

Identification and olfactometry of French fries flavour extracted at mouth conditions

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Abstract

The aim of this study was to isolate and identify odour active compounds from French fries at mouth conditions. Volatile compounds were released from French fries by purge-and-trap, trapped on Tenax TA, and identified with GC–MS. GC–olfactometry was used to determine odour active compounds with a trained panel using the detection frequency method. A total of 122 compounds were identified of which 85% originated from either sugar degradation and/or Maillard reaction and 15% from lipid degradation, based on relative areas. About 50 odour active compounds were, due to coelution, responsible for 41 odours perceived by the panel. 3-Methylbutanal and 2-methylbutanal, hexanal, 2,3-dimethylpyrazine, 2-methylpropanal, 2,3-butanedione, pyridine, heptanal, 2,5-dimethylpyrazine and/or 2,6-dimethylpyrazine and/or ethylpyrazine, dimethyl trisulfide, octanal, phenylacetaldehyde, 2,5-diethylpyrazine, (*E*)-2-nonenal, 3-methylbutanoic acid and/or 2-methylbutanoic acid, (*E,Z*)-2,4-heptadienal, (*E*)-2-octenal, 5-ethyl-2,3-dimethylpyrazine and/or 2-ethyl-3,5-dimethylpyrazine, nonanal, and tentatively 2-methylpyrrole had the highest detection frequencies. This resulted in a strong malty and fried potato note, combined with caramel/buttery, green, spicy, and deep-fried notes. Also chemical and sweaty odours were observed.

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1. Introduction

French fries are popular around the world because of their pleasant taste, which is a combination of a crispy crust, soft inside and typical fried potato flavour. The flavour of potato has been investigated extensively. According to a review by Maga (1994), more than 500 volatiles have been identified so far.

The composition of volatiles obtained from major cooking procedures differs significantly (Whitfield & Last, 1991). Therefore, a distinction should be made between raw (Petersen, Poll, & Larsen, 1998), boiled (Josephson & Lindsay, 1987; Nursten & Sheen, 1974; Oruna-Concha, Bakker, & Ames, 2002b; Petersen et al., 1998), oven baked (Buttery, Guadagni, & Ling, 1973;

Coleman & Ho, 1980; Coleman, Ho, & Chang, 1981; Duckham, Dodson, Bakker, & Ames, 2001; Duckham, Dodson, Bakker, & Ames, 2002; Ho & Coleman, 1980; Oruna-Concha, Duckham, & Ames, 2001), microwave baked (Oruna-Concha, Bakker, & Ames, 2002a; Oruna-Concha et al., 2002b), and French fried potatoes (Carlin, Jin, Huang, Ho, & Chang, 1986; Wagner & Grosch, 1997; Wagner & Grosch, 1998). Lipid oxidation is the major source of volatiles in raw potatoes, because the lipoxigenase content is relatively high (Josephson & Lindsay, 1987). In boiled potatoes the concentration of lipid-derived compounds is lower than in raw potatoes. This is either due to degradation during boiling or to less enzymatic activity since shredding is done after boiling, when enzymes are inactivated (Petersen et al., 1998). The composition of volatiles changes upon heating. The Maillard reaction becomes predominant during oven-baking of potatoes, while a microwave treatment results in a composition of volatiles between boiled and

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oven-baked potatoes (Oruna-Concha et al., 2002b). Duckham et al. (2001) compared volatiles of oven-baked potatoes from eleven cultivars and classified them by their origin: lipid degradation, sugar degradation and/or Maillard reaction not involving sulfur amino acids, sulfur amino acid degradation, methoxypyrazines, and terpenes. There are great similarities between the composition of oven-baked potatoes and potato chips, but also some notable differences (Whitfield & Last, 1991). In fried potato products flavour compounds are not only formed from the potato, but also from the frying oil and from the interaction of Maillard reaction compounds and lipids (Maga, 1994; Whitfield, 1992). Frying temperature is an important parameter as it has a great influence on the formation of pyrazines in potato chips (Maga & Sizer, 1978; Martin & Ames, 2001a). Brewer, Vega, and Perkins (1999) emphasize that odour characteristics of French fries reflect the odour characteristics of the frying oils, so that typical lipid oxidation products such as hexanal increase in the product when the same oil is used over a longer period.

