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Odour-active compounds in moderately roasted sesame

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Aroma extract dilution analysis (AEDA) of an extract prepared from moderately roasted sesame (180°C; 10 min) revealed 41 odour-active volatiles, 31 of which could be identified by comparison of their mass spectra, retention indices, odour quality and odour threshold with reference compounds. Of the 18 aroma compounds showing very high Flavour Dilution factors in the range of 128–2048, 10 compounds [2-furfurylthiol, 2-phenylethylthiol, 2-methoxyphenol, 4-hydroxy-2,5-dimethyl-3(2H)-furanone, 2-pentylpyridine, 2-ethyl-3,5-dimethylpyrazine, acetylpyrazine, (E,E)-2,4-decadienal, 2-acetyl-1-pyrroline and 4-vinyl-2-methoxyphenol] were quantified by means of stable isotope dilution assays and their odour activity values (OAV; ratio of concentration to odour threshold) were calculated. On the basis of high OAVs in oil, especially 2-acetyl-1-pyrroline (roasty), 2-furfurylthiol (coffee-like), 2-phenylethylthiol (rubbery) and 4-hydroxy-2,5-dimethyl-3(2H)-furanone (caramel-like) were elucidated as important contributors to the overall roasty, sulphury odour of the crushed sesame material.

INTRODUCTION

Roasting of the rather odourless sesame seeds generates an intense flavour characterized by roasty, burnt, meat-like or sulphury odour notes. Until recently, only a few volatiles had been identified in extracts of roasted sesame (Takei *et al.*, 1969; Manley *et al.*, 1974). However, recent results (Takei, 1988; Nakamura *et al.*, 1989) have increased the number of volatiles identified to more than 220.

A combination of instrumental with sensory analysis had not been applied in these investigations and, therefore, the contributions of single volatiles to the sesame aroma were still open. By application of an aroma extract dilution analysis (AEDA) on an extract of roasted white sesame seeds, the author (Schieberle, 1993) could recently identify especially 2-furfurylthiol, 2-phenylethylthiol and 4-hydroxy-2,5-dimethyl-3(2H)-furanone as the most odour-active compounds in sesame seeds which had been roasted at 180°C for 30 min.

The overall flavour of roasted sesame significantly depends on the roasting conditions and changes in the relative concentrations of volatiles have been observed depending on roasting time and temperature (Takei, 1988).

The purpose of the present investigations was, therefore, to identify the key odorants in moderately roasted seeds and to compare them with our previous results.

MATERIALS AND METHODS

The following chemicals were obtained from the sources given in parentheses: phenylacetic acid chloride and compounds numbered 7a, 8, 14, 15a, 16, 18, 20, 22, 24, 25, 27, 30, 32, 35, 38 and 39 in Table 1 (Aldrich, Steinheim, Germany); Nos 1, 34 and 36 (Lancaster, Mühlheim, Germany); No. 2 (Merck, Darmstadt, Germany) and No. 13 (Pyrazine Specialties, Atlanta, USA).

Synthesis

2-Pentylpyridine

1-Chloropentane (50 mM), dissolved in *n*-pentane (20 ml), was added within 1 h to a stirred suspension of lithium (100 mM) in *n*-pentane (20 ml) which was refluxed under a nitrogen atmosphere. After heating for another hour, the mixture was cooled to 0°C and then a solution of pyridine (50 mM) in *n*-pentane (10 ml) was added dropwise within 30 min. The mixture was refluxed for another 15 min, and then poured onto a mixture of ice (25 g)–hydrochloric acid (25 ml; 1% by weight). After adjusting the pH to 7.0, the corresponding alkylpyridine was extracted with diethylether (total volume 240 ml) and, after drying over NA_2SO_4, purified by distillation *in vacuo* (K_p 121°C at 10 mm). The target compound was characterized by its mass spectra

Table 1. Odour-active compounds [Flavour Dilution (FD)≥8] in moderately roasted sesame seeds

