Identification of the Most Potent Odorants in Huitlacoche (*Ustilago maydis*) and Austern Pilzen (*Pleurotus* sp.) by Aroma Extract Dilution Analysis and Static Head-Space Samples

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Aroma extract dilution analysis (AEDA) and gas chromatography−olfactometry of static head-space analysis of huitlacoche (*Ustilago maydis*) and austern pilzen (*Pleurotus* sp.) were performed. Comparison of the two mushrooms' volatiles revealed tremendous differences, since 32 odorants were found in huitlacoche and only 19 in austern pilzen. However, only eight out of 32 compounds found by the AEDA screening were also detected by the head-space analysis for huitlacoche. On the other hand, 11 out of 19 were found in both analyses for austern pilzen. Among these, the most potent odorant was sotolon with a flavor dilution factors of 10 and 1000 for huitlacoche and austern pilzen, respectively. The results demonstrated that a large number of compounds (hexanal, octanal, decanal, (*E*,*E*)-deca-2,4-dienal, (*E*)-undec-2-enal, and vanillin) play an important role in the overall aroma of huitlacoche and only two (sotolon and an unknown) in the case of austern pilzen.

Keywords: *Potent odorants; huitlacoche; Ustilago maydis; austern pilzen; Pleurotus sp.; AEDA; GCO-SH*

INTRODUCTION

Several fungal species are edible (Gray, 1970), and they are consumed due to their flavor properties which come mainly from the volatile fraction (Hadar and Dosoretz, 1991). Huitlacoche is the name given by the Aztecs to the young and edible fruiting bodies (gall) of the basidiomicete *Ustilago maydis*, which is the causal agent of common smut of maize (*Zea mays*). Those galls are harvested from fresh ears and are prepared in a variety of ways in Mexican cooking (Kealey and Kosikowski, 1981). In the United States, huitlacoche has been marked as maize mushroom or Mexican truffle and is consumed similarly to other mushrooms (Valverde et al., 1995). Approximately 80 compounds have been reported in the volatile fraction of huitlacoche (Lizárraga-Guerra, 1995); however, the most important odor compounds have not been identified yet.

Pleurotus is an important edible mushroom in the world (Chang, 1990). Its flavor has been attributed mainly to the C8 compounds, mainly 1-octen-3-ol, which stem from the oxidative degradation of unsaturated C18 fatty acids (Drawert et al., 1983). However, its flavor composition after cooking has not been investigated.

Aroma extract dilution analysis (AEDA) is a screening method to evaluate the potent odorants present in a food product (Ullrich and Grosch, 1987; Grosch, 1993; Blank and Grosch, 1991). AEDA is limited to odorants with boiling points higher than the solvent used for the extraction and dilution steps. Furthermore, odorants whose boiling points are in the same range as that of the extraction solvent are partially lost during concentration of the extract by distilling off the solvent. To overcome this limitation, an AEDA should be performed by gas chromatography−olfactometry of a static head-space sample (GCO-SH) (Holscher and Steinhart, 1992; Guth and Grosch, 1993; Semmelroch and Grosch, 1995). The results of this analysis are expressed as a flavor dilution (FD) factor. The FD factor for a compound is the ratio in the initial extract to its concentration in the most dilute extract in which an odor was detected by GCO.

In the present study AEDA analysis and GCO-SH were performed to identify the most potent odorants of huitlacoche and austern pilzen in cooked samples.

MATERIALS AND METHODS

Samples. Huitlacoche was harvested in Mexico in an immature stage, and the galls were separated and cleaned from the corn cob and other extraneous and green materials. Austern pilzen was purchased from a local market in Munich, Germany. Both materials were lyophilized and stored in sealed containers under refrigeration prior to analysis. Reference compounds 1−5, 8−12, 14−20, 22, 24, 25, 28, 30, 31, 35−37, and 39−43 were purchased from Aldrich (Steinheim, Germany) (Table 1). 1-Octen-3-one was a gift of Dr. Emberger (Haarmann Reimer, Hotzminchen, Germany). Compounds 21, 27, and 32 were synthesized according to Ullrich and Grosch (1988), Schieberle and Grosch (1988), and Gassenmeier and Schieberle (1994), respectively.