Although potato flavour has received much attention, the number of papers about flavour compounds of French fries is limited. The doctoral thesis by Carlin (1983) describes 429 volatiles extracted from French fries, and two papers have been published from this thesis: one focusing on alkyloxazoles (Carlin et al., 1986) and the other on 3-(methylthio)alkanals (Carlin, Ho, Chang, Velluz, & Pickenhagen, 1990). There is however no information about odour impact of the volatiles given. Wagner and Grosch (1997) screened 48 compounds on their contribution to French fries flavour using aroma extract dilution analysis (AEDA) and gas chromatography–olfactometry of headspace samples (GCO-H). In a second paper (Wagner & Grosch, 1998) the authors evaluated the flavour profile of a model system with 21 potent flavour compounds dissolved in sunflower oil in comparison with the original extract. 2-Ethyl-3,5-dimethylpyrazine, 3-ethyl-2,5-dimethylpyrazine, 2,3-diethyl-5-methylpyrazine, 3-isobutyl-2-methoxypyrazine, 2,4-decadienal (*E,E*- and *E,Z*-), *trans*-4,5-epoxy-(*E*)-2-decenal, 4-hydroxy-2,5-dimethyl-3(2H)-furanone, methylpropanal, 2- and 3-methylbutanal, and methanethiol were identified as character impact odorants.

Starch and lipids, both important constituents of French fries, are known to interact with flavour compounds (Arvisenet, Voilley, & Cayot, 2002; Doyen, Carey, Linforth, Marin, & Taylor, 2001). Moreover, during consumption food is mixed and diluted with saliva by mastication, and starch will be degraded to some extent by amylase in saliva. The breakdown of the food matrix affects the food volatile composition (Van Ruth, Roozen, & Cozijnsen, 1995), but is often not taken into account when key flavour compounds are determined.

The aim of this study was to isolate and identify odour active compounds of French fries at mouth conditions. Mouth conditions will be created to mimic release of volatile compounds from the food to the nose epithelia, where odour is sensed, by taking into account the amount of product in relation to the mouth volume, the temperature, and by mixing the product with artificial saliva.

2. Materials and methods

2.1. Materials

French fries (10 × 10 mm, from variety Agria), obtained from Boots Frites BV (Purmerend, The Netherlands), were deep-fried in a Princess Classis household fryer (Princess Household Appliances, Middelharnis, The Netherlands) in partially hydrogenated vegetable frying oil (Remia, Den Dolder, The Netherlands, fatty acid composition: 9.7% C16:0, 5.9% C18:0, 72.3% C18:1, 11.5% C18:2, and 0.8% C18:3) for 4 minutes at 180 °C.

Artificial saliva was prepared in demineralized water according to Van Ruth et al. (1995) and consisted of K₂PO₄ (1.37 g l⁻¹), KCl (0.45 g l⁻¹), CaCl₂ · H₂O (0.44 g l⁻¹), NaCl (0.88 g l⁻¹), NaHCO₃ (5.2 g l⁻¹), porcine stomach mucine (2.16 g l⁻¹, type II M2378, Sigma, Zwijndrecht, The Netherlands) and α-amylase from *A. oryzae* (10.5 g l⁻¹, type X-A, 500,000 units, Sigma, Zwijndrecht, The Netherlands). NaN₃ (0.2 g l⁻¹) was used for preservation.

2.2. Isolation of volatile compounds

Volatiles were adsorbed on a glass tube (length 100 mm, 3.0 mm internal diameter) filled with 100 mg of Tenax TA (20/35 mesh, Alltech Nederland BV, Zwijndrecht, The Netherlands) using purge-and-trap. A sample of French fries was cut in pieces (< 0.5 cm³), 3.0 g was weighed and transferred into a glass flask (70 ml), and 4 ml of artificial saliva was added. The flask was placed in a water bath of 37 °C and a flow of purified nitrogen (50 ml min⁻¹) was passed through the sample for 30 min. The sample was mixed constantly by a magnetic stirrer. A cold trap with ethanol of -10 °C was used to prevent water vapour entering the Tenax tube.