No.	Compound^{a}	Odor description^{b}	Fraction^{c}	RI on		FD-factor	Earlier reported among the volatiles of sesame^{d}
				DB-5	FFAP		
1	2,3-Butanedione	Buttery	A	< 600	980	8	—
2	3-Methylbutanal	Malty	A	< 600	905	128	—
3	Unknown	Sulphury, meaty	—	730	—	16	—
4	Unknown	Sulphury, wort-like	—	830	—	16	—
5	Unknown	Meat-like, sulphury	—	885	—	128	—
6	Unknown	Catty	A	895	—	256	—
7a	Methional	Cooked potato	C	908	1449^{e}	} 128	—
7b	Unknown	Roasty, catty	A	—	—		
8	2-Furfurylthiol	Roasty, coffee-like	A	912	1430^{e}	2048	4, 6
9	2-Acetyl-1-pyrroline	Roasty, popcorn-like	D	921	1314^{e}	512	—
10	4-Methyl-3-thiazoline	Garlic, carbide-like	D	936	1438	128	—
11	1-Octen-3-one	Mushroom-like	B	980	1278	256	—
12	Unknown	Catty	A	—	—	64	—
13	2-Ethyl-(5)6-methylpyrazine	Pyrazine-like, fruity	D	998	1390	8	2–6
14	Trimethylpyrazine	Burnt, potato-like	D	1000	1287	32	2–4, 6
15a	2-Acetylthiazol	Roasty, sulphury	D	1013	1137^{f}	} 16	4,6
15b	5-Methyl-2-furfurylthiol	Roasty, sulphury	A	1016	1527^{e}		—
16	Acetylpyrazine	Roasty	D	1023	1603	64	1, 2, 6
17	Unknown	Sulphury	—	—	—	256	—
18	Phenylacetaldehyde	Honey-like	B	1050	1610	64	—
19	Unknown	Sulphury	—	—	—	256	—
20	4-Hydroxy-2,5-dimethyl-3(2H)-furanone^{g}	Caramel-like	acidic	1070	2066	512	4
21	2-Ethyl-3,5-dimethylpyrazine	Potato-like, roasty	D	1083	1458	512	2–4, 6
22	2-Methoxyphenol	Burnt, sweet	C	1092	1863	256	1, 3, 4, 6
23	(Z)-2-Nonenal	Green, tallowy	B	1148	1479	8	—
24	2,3-Diethyl-5-methylpyrazine	Potato-like, roasty	D	1158	1483	128	2, 3, 6
25	(E)-2-Nonenal	Green	B	1161	1520	64	3, 6
26	2-Methyl-[3-methyldithio]-furane	Meat-like	A	1172	1676^{e}	16	—
27	2-Phenylethylthiol	Burnt-rubbery	A	1176	1622	512	—
28	Unknown	Green, leaf-like	—	—	—	—	—
29	2-Pentylpyridine	Fatty, tallowy	D	1192	1507	64	6
30	(E,E)-2,4-Nonadienal	Fatty, waxy	B	1213	1704	32	—
31	Unknown	Green, fatty	—	—	—	32	—
32	Indole	Mothball-like	D	1292	1550^{e}	16	6
33	(E,Z)-2,4-Decadienal	Green, tallowy	B	1296	1413^{e}	8	—
34	2-Methoxy-4-vinylphenol	Spicy	C	1312	1474^{e}	512	4, 6
35	(E,E)-2,4-Decadienal	Fatty, waxy	B	1318	1800	128	3, 6
36	(E)-2-Undecenal	Fatty, green	B	1366	1465	8	—
37	4,5-Epoxy-(E)-2-decenal	Metallic	B/C	1381	1962	128	—
38	3-Methylindole (Skatol)	Mothball-like	D	1390	1629^{f}	64	6
39	Vanillin^{g}	Vanilla-like	acidic	1403	2577	64	6

[a] The compound was identified by comparing it with the reference substance on the basis of the following criteria: Retention index (RI) on the capillaries given in the table, mass spectra obtained by electron impact (MS/EI) or chemical ionization (MS/CI), odour quality perceived at the sniffing port and odour threshold in air.

[b] Odour description assigned during aroma extract dilution analysis (AEDA).

[c] Fraction in which most of the compound appeared after separation of the volatiles by column chromatography.