All solvents were purified as done by Schieberle and Grosch (1983). Silica gel 60 (Merck, Darmstadt, Germany) was treated with HCl and dried at 105 °C to a water content of 1.5% by mass according to Esterbauer (1968).

Isolation of Volatiles. Lyophilized samples of huitlacoche or austern pilzen (100 g) were soaked in water (1 L) and then homogenized in a Waring blender for 1 min. The samples were cooked for 3 min at 120 °C and homogenized again for 30 s by mean of ultraturax. The suspension was extracted twice with 500 mL of diethyl ether. The combined organic layers were

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Table 1. Odorants of Huitlacoche and Austern Pilzen on the Basis of AEDA

compound	RI[a]		odor quality	FD[b] Factor	
	SE-54	FFAP		huitlacoche	austern pilzen
1. methylpropanal[c]		<700	malty	>[f]	1
2. butane-2,3-dione[c]	<700	981	buttery	1	
3. hexanal[c]	800	1075	green	100	1
4. methional[d]	900	1448	boiling potatoes	1	10
5. phenol[c]	910	1995	fenolic	1	>
6. 2-acetyl-2-pyrroline	916	1327	roasty	>	10
7. 1-octen-3-one	963	1290	mushroom-like	1	1
8. 1-octen-3-ol[c]	978	1440	mushroom-like	1	10
9. 3-octanol[c]	987	1379	sweet	>	10
10. propanoic acid[c]	993	1540	soapy-sweet	>	1
11. octanal[c]	1000	1279	soapy-fruity	100	1
12. phenylacetaldehyde[c]	1035		sweet honey-like	1	>
13. 5-hydroxy-6-methyl-(2*H*)-pyran-2-one[e]	1040	2119	caramel	>	1
14. (*E*)-oct-2-enal[c]	1054		fatty	1	>
15. 2,5-dimethyl-4-hydroxy-3(2*H*)-furanone	1065	2015	caramel	>	1
16. *o*-methoxyphenol (guaiacol)	1099	1859	fenolic	1	>
17. nonanal[c]	1100		fatty	1	>
18. 2-acetyl-2-thiazoline	1106	1781	roasty	>	1
19. phenylethanol[c]	1118	1889	flowery-sweet	10	>
20. 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone (sotolon)[d]	1122	2172	caramel	10	1000
21. (*Z*)-non-2-enal[d]	1142	1500	fatty	1	>
22. (*E*)-non-2-enal[c]	1156	1522	fatty	1	>
23. unknown 1	1192		fatty	1	>
24. decanal[c]	1207	1500	sweet-orange	100	>
25. (*E*,*E*)-nona-2,4-dienal[c]	1218	1696	fatty	10	1
26. dec-2-enal[e]	1245		fatty	1	>
27. (*E*,*Z*)-deca-2,4-dienal[c]	1290		fatty	1	>
28. (*E*,*E*)-deca-2,4-dienal[c]	1309	1790	fatty-fried fat	100	1
29. undec-2-enal[e]	1346		sweet-fresh	1	>
30. γ-nonalactone[c]	1352	2018	coconut	1	>
31. (*E*)-undec-2-enal	1361	1736	sweet-fresh	100	>
32. (*E*)-4,5-epoxydec-2-enal[c]	1378	1990	metallic	1	>
33. 2-nonenoic acid γ-lactone	1394	2052	coconut	1	>
34. vanillin[c]	1408	2550	vanilla-like	100	1
35. (*E*,*E*)-undeca-2,4-dienal	1416		fatty	1	>
36. γ-decalactone[c]	1445		coconut	>	1
37. unknown 2	1746	2368	mushroom-like	>	1000
38. octanoic acid[c]		2042	sweet	>	1
39. butyric acid[c]		1625	fatty-rancid	1	>
40. 3-methylbutyric acid[c]		1665	fatty-rancid	1	>
41. hexanoic acid[c]		1835	rancid	10	>
42. heptanoic acid[c]		1942	rancid	10	>