2.3. Gas chromatographic analysis

Volatiles were desorbed onto the column using a thermal desorption unit (5 min at 245 °C) and cold trap (-120 °C/260 °C) device (Carlo Erba TDAS 5000, Inter-science BV, Breda, The Netherlands). Compounds were separated on a 60 m × 0.25 mm × 0.25 μm Supelco MDN-5S capillary column (Sigma, Zwijndrecht, The Nether-

lands) in a Carlo Erba MEGA 5300 system (Interscience BV, Breda, The Netherlands). The oven temperature was kept at 40 °C for 4 min, and then increased to 270 °C at 4.0 °C min⁻¹ with a final hold for 5 min. Helium was used as carrier gas at a constant pressure of 150 kPa and a FID detector was used at 300 °C. Retention indices of compounds were determined with a homologue series of alkanes (C5–C16) and relative areas of the compounds were calculated based on four replicates.

Identification was performed with a Varian 3400 gas chromatograph (Varian, Bergen op Zoom, The Netherlands) equipped with a thermo desorption device (Chrompack TCT injector 16200, Chrompack, Middelburg, The Netherlands), coupled with a Finnigan MAT95 mass spectrometer (Thermo Electron, Bremen, Germany). The mass spectrometer was operated in the 70 eV EI ionisation mode and scanned from mass 24 to 320 with a cycle time of 0.65 s. Identification was made by matching sample spectra against the Wiley/NIST 7th edition library and the Wageningen library, and by retention indices when available (Adams, 2001; Kondjuyan & Berdague, 1996).

2.4. Gas chromatography–olfactometry analysis

Gas chromatography–olfactometry analysis (GC–O) analysis was performed with a comparable system, column, and conditions as described in the previous paragraph. The effluent was however split 10:45:45 at the end of the column for FID detector and two sniffing ports, respectively. Humidified air ran through stainless steel tubing to the sniffing port in order to prevent drying out of assessors' nasal membranes during the 40 min sniffing experiments.

2.5. Olfactometry data acquisition

Twelve assessors, nine females and three males aged 19–22, were selected for the olfactometry experiment from thirty volunteers. The selection procedure included a questionnaire and a test for odour sensitivity. The questionnaire was used to test their creativity by describing odours and odour differences between several products including French fries. Also questions about their availability and physical condition were included. The odour sensitivity test was carried out by putting a concentration series of 2-methylbutanal in paraffin (0.08, 0.13, 0.40, 2.0, and 10 µl l⁻¹) in ascending order. The concentration range was determined in preliminary experiments. Sixteen volunteers put the concentration either in the correct order or mixed up only two consecutive concentrations. Twelve of them were selected based on the results of the questionnaire.

The detection frequency method (Van Ruth et al., 1995) was used to determine the importance of odour active compounds and a program to acquire sniffing

data was written in Pascal. Assessors recorded the beginning and end of an odour by pressing a key on a laptop computer. The number of assessors detecting an odour simultaneously is used as a measure for the importance and from this data an aromagram can be constructed. Three training sessions were carried out with the same sample as in the final experiment to make the assessors familiar with the procedure. To generate flavour descriptors, assessors were asked to give a description at the end of each odour impression. From the list of descriptors twelve groups were created based on similarity and occurrence in a panel discussion. In the olfactometry experiment assessors were forced to choose the group best describing the odour at the end of each odour impression. Blank Tenax TA tubes were used as dummy samples to determine the noise level of the panel. Retention index, mass spectrum, and odour description (Maarse, 1991; Sigma–Aldrich, 2003) were used to identify odour active compounds.

3. Results and discussion

The groups of odour descriptors resulting from the attribute generation and panel discussion are shown in Table 1. Because compounds often eluted closely after each other and assessors had to react quickly, we decided to create not more than twelve groups. For descriptors often mentioned (e.g. French fries, chocolate) a separate group was created, and from some similar descriptors a logical group name proceeded (e.g. nutty, green). For the remaining descriptors a general group name (e.g. spicy, baked flavour) was agreed on.