[d] 1, Takei *et al.*, 1969; 2, Manley *et al.*, 1974; 3, Soliman *et al.*, 1975; 4, Takei, 1988; 5, El-Sawy *et al.*, 1989; 6, Nakamura *et al.*, 1989.

[e] The RI was determined on capillary DB-Wax.

[f] The RI was determined on capillary OV-1701 (30 m × 0.32 mm fused silica capillary DB-7; Fisons Instruments, Mainz, Germany).

[g] The acidic volatiles were isolated by treatment of the etheral extract with an aqueous sodium bicarbonate solution (0.1 mol litre^{-1}).

obtained in the electron impact mode (MS/EI; Fig. 1A) and in the chemical ionization mode (MS/CI; Fig. 2A).

[^{2}H]-2-Pentylpyridine

The labelled standard was synthesized from 1-chloropentane and [^{2}H]$_{5}$-pyridine (MSD isotopes, Munich, Germany) as described above for the unlabelled compound. A comparison of the MS/EI of the unlabelled (Fig. 1A) and the labelled 2-pentylpyridne (Fig. 1B) in combination with the data obtained by MS/CI (Fig. 2A and B) confirmed the incorporation of four deuterium atoms in the [^{2}H]-2-pentylpyridine.

[^{2}H]-2-Phenylethylthiol

Lithium aluminum deuteride (1 g) was suspended in dry diethyl ether (50 ml) maintained under a nitrogen atmosphere. With stirring, a solution of phenylacetic acid chloride (20 mM) was added dropwise within 1 h such as to maintain gentle reflux. After stirring for another 30 min, the mixture was cooled to 0°C and,