[a] Retention index. [b] Flavor dilution factor by GCO. [c] The compound was identified by comparison with a reference substance on the basis of the following criteria: RI on two capillary columns, mass spectra obtained by MS-EI and MS-CI and odor quality perceived at the sniffing port. [d] The compound was identified by comparison it with a reference compound on the basis of RI on two capillary columns. [e] Identification based on comparison with MS data from literature. [f] The symbol > designates that the odorant was not perceived by AEDA.

washed with 400 mL of an aqueous saturated NaCl solution and dried over anhydrous Na_2SO_4.

The extract obtained was concentrated to 100 mL on a Vigreux column (50 × 1 cm) by distilling off the solvent at 40 °C. The volatile compounds were isolated on a high-vacuum distillation apparatus previously described by Sen et al. (1991). The extract was poured into the flask of a distillation apparatus equipped with two traps cooled with liquid nitrogen. After the sample was frozen in the distillation flask with liquid nitrogen, the pressure in the apparatus was reduced to 6 mPa, and the solution of the volatiles was distilled within 2 h. The temperature of the water bath was increased by 50 °C, and the distillation continued for a further 2 h (Semmelroch et al., 1995). The condensate of the first trap was rinsed with diethyl ether (10 mL) and then separated into acid and neutral fractions by treatment with aqueous Na_2CO_3 (0.5 M, 2 × 50 mL). The pH of the combined aqueous solution was adjusted to 3 by addition of 32% HCl, and then the solution was extracted twice with diethyl ether (Guth and Grosch, 1993; Schieberle and Grosch, 1987a). The organic solutions of the neutral and acid compounds were each concentrated stepwise to 2 mL in a Vigreux column (50 × 1 cm) and to 100 μL by microdistillation in the apparatus described by Bemelmans (1979).

Column Chromatography. The neutral fractions of both samples (100 g) were fractionated at 10−12 °C on a water-cooled column (30 × 1.6 cm) packed with a slurry of silica gel 60 in pentane. The volatiles were eluted using the following solvent sequence: 200 mL of 5% (v/v) fraction A, 150 mL of 10% (v/v) fraction B, 150 mL of 30% (v/v) fraction C, and 150 mL of 100% (v/v) diethyl ether in pentane. Each of the fractions was concentrated first on a Vigreux column and then by microdistillation (Bemelmans, 1979).

Capillary Gas Chromatography−Mass Spectrometry (MS). Capillary gas chromatography (HRGC) was performed by means of Carlo Erba gas chromatography, type 4200. The thin film capillaries used were (i) capillary SE-54 (30 m × 0.32 mm) and (ii) DB-FFAP (30 m × 0.32 mm). The samples were applied by an on-column injection technique at 35 °C, and the temperature of the capillaries was raised by 40 °C/min to 60 °C, held for 1 min isothermally, and then raised by 6 °C/min to 250 °C. The flow rate of the carrier gas (helium) was 2.2 mL/min. At the end of the capillaries, the effluent was split 1:1 to a flame ionization detector (FID) and a sniffing port. The FID and the sniffing port were held at a temperature of 200 °C. The splitter was flushed with helium to accelerate the split flow to 10 mL/min. Nitrogen (20 mL/min) was used as a makeup gas for the FID (Jung et al., 1992; Schieberle and Grosch, 1983).