A total of 122 compounds identified with GC–MS are listed in Table 2. Based on the relative areas approximately 85% of the aroma compounds originated from sugar degradation and/or Maillard reaction not involving sulfur amino acids and 15% were lipid-derived. Less than 0.1% consisted of a number of sulfur compounds and one terpene (limonene). Duckham et al. (2001) compared volatiles from oven-baked potatoes of eleven cultivars, and found that the relative amount of lipid derived volatiles ranged from 22% to 69% among cultivars. For volatiles originating from sugar degradation and/or Maillard reaction not involving sulfur amino acids the range was from 25% to 77%. As the lipid content of raw potato is only 2–3 g kg⁻¹ (Duckham et al., 2001) and French fries are prepared in a large amount of oil, one might expect the amount of lipid-derived volatiles to be higher in French fries than in oven-baked potatoes. It seems however that the high heat transfer from the oil to the product is more favourable for sugar degradation and/or the Maillard reaction than for lipid degradation. Furthermore, melanoidines formed in the Maillard reaction are known to have an antioxidative effect (Morales &

Table 1
Descriptor groups used for olfactory analysis

Group	Descriptor	Descriptors from attribute generation
1	French fries	French fries, potato chips, baked potato, frying fat, frying odour
2	Baked flavour	Popcorn, cookie, baked meat, baked fish, bread
3	Potato, earthy	Raw potato, cooked potato, earthy
4	Nutty	Nutty, peanut, almond, nougat, marzipan
5	Chocolate	Chocolate, cacao
6	Sweet, flowery, fresh	Sweet, flowery, mint, anise, apple, lemon, soap, pine, melon
7	Butter, vanilla, caramel	Butter, vanilla, caramel
8	Grass, green	Vegetable, grass, plant, cucumber, carrots
9	Cheese, sweat	Cheese, sweat
10	Spicy	Spicy, mushroom, wood, garlic, onion, leather, fungus
11	Chemical, paint, glue	Chemical, paint, glue, plastic
12	Metal, burnt	Metal, burnt

Table 2
Relative peak area and retention indices (RI) of volatile compounds identified in French fries, fried for 4 min at 180 °C

Compounds by main origin	RI _{exp}	RI _{lit}	Relative peak area (%)
<i>Lipid degradation</i>			
Ethanol ^{a,b,c,e}	<500	503 ^g	3.1
2-Propanol	514	524 ^g	4.1
Methyl acetate	525	515 ^g	<0.1
2,3-Butanedione ^{a,b,c,e}	585	593 ^g	0.4
2-Butanone ^{a,c,d,e}	589	597 ^g	0.3
2-Methylfuran ^{b,c,d}	595	606 ^g	<0.1
2-Butanol ^{c,e}	601	591 ^g	<0.1
Methyl 2-propenoate	607		<0.1
Tetrahydrofuran	620	628 ^g	0.5
2-Methyl-1-propanol ^c	624	619 ^g	<0.1
Acetic acid ^{a,b,c,c}	649	660 ^g	<0.1
1-Penten-3-ol ^c	682	683 ^g	<0.1
2-Pentanone	685	687 ^g	<0.1
1-Heptene	689	692 ^g	<0.1
2-Ethylfuran ^{b,c,d}	701	704 ^g	0.5
2-Vinylfuran	721		<0.1
2,5-Dihydro-3,4-dimethylfuran ^f	732		<0.1
2-Methyl-2-butenal ^{c,d,e}	740	742 ^h	<0.1
2-Pentenal, (<i>E</i>)- ^{a,b,c,d}	753	754 ^g	<0.1
Methyl 2-butenate, (<i>E</i> -)	761		<0.1
1-Pentanol ^{a,b,e}	765	771 ^h	<0.1
Toluene ^{b,c,d,e}	766	770 ^g	0.2
Methyl 3-methylbutanoate	775	775 ^g	<0.1
2-Ethyl-5-methylfuran	775		<0.1
3-Hexanone ^{a,b,c,d,e}	783	787 ^g	<0.1
2-Methyl-3-hexanone	784	734 ^g	<0.1
2-Hexanone tentative ^{a,c,e}	787	789 ^g	<0.1
2-Propylfuran ^{c,e}	788	787 ^g	<0.1
1-Octene	790	792 ^g	<0.1
Cyclopentanone ^{c,e}	791		<0.1
Hexanal ^{a,b,c,d,e}	801	802 ^h	1.1
Butanoic acid ^{c,e}	805	779 ^g	<0.1
2-Octene, (<i>E</i> -)	805	818 ^g	<0.1
2-Octene, (<i>Z</i> -) ^b	813	808 ^g	<0.1
2-Ethyl-2-butenal, (<i>E</i>)- ^f	815		<0.1
2,5-Furandione ^f	830		<0.1
Propylcyclopentane	833		<0.1