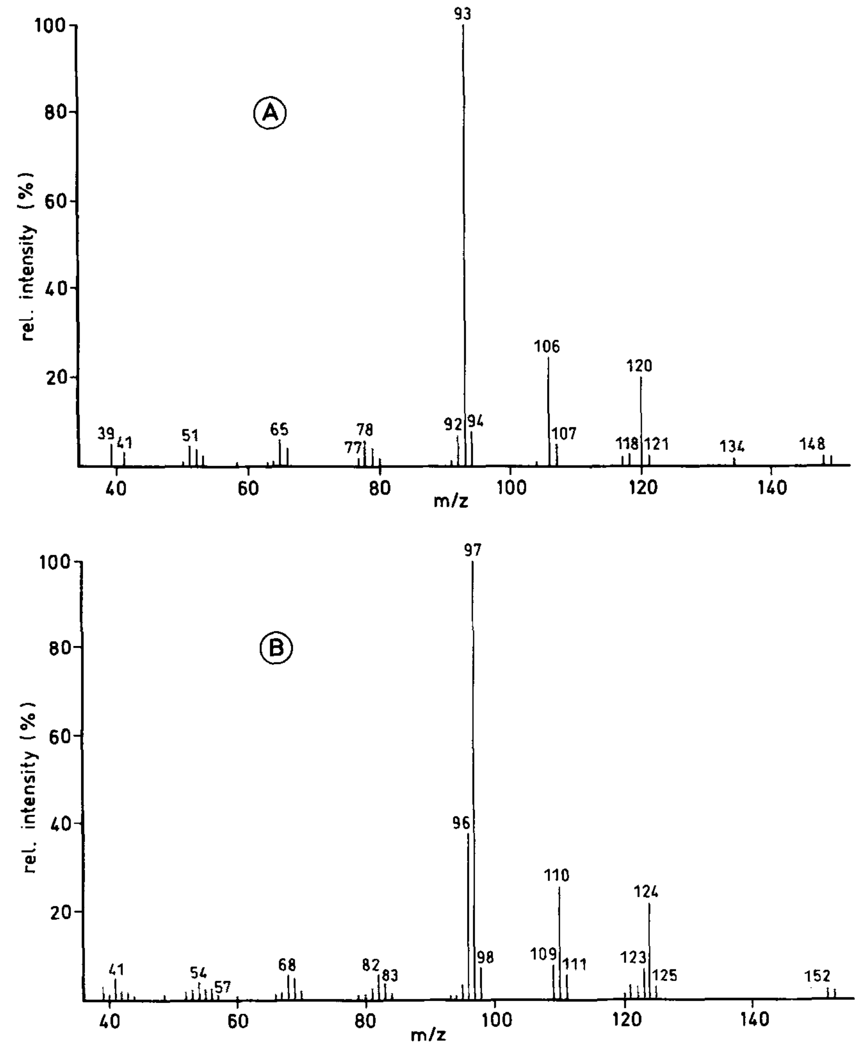

Fig. 1. Mass spectra (electron impact mode) of (A) 2-pentylpyridine and (B) [^{2}H]-2-pentylpyridine.

after addition of water, extracted with diethylether (total volume: 100 ml). After drying over Na_2SO_4 the solvent was carefully distilled off. The [^{2}H]-2-phenylethanol obtained was dissolved in a mixture of dichloromethane (20 ml)–pyridine (60 mM) and after addition of PBr_3 (10 mM) with cooling, stirred for 20 h at room temperature. The [^{2}H]-2-phenylethylbromide formed was extracted from the reaction mixture with *n*-pentane (total volume: 150 ml) and then converted into the target compound by a reaction with thiourea as described recently in the preparation of [^{2}H]-2-furfurylthiol (Sen & Grosch, 1991). A comparison of the mass spectrum (MS/EI) of the labelled and the unlabelled 2-phenylethylthiol (Fig. 3A and B) confirmed the presence of two deuterium atoms in the [^{2}H]-2-phenylethylthiol by a shift of the M^+-ion from *m/z* 138 to *m/z* 140 in the labelled thiol. For quantitative measurements the fragments *m/z* 107 and *m/z* 105 (cf. Table 2) formed by elimination of H_2S from the protonated molecular ions ($M^+ + 1$: 141, [^{2}H]-2-phenylethylthiol; $M^+ + 1$: 139, 2-phenylethylthiol) were used.

The following compounds were synthesized as reported in the sources given in parenthesis: [^{13}C]-4-hydroxy-2,5-dimethyl-3(2H)-furanone (Sen *et al.*, 1991*a*); [^{2}H]-2-furfurylthiol (Sen & Grosch, 1991); [^{2}H]-2-methoxyphenol and [^{2}H]-2-ethyl-3,5-dimethylpyrazine (Cerny & Grosch, 1993); [^{2}H]-2-acetyl-1-pyrroline and [^{2}H]-acetylpyrazine (Schieberle & Grosch, 1987); [^{2}H]-(E,E)-2,4-decadienal (Guth & Grosch, 1990); 2-acetyl-1-pyrroline (Buttery & Ling, 1982); 2-methyl-[3-methyldithio]-furane (Gasser & Grosch, 1990); 4,5-epoxy-(E)-2-decenal (Schieberle & Grosch, 1991); 5-methyl-2-furfurylthiol (Hofmann & Schieberle, in preparation); 1-octen-3-one (Guth & Grosch, 1990); 4-methyl-3-thiazoline (Schieberle *et al.*, in preparation). (E,Z)-2,4-decadienal was isolated from commercial (E,E)-2,4-decadienal and characterized as reported recently (Gassenmeier & Schieberle, 1994).

Roasting of the sesame seeds

White sesame seeds (Mexico) were filled into glass tubes (4.5 × 1 cm internal diameter) and heated for 10 min at 180°C in a metal block. After freezing with liquid nitrogen, the seeds were crushed by means of a commercial blender.