Retention data of the compounds are presented as retention indices (RI) calculated according to Halang et al. (1978). Capillary gas chromatography−mass spectrometry (HRGC−

Table 2. Results of the Static Head-Space Analysis of Huitlacoche (*U. maydis*) and Austern Pilzen (*Pleurotus* sp.)

odorant	RI[a] SE-54	vol[b] (mL)	
		huitlacoche	austern pilzen
propanal	<500	1	>[c]
methylpropanal	<600	1	0.1
butane-2,3-dione	<600	5	1
3-methylbutanal	650	1	1
2-methylbutanal	660	1	1
pentanal	700	5	>
ethyl isobutyrate	763	1	>
unknown	784	>	10
hexanal	800	1	1
unknown	822	>	10
ethyl 2-/3-methylbutyrate	852	1	>
heptan-2-one	899	>	10
methional	906	5	10
2-acetyl-2-pyrroline	929	>	10
1-octen-3-one	908	10	0.1
1-octen-3-ol	978	>	0.1
3-octanol	987	>	1
unknown	992	5	>
propanoic acid	993	>	0.1
octanal	1000	1	0.1
unknown	1059	>	10
(*Z*)-non-2-enal	1146	10	>
(*E*,*E*)-deca-2,4-dienal	1318	>	10
(*E*)-undec-2-enal	1367	10	>

[a] Retention index in SE-54 capillary column. [b] The lowest head-space volume which was required to perceive the odorant at the sniffing port. [c] The symbol > designates that the odorant was not perceived by GCO-SH.

MS) analyses were performed with a MS 8230 (Finnigan, Bremen, Germany) in tandem with the capillaries and conditions described above. Mass spectra in the electron impact mode (MS-EI) were generated at 70 eV and in the chemical ionization mode (MS-CI) at 115 eV with isobutane as reagent gas.

Aroma Extract Dilution Analysis. The FD factors of the odorants were determined by an AEDA (Ullrich and Grosch, 1987; Schieberle and Grosch, 1987b; Guth and Grosch, 1990). The volatiles distilled off from 10 g of sample were dissolved in a total volume of 100 mL of diethyl ether and concentrated to 1 mL by distillation and microdistillation (Bemelmans, 1979). This concentrate was diluted stepwise in a dilution series of 1:10, 1:100, and 1:1000. The HRGC effluent sniffing was performed with samples of 0.5 mL of each dilution in a DB-FFAP capillary with the conditions described above.

Gas Chromatography−Olfactometry of Static Head-Space. Five milliliters of water and 500 mg of lyophilized sample of huitlacoche or austern pilzen were put into a vessel (volume 25 mL) sealed with a septum and cooked for 5 min at 120−130 °C. After that, the samples were held for 1 h in a water bath at 40 °C. A head-space volume detailed in Table 2 was drawn by a gastight syringe and then injected into a CP-9001 gas chromatograph connected to the purge and trap system TC/PTI 4001 (Chrompack, Frankfurt, Germany) operated in similar way as reported earlier by Guth and Grosch (1993) and Semmelroch and Grosch (1995). The thin film capillary used for static GCO-SH was SE-54 (fused silica, 30 m × 0.53 mm, film thickness 1.5 μm), and the flow of the carrier gas was 3 mL/min. The oven temperature was held at 35 °C for 1 min and then raised quickly (8 °C/min) to 230 °C. At the end of each capillary column, the effluent was split (1 + 1 v/v) into a FID and a sniffing port using deactivated fused silica capillaries (30 cm × 0.15 mm). The FID and the sniffing port were held at a temperature of 200 °C. Nitrogen (20 mL/min) was used as makeup gas for the FID. After each GCO-SH run, the purge system was automatically cleaned (cleanup flow, 50 mL of helium; cleanup temperature, 275 °C).

RESULTS AND DISCUSSION

The volatile fractions of huitlacoche and austern pilzen mushrooms originating from Mexico and Germany, respectively, were isolated and analyzed by AEDA and static head-space analysis. The number of odorants found in the two mushroom samples are listed in Table 1. As it can be seen, there are 32 compounds for huitlacoche and 19 for austern pilzen. Among these compounds the FD factors are in the range 1−1000.