Table 2 (continued)

Compounds by main origin	RI _{exp}	RI _{lit}	Relative peak area (%)
2-Hexenal, (<i>E</i>)- ^{a,b,c,d}	853	855 ^h	<0.1
2-Heptanone ^{a,b,c,e}	889	892 ^h	<0.1
Styrene ^{b,c,d}	893		0.1
Heptanal ^{a,b,c,d,e}	902	902 ^h	0.4
Acetylfuran ^{c,e}	910	912 ^h	<0.1
Propylcyclohexane ^{b,d}	935	939 ^g	<0.1
Butylcyclopentane	937	941 ^g	<0.1
2-Heptenal, (<i>E</i>)- ^{a,b,c,d}	959	956 ^g	0.3
5-Methylfurfural ^c	962	964 ^h	<0.1
1-Heptanol ^{a,b,c,e}	971	967 ^h	<0.1
Phenol ^c	980	971 ^g	<0.1
1-Octen-3-ol ^{a,b,c,d,e}	981	979 ^h	<0.1
Hexanoic acid ^{b,c,e}	987	970 ^g	<0.1
2-Pentylfuran ^{a,b,c,d,e}	991	993 ^g	<0.1
2,4-Heptadienal, (<i>E,Z</i>)- ^{b,c}	998		<0.1
Octanal ^{a,b,c,d}	1005	1004 ^g	~ 0.2
2,4-Heptadienal, (<i>E,E</i>)- ^{b,c,d,e}	1014	1009 ^g	<0.1
5-Ethyl-1-cyclopentene-1-carboxaldehyde	1035		<0.1
Benzyl alcohol ^{a,b,c,e}	1038		<0.1
2-Hydroxybenzaldehyde	1048	1045 ^h	<0.1
2-Octenal, (<i>E</i>)- ^{a,b,c,d,e}	1060	1060 ^g	0.2
Acetophenone ^{d,e}	1071	1065 ^h	0.1
Nonanal ^{b,c,d,e}	1107	1101 ^h	1.0
2-Nonenal, (<i>E</i>)- ^{a,b,c,d,e}	1162	1162 ^h	<0.1
Benzoic acid ^c	1172		<0.1
Decanal ^{b,c,d,e}	1207	1202 ^h	<0.1
2,4-Nonadienal, (<i>E,E</i>)- ^{a,b,c,e}	1229	1212 ^h	<0.1
2-Decenal, (<i>E</i>)- ^{c,e}	1265	1264 ^h	<0.1
2,4-Decadienal, (<i>E,Z</i>)- ^{a,b,c,e}	1298	1293 ^h	<0.1
2,4-Decadienal, (<i>E,E</i>)- ^{a,b,c,e}	1323	1317 ^h	<0.1
2-Undecenal, (<i>E</i>)- ^c	1368	1366 ^g	<0.1
1-Undecanol	1374	1370 ^h	<0.1
Total			14.6
<i>Sugar degradation and/or Maillard reaction not involving sulfur amino acids</i>			
2-methylpropanal ^{a,b,c,e}	554	552 ^g	20.3
3-Methylbutanal ^{a,b,c,d,e}	658	654 ^g	29.3
2-Methylbutanal ^{a,b,c,d,e}	669	662 ^g	31.4

Table 2 (continued)