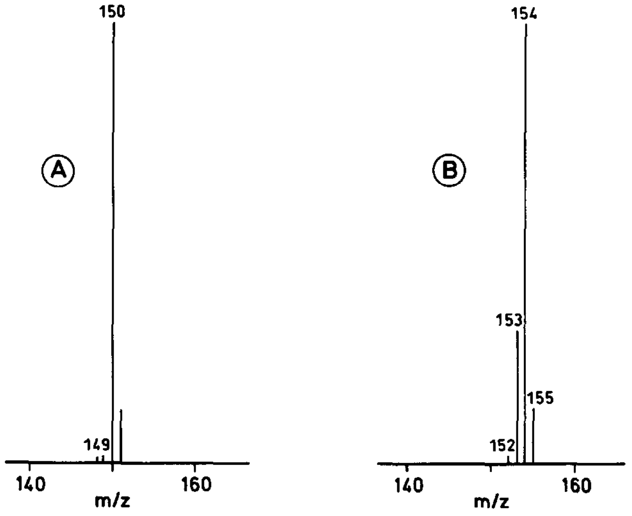

Fig. 2. Mass spectra (chemical ionization) of (A) 2-pentylpyridine and (B) [^{2}H]-2-pentylpyridine.

Isolation of the volatiles

A suspension of the freshly roasted, slightly brownish material (100 g) in diethylether (150 ml) was stirred for 20 min, and then filtered. The procedure was repeated twice, the combined etheral extracts (total volume: 250 ml) were dried over Na_2SO_4, concentrated to 150 ml by distilling off the solvent at 35°C over a Vigreux-column. The volatiles and the solvent were isolated by high vacuum stripping of the oily liquid by using the apparatus described by Guth & Grosch (1989). The distillate was concentrated to 100 μl and evaluated by aroma extract dilution analysis (AEDA) as described recently (Schieberle, 1991).

For the identification experiments, the volatiles obtained from 1 kg of roasted material were applied onto five water cooled columns (20 × 1 cm) filled with a slurry of silica gel and separated at 10–12°C into four fractions (A–D; cf. Schieberle, 1991) by using *n*-pentane–diethyl ether mixtures of increasing polarity.

High resolution gas chromatography (HRGC)–mass spectrometry (MS)

HRGC was performed on a gas chromatograph Type 5300 (Fisons, Mainz, Germany) using the following thin-film capillary columns: capillary DB-5 (30 m × 0.32 mm fused silica capillary DB-5 equals SE-54; Fisons Instruments, Mainz, Germany), capillary FFAP (30 m × 0.32 mm fused silica capillary FFAP; Fisons Instruments, Mainz, Germany) and capillary DB-Wax (30 m

