustes analysis applied to six French chardonnay wines. *J. Agric. Food Chem.* **2003**, *51*, 443–452.
- (12) Venkateshwarlu, G.; Let, M. B.; Meyer, A. S.; Jacobsen, C. Modeling the sensory impact of defined combinations of volatile lipid oxidation products on fishy and metallic off-flavors. *J. Agric. Food Chem.* **2004**, *52*, 1635–1641.
- (13)   vila, M.; Garde, S.; Fern  ndez-Garc  a, E.; Medina, M.; Nu  ez, M. Effect of high-pressure treatment and a bacteriocin-producing lactic culture on the odor and aroma of Hisp  nico cheese: Correlation of volatile compounds and sensory analysis. *J. Agric. Food Chem.* **2006**, *54*, 382–389.
- (14) Chung, S.-J.; Heymann, H.; Gr  n, I. U. Application of GPA and PLSR in correlating sensory and chemical data set. *Food Qual. Pref.* **2003**, *14*, 485–495.
- (15) Lobo, A. P.; Madrera, R. R.; Alonso, J. J. M. A study of cider distillates using sensory and chromatographic data and chemometric analysis. *J. Food Sci.* **2005**, *70*, 204–207.
- (16) Martens, H.; Martens, M. *Multivariate Analysis of Quality. An Introduction*; Wiley: London, United Kingdom, 2001.
- (17) Korean National Forestry Cooperatives Federation. <http://www.nfcf.or.kr> (accessed 03/03/2006).

- (18) Cho, I. H.; Choi, H.-K.; Kim, Y.-S. Difference in the volatile composition of pine-mushrooms (*Tricholoma matsutake* Sing.) according to their grades. *J. Agric. Food Chem.* **2006**, *54*, 4820–4825.
- (19) Cho, I. H.; Kim, S. Y.; Choi, H.-K.; Kim, Y.-S. Characterization of aroma-active compounds in raw and cooked pine-mushrooms (*Tricholoma matsutake* Sing.). *J. Agric. Food Chem.* **2006**, *54*, 6332–6335.
- (20) Casimir, D. J.; Whitfield, F. B. Flavour impact values: a new concept for assigning numerical values for the potency of individual flavour components. *Processings of the Symposium "Aromastoff in Früchten und Fruchtsäften"*; Bern, Switzerland, 1978; pp 325–347.
- (21) Rychlik, M.; Schieberle, P.; Grosch, W. *Compilation of Odor Thresholds, Odor Qualities and Retention Indices of Key Food Odorants*; Deutsche Forschungsanstalt für Lebensmittelchemie and Institut für Lebensmittelchemie der Technischen Universität München: Garching, Germany.
- (22) van den Dool, H.; Kratz, P. D. A generalisation of the retention index system including linear temperature programmed gas-liquid partition chromatography. *J. Chromatogr.* **1963**, *11*, 463–471.
- (23) Stone, H.; Sidel, J. L. *Sensory Evaluation Practices*, 2nd ed.; Academic Press: London, United Kingdom, 1992.
- (24) Muñoz, A. M.; Civil, G. V. The spectrum descriptive analysis method. In *Manual on Descriptive Analysis Testing for Sensory Evaluation*; Hootman, R. C., Ed.; American Society for Testing and Materials: Philadelphia, PA, 1992; pp 22–34.
- (25) Rychlik, M.; Schieberle, P.; Grosch, W. *Compilation of Odor Thresholds, Odor Qualities and Retention Indices of Key Food Odorants*; Deutsche Forschungsanstalt für Lebensmittelchemie and Institut für Lebensmittelchemie der Technischen Universität München: Garching, Germany.
- (26) Fan, X.; Argenta, L.; Mattheis, J. Impacts of ionizing radiation on volatile production by ripening gala apple fruit. *J. Agric. Food Chem.* **2001**, *49*, 254–262.
- (27) Kilic, A.; Hafizoglu, H.; Kollmannsberger, H.; Nitz, S. Volatile constituents and key odorants in leaves, buds, flowers, and fruits of *Laurus nobilis* L. *J. Agric. Food Chem.* **2004**, *52*, 1601–1606.
- (28) Lopez, R.; Ortin, N.; Perez-Trujillo, J. P.; Cacho, J.; Ferreira, V. Impact odorants of different young white wines from the Canary islands. *J. Agric. Food Chem.* **2003**, *51*, 3419–3425.
- (29) Kasuga, A.; Fujihara, S.; Aoyagi, Y. The relationship between the varieties of dried shiitake mushrooms and chemical composition. The effect of the varieties of dried shiitake mushrooms on their taste. Part 1. *J. Jpn. Soc. Food Sci. Technol.* **1999**, *46*, 692–703.
- (30) Stijve, T.; de A. Amazonas, M. A. L.; Griller, V. Flavour and taste components of *Agaricus blazei* ss. Heinem.-a new gourmet and medicinal mushroom. *Dtsch. Lebensm.-Rundsch.* **2002**, *89*, 448–453.
- (31) Harada, A.; Gisusi, S.; Yoneyama, S.; Aoyama, M. Effects of strains and cultivation medium on the chemical composition of the taste components in fruit-body of *Hypsizygus marmoreus*. *Food Chem.* **2004**, *84*, 265–270.

Received for review September 21, 2006. Revised manuscript received January 3, 2007. Accepted January 7, 2007. This study was supported by a grant (R01-2004-000-10276-0) from the Korea Science and Engineering Foundation.

JF062702Z