Compounds by main origin	RI _{exp}	RI _{lit}	Relative peak area (%)
2,3-Pentanedione ^{b,c,e}	698	700 ^h	0.7
Pyrazine	732		<0.1
3-Methyl-1-butanol ^{a,b,c}	734	737 ^g	<0.1
1-Methylpyrrole ^{c,d}	736	749 ^g	<0.1
Pyridine ^{a,b,c}	746	751 ^g	<0.1
Pyrrole	748	762 ^g	0.2
4,5-Dimethyloxazole ^f	755		<0.1
2,4,6-Trimethyl-1,3,5-trioxane ^c	781	776 ^g	<0.1
2-Methylpropanoic acid ^{c,e}	797		0.8
Dihydro-2-methyl-3(2H)-furanone ^{c,e}	807		<0.1
1-Ethylpyrrole	811	820 ^g	<0.1
Methylpyrazine ^{c,e}	825	826 ^h	0.7
Furfural ^{a,b,c,d,e}	832	836 ^h	<0.1
2-Methylpyrrole ^d	843		<0.1
3-Methylbutanoic acid ^{c,e}	878	843 ^g	<0.1
2-Methylbutanoic acid ^e	888	838 ^g	0.2
2,5- and/or 2,6-dimethylpyrazine ^{c,e}	915	913 ^h	0.4
Ethylpyrazine ^c	918	916 ^h	0.1
2,3-Dimethylpyrazine ^{c,e}	921	920 ^h	0.3
Vinylpyrazine ^c	934		<0.1
Benzaldehyde ^{a,b,c,d,e}	966	960 ^h	0.1
2-Ethyl-6-methylpyrazine ^c	1000		0.2
2-Ethyl-3-methylpyrazine ^c	1004	1003 ^h	~ 0.2
2-Ethyl-5-methylpyrazine ^{c,e}	1006		~ 0.2
2-Methyl-6-vinylpyrazine ^c	1020		<0.1
Isopropenylpyrazine	1021		<0.1
2-Methyl-5-vinylpyrazine ^{c,e}	1025		<0.1
1-Methyl-2-pyrrolidone	1043	1042 ^g	<0.1
Phenylacetaldehyde ^{a,b,c,d,e}	1048	1049 ^g	0.1
3-Ethyl-2,5-dimethylpyrazine ^{c,e}	1079	1060 ^g	<0.1
2,6-Diethylpyrazine	1081		<0.1
2,3-Diethylpyrazine ^c	1082	1085 ^h	<0.1
2-Ethyl-3,5-dimethylpyrazine	1085		<0.1
5-Ethyl-2,3-dimethylpyrazine	1088		<0.1
2,5-Diethylpyrazine	1094		<0.1
Dimethylvinylpyrazine isomer ^{c,e,f}	1100		<0.1
2,3-Dihydroindole	1120		<0.1
Isobutylmethylpyrazine isomer ^{c,e}	1139	1137 ^h	<0.1
2,3-Diethyl-5-methylpyrazine ^{c,e}	1153		<0.1
3,5-Diethyl-2-methylpyrazine ^{c,e}	1157		<0.1
Methylpropenylpyrazine isomer ^c	1179		<0.1
Dimethylisobutylpyrazine isomer ^c	1201		<0.1

Table 2 (continued)

Compounds by main origin	RI _{exp}	RI _{lit}	Relative peak area (%)
Isopentylmethylpyrazine isomer ^c	1254		<0.1
Isopentylmethylpyrazine isomer ^c	1316		<0.1
Total			85.4
<i>Sulfur compounds</i>			
Dimethyl disulfide ^{b,c,d,e}	743	744 ^g	<0.1
2-Methylthiophene	770	779 ^g	<0.1
3-Methylthiophene	779	786 ^g	<0.1
Dimethyl trisulfide ^{b,c,d,e}	975	950 ^g	<0.1
Dimethyl tetrasulfide ^{b,c,e}	1241	1215 ^g	<0.1
Total			<0.1
<i>Terpenes</i>			
Limonene ^{a,b,c,d,e}	1034	1031 ^g	<0.1
Grand total			100

^a Found in raw potatoes (Adams, 2001; Berdague, Denoyer, Le Quéré, & Semon, 1991).

^b Found in boiled potatoes (Adams, 2001; Berdague et al., 1991; Brewer et al., 1999; Buttery et al., 1973; Carlin, 1983).