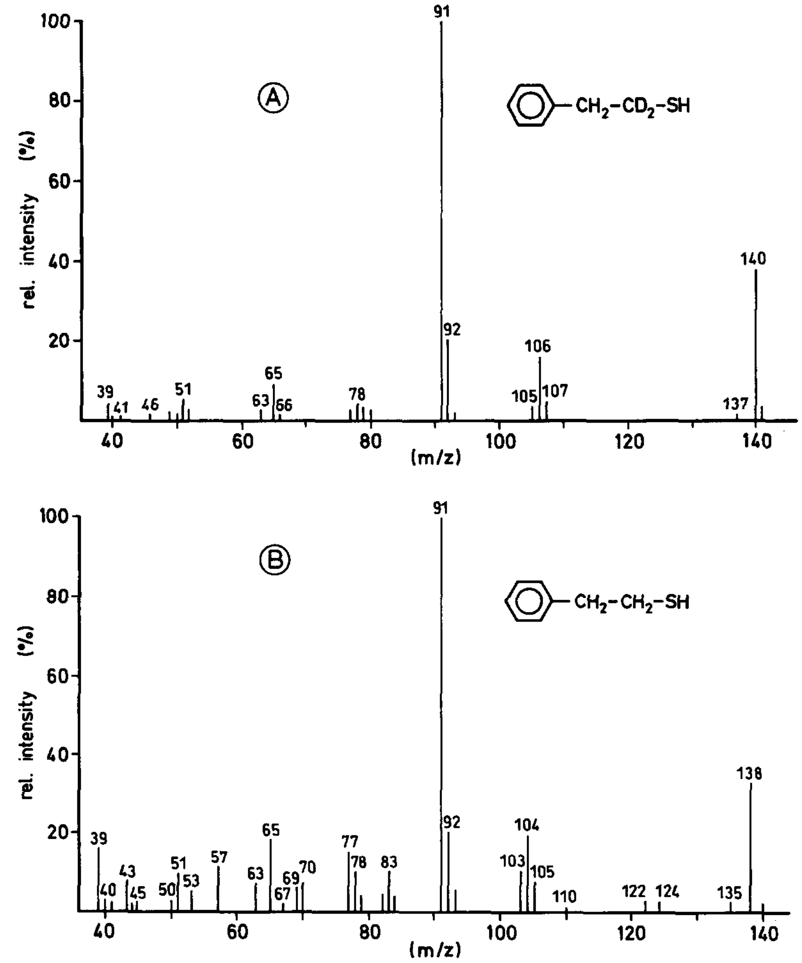

Fig. 3. Mass spectra of (A) [^{2}H]-2-phenylethylthiol and of (B) 2-phenylethylthiol.

Table 2. Selected ions used for the quantification of key odour active compounds of sesame by mass chromatography

Odorant	Fraction[a]	Selected ion (m/z) of	
		unlabelled compound	labelled compound
2-Furfurylthiol[b]	A	81	83
2-Phenylethylthiol	A	105	107
2-Methoxyphenol	B + C	125	128
4-Hydroxy-2,5-dimethyl-3(2H)-furanone[c]	acidic	129	131
2-Pentylpyridine	D	150	154
2-Ethyl-3,5-dimethylpyrazine	D	137	140
Acetylpyrazine	D	123	126
(E,E)-2,4-Decadienal	B	153	155–157
2-Acetyl-1-pyrroline	D	112	114–117
4-Vinyl-2-methoxyphenol[d]	B + C	151	155

[a] Fraction obtained by column chromatography in which the aroma compound and the internal standard was determined.
[b] Measured in the electron impact mode (MS/EI).
[c] Analysis was performed on capillary FFAP.
[d] Determined by using [^{2}H]-4-ethyl-2-methoxyphenol as the internal standard (Blank *et al.*, 1992).

Table 3. Concentrations and odour activity values (OAV) of 10 selected flavour compounds in sesame

Flavour compound(odour threshold in g litre^{-1} oil)	Concentration (μg kg^{-1})	OAV[a]
2-Acetyl-1-pyrroline (0.1)	30	300
2-Furfurylthiol (0.4)	54	135
2-Phenylethylthiol (0.05)	6	120
4-Hydroxy-2,5-dimethyl-3(2H)-furanone (50)	2511	50
2-Ethyl-3,5-dimethylpyrazine (3)	53	18
2-Methoxyphenol (19)	269	14
2-Pentylpyridine (5)	19	4
Acetylpyrazine (10)	26	3
4-Vinyl-2-methoxyphenol (50)	72	1
(E,E)-2,4-Decadienal (180)	89	< 1

[a] Odour activity values (OAVs) were calculated by deviding the concentration by the odour threshold in sunflower oil.

× 0.32 mm fused silica capillary DB-Wax; Fisons Instruments, Mainz, Germany) by the cold on-column technique as described recently (Schieberle, 1991). MS analyses were performed by means of a mass spectrometer MS 8230 (Finnigan, Bremen, Germany) in tandem with the capillaries 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 (AEDA)

The flavour dilution (FD) factors of the odour active compounds were determined by HRGC–olfactometry (cf. review by Grosch, 1993) of the following dilution series: the original extract (100 μl) containing the volatiles from 100 g of roasted sesame seeds was diluted stepwise by addition of diethyl ether. Aliquots (0.5 μl) were analysed by HRGC–olfactometry using the capillary DB-5. Retention indices (RI) were calculated by using mixtures of *n*-alkanes.

Analyses were performed by two assessors in duplicates. The results differed to not more than two Flavour Dilution (FD) factors.

Odour thresholds in oil

Ethanol (100 μl) containing a definite amount of the purified reference odorant was pipetted into sunflower oil (250 ml; commercial rather odourless product) and the solution was equilibrated by stirring for 30 min at room temperature. This stock solution was diluted stepwise (1:1 by vol.) with sunflower oil and 25 ml of the dilutions were filled into closed glass vessels (65 mm height; i.d. 40 mm; total volume 45 ml). The samples with increasing concentrations were smelled by eight panellists using the triangular test. Amounts detected by at least six of the eight panellists are reported in Table 3. Twenty-five millilitres of an oil prepared from a stock solution containing 10 μl of pure ethanol or the corresponding dilutions, respectively, were used as the control.