According to the results listed in Table 1, only nine compounds were the same in both mushrooms. Among these, the most important was sotolon (FD factor of 10 and 1000 for huitlacoche and austern pilzen, respectively). This compound was previously reported by Guth and Grosch (1993); however, this is the first knowledge of sotolon in mushroom material. Another very potent compound found in austern pilzen was an unknown with a FD factor of 1000 which has a mushroom-like note. On the basis of their high FD factors, hexanal, octanal, decanal, (*E*,*E*)-deca-2,4-dienal, (*E*)-undec-2-enal, and vanillin are considered the main key odorants for huitlacoche, and sotolon and an unknown, for austern pilzen, respectively.

Table 2 shows the results obtained from the static head-space analyses of both mushrooms. On the basis of their chromatographic and sensorial characteristics, odorants such as methylpropanal, 1-octen-3-one, 1-octen-3-ol, propanoic acid, and octanal presented the lowest volumes needed for their detection. These compounds were also detected by the AEDA studies (Table 1); with the exception of propanoic acid, the other four odorants in austern pilzen were also detected in huitlacoche. The presence of compounds such as hexanal, 3-methylbutanal, phenol, 1-octen-3-ol, octanal, phenylacetaldehyde, nonanal, decenal, decanal, (*Z*)-undec-2-enal, and (*E*)-undec-2-enal was previously reported by Lizárraga-Guerra (1995) in huitlacoche. On the other hand, only 1-octen-3-ol and 1-octen-3-one have been reported for *Pleurotus flabellatus* (Bano and Rajarathnam, 1988). Other compounds such as methional and DMHF have been identified in popcorn (Schieberle, 1991), and the same compounds have also been found in tomato aroma (Buttery et al., 1995). (*E*)-4,5-Epoxydec-2-enal in rapeseed oil (Guth and Grosch, 1990) and methylpropanal, butane-2,3-dione, and 2- or 3-methylbutanal in roasted coffee (Semmelroch and Grosch, 1995) have been identified in AEDA studies or head-space analysis. In Table 2, only four odorants were not identified by the head-space analysis, one for huitlacoche with a 5 mL volume and three for austern pilzen all with 10 mL volume. In addition, compounds such as propanal, pentanal, isobutyric acid ethyl ester, ethyl 2-/3-methylbutyrate, (*Z*)-non-2-enal, and (*E*)-undec-2-enal found in huitlacoche were not detected in austern pilzen.

The major difference between the volumes reported for 1-octen-3-one for both mushroom materials can be explained on the basis of the concentration of this compound in each sample (Table 2).

Only 8 out of 32 compounds found by the AEDA screening were also detected by the head-space analysis in huitlacoche. On the other hand, 11 out of 19 were found in both analyses for austern pilzen.

That the huitlacoche volatiles are mainly aldehydes may be due to the oxidation of fatty acid such as oleic and linoleic acids (Ullrich and Grosch, 1987); this assertion agrees well with the fatty acids composition found for huitlacoche (unpublished data).

This study demonstrated that a large number of compounds play an important role in the overall aroma of huitlacoche (hexanal, octanal, decanal, (*E*,*E*)-deca-2,4-dienal, (*E*)-undec-2-enal, and vanillin) and only a few

in the case of austern pilzen (sotolon and an unknown). The results also indicated that compounds such as hexanal, octanal, decanal, (*E*,*E*)-deca-2,4-dienal, (*E*)-undec-2-enal, and vanillin, and sotolon and a compound of unknown structure, were the most potent odorants of huitlacoche and austern pilzen, respectively. On the other hand, the FD factors of each of the odorants found in both mushrooms varied widely; this indicates that the relative concentrations of the odorants were different in both samples.

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