^c Found in oven-baked potatoes (Adams, 2001; Carlin et al., 1986, 1990; Coleman & Ho, 1980; Coleman et al., 1981; Doyen et al., 2001; Duckham et al., 2001, 2002).

^d Found in microwave baked potatoes (Oruna-Concha et al., 2002a, 2002b).

^e Found in French fries (Maga, 1994; Carlin et al., 1986; Wagner & Grosch, 1997).

^f Tentative identification.

^g According to Adams (2001).

^h According to Kondjoyan and Berdague (1996).

Jimenez-Perez, 2001). This may have an effect on the amount of lipid-derived compounds, because the majority of these compounds is formed through oxidation. In this study fresh frying oil was used for all experiments. The composition of volatiles may however change as the frying oil gets older, because the odour characteristics of French fries reflect the odour characteristics of the oils in which they are fried (Brewer et al., 1999).

Many of the identified compounds have been described previously as volatiles from French fries or (processed) potato (see Table 2). The three main contributors of sugar degradation and/or Maillard reaction were 3-methylbutanal, 2-methylbutanal, and 2-methylpropanal, Strecker aldehydes of respectively isoleucine, leucine, and valine (Whitfield, 1992), and they accounted for 81% of the total yield of volatiles. Other contributors were phenylacetaldehyde, the Strecker aldehyde of phenylalanine (Whitfield, 1992), 2,3-pentanedione, and many heterocyclic nitrogen containing compounds of which pyrazines were most abundant. A total of 26 pyrazines were found of which eleven have not been reported as volatiles from French fries, and six not from potato previously. Pyrazines are formed mainly from glutamine and asparagine, because these amino acids are present in potato in large quantities (Martin & Ames,

2001b; Oruna-Concha et al., 2001), and pyrazines are considered to be key components of baked potato flavour (Duckham et al., 2002). Benzaldehyde was not put in the list of lipid-derived products as was done previously (Duckham et al., 2001; Oruna-Concha et al., 2002a), because it is formed from phenylacetaldehyde (Martin & Ames, 2001b). The formation of 2- and 3-methylbutanoic acid and 2-methylpropanoic acid can be explained by further oxidation of their corresponding aldehydes, the three main contributors. Lipid degradation resulted in the formation of several aldehydes, ketones, alcohols, hydrocarbons, acids, esters, and furans, of which many are secondary oxidation products. The highest relative areas were obtained by ethanol, 2-propanol, nonanal, and hexanal. Nonanal is formed from oleic acid, which is the main fatty acid of the frying oil used, and hexanal is a typical oxidation product of linoleic acid (Brewer et al., 1999; Whitfield, 1992). Ethanol and 2-propanol are probably formed after several degradation steps, induced by the high frying temperature. The three sulfides and two thiophenes that were found in this study were formed from sulfur containing amino acids. As these sulphides are formed in the Maillard reaction they have been found in boiled, baked, and fried potato, but not in raw potato. 2- and 3-Methylthiophene were suggested to be formed from the interaction between fatty aldehydes and cysteine (Macku & Shibamoto, 1991). 2-Pentanone and 1-methyl-2-pyrrolidinone have not been found in potato, but they have been reported as volatiles from cured ham (Berdague et al., 1991). Methional and methanethiol, both appointed as potent odorants of French fries flavour (Wagner & Grosch, 1997), were not detected.

In Fig. 1 the chromatogram from the FID-detector and the aromagram resulting from the olfactometry experiments is shown, and identification of the 41 peaks is explained in Table 3. Not all aromagram peaks could be identified conclusively, because sometimes compounds having similar odours eluted closely after each other. Compounds with the highest detection frequency contribute most to French fries flavour. In this way 2-methylbutanal and/or 3-methylbutanal, hexanal, 2,3-dimethylpyrazine, 2-methylpropanal, 2,3-butanedione, pyridine, heptanal, 2,5-dimethylpyrazine and/or 2,6-dimethylpyrazine and/or ethylpyrazine, dimethyl trisulfide, octanal, phenylacetaldehyde, 2,5-diethylpyrazine, (*E