Stable isotope dilution analyses

The roasted material (100 g) was suspended in diethyl ether (250 ml) containing the 10 labelled standards (cf. Table 2; 20 μg each) and stirred for 30 min. From the filtrate, the volatiles, the internal standards and the

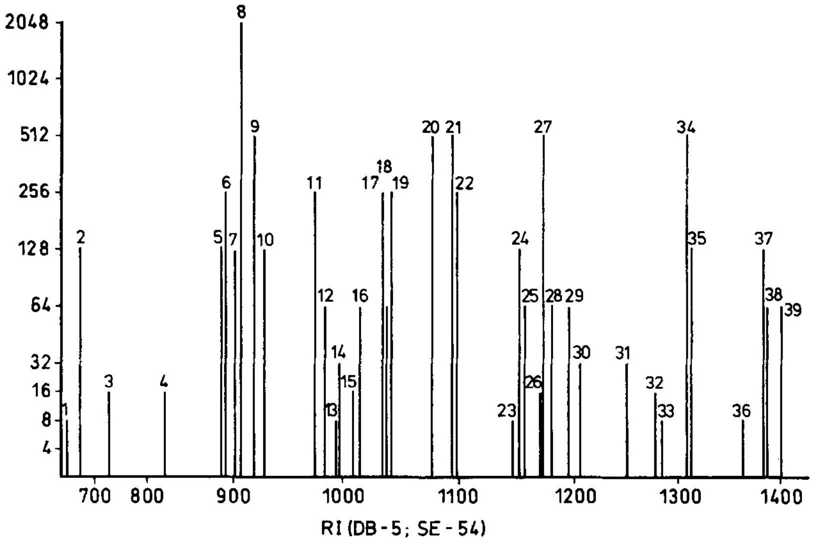

Fig. 4. Flavour Dilution (FD)-chromatogram [FD-factors vs retention indices (RI)] of the odour-active compounds in an extract from moderately roasted sesame seeds.

solvent were then isolated by high vacuum stripping as described in 'Isolation of the volatiles'. The sublimate was separated by column chromatography into four fractions (A–D; Schieberle, 1991) and, after concentration of the fractions to about 100 μl analysed by mass chromatography on column DB-5 as detailed in Table 2. The column was coupled to the ion trap detector (ITD 800; Finnigan, Bremen, Germany) running in the chemical ionization mode with methanol as the reagent gas. Calibration factors were calculated as recently described (Sen *et al.*, 1991*b*).

RESULTS

Extract dilution analysis

The flavour of the freshly roasted, crushed kernels was evaluated by a group of eight experienced panellists and described as sweet-roasty, caramel-like and fatty, somewhat resembling fried chicken skin. To determine the compounds contributing to the overall flavour, the volatiles were isolated and analysed by aroma extract dilution analysis (AEDA). Thirty-nine odour-active regions were detected in the FD-factor range 8–2048 (Fig. 4) among which compound 8 (roasty, coffee-like) followed by compounds 9 (roasty, popcorn-like), 20 (caramel-like), 21 (roasted potato), 27 (burnt, rubbery) and 34 (spicy) showed the highest FD factors. The results of the identification experiments (Table 1) performed by using an extract from 1 kg of the roasted material revealed 2-furfurylthiol (No. 8), 2-acetyl-1-pyrroline (No. 9), 4-hydroxy-2,5-dimethyl-3(2H)-furanone (No. 20), 2-ethyl-3,5-dimethylpyrazine (No. 21), 2-phenylethylthiol (No. 27) and 4-vinyl-2-methoxyphenol (No. 34) as the most odour-active compounds in the extract.

In addition, another 20 aroma compounds (Nos 2, 5–7, 10–12, 16–19, 22, 24, 25, 28, 29, 35, 37–39; Fig. 4) showed comparatively high FD-factors among the volatiles of the flavour extract indicating that a relatively high number of compounds is involved in the flavour of the moderately roasted sample. Fourteen of these flavour compounds were identified by comparing their analytical data (MS/EI and MS/CI; retention indices) and sensory properties (odour threshold; odour quality) with the corresponding reference compounds. In particular, the results (Table 1) indicated 3-methylbutanal (No. 2), methional (No. 7a), 4-methyl-3-thiazoline (No. 10), 1-octen-3-one (No. 11), acetylpyrazine (No. 16), phenylacetaldehyde (No. 18), 2-methoxyphenol (No. 22), 2,3-diethyl-5-methylpyrazine (No. 24), (E)-2-nonenal (No. 25), 2-pentylpyridine (No. 29), (E,E)-2,4-decadienal (No. 35), 4,5-epoxy-(E)-2-decenal (No. 37), 3-methylindole (No. 38) and vanillin (No. 39) as additional important contributors to the sesame flavour.

Odour activity values

The roasted sesame material contained about 50% fat and it seems likely that the flavour compounds are mostly dissolved in the fat phase. Therefore, the flavour contribution of 10 selected compounds was additionally elucidated by a calculation of odour activity values (ratio of concentration to odour threshold) on the basis of odour thresholds in oil.

Very low odour thresholds were determined for 2-acetyl-1-pyrroline and 2-phenylethylthiol (Table 3). On the basis of quantitative data obtained by stable isotope dilution assays the highest odour activities were calculated for 2-acetyl-1-pyrroline, followed by 2-furfurylthiol and 2-phenylethylthiol (Table 3). Contrary, 4-vinyl-2-methoxyphenol and (E,E)-2,4-decadienal did not contribute much to the sesame flavour, because their concentrations only just or did not reach their odour threshold in oil, respectively.

DISCUSSION

The results indicate 2-acetyl-1-pyrroline, 2-furfurylthiol and 2-phenylethylthiol as the most important contributors to the overall roasty, caramel-like flavour note of the moderately roasted sesame. The two thiols, but not the 2-acetyl-1-pyrroline, were also identified as key contributors to the flavour of white and black sesame seeds, which had been manufactured by longer roasting times and which elicited intense burnt or even rubbery odour notes (Schieberle, 1993, 1994). These results imply that the flavour differences observed in sesame samples heated under different conditions are undoubtedly due to significant variations in the concentrations of the key flavour compounds. These differences are probably caused by the different thermal stabilities of the respective flavour precursors on the one hand, and by the different thermal stabilities of the aroma compounds on the other hand.

2-Acetyl-1-pyrroline (ACPY) has been established also as a key flavour compound in Basmati rice (Buttery & Ling, 1982), wheat bread crust (Schieberle & Grosch, 1985), popcorn (Schieberle, 1991) and cooked sweet corn (Buttery *et al.*, 1994). This compound has been shown to be formed from a reaction of the amino acid proline with carbohydrates (Tressl *et al.*, 1985) and recently, it was shown by CHARM analysis (Roberts & Acree, 1994) that ACPY, besides 2-acetyltetrahydropyridine, is in fact a key flavour compound in such proline-containing Maillard model mixtures. Investigations on the precursors of this aroma compound in wheat bread crust and bakers yeast (Schieberle, 1990) have revealed the amino acid ornithine as a very specific precursor of ACPY. However, since no data on the amounts of free proline or ornithine in sesame are available, it is still open, whether a similar formation occurs during roasting of sesame seeds.

2-Furfurylthiol has also been detected as a key flavour compound in roasted coffee (Holscher *et al.*, 1990; Blank *et al.*, 1992) and popcorn (Schieberle, 1990). The compound has been identified in the volatile fractions of Maillard model mixtures containing cysteine and pentoses (cf. review by Tressl *et al.*, 1989) and, therefore, its formation in foods is also assumed to proceed from these precursors. However, recent results suggested (Schieberle, 1993) that in sesame the 2-furfurylthiol is formed from a water-insoluble precursor, with higher roasting temperatures and longer roasting times favour the generation of the flavour compound in sesame (Schieberle, 1994).

In contrast, the 2-phenylethylthiol has not yet been established as an important aroma compound in other food flavours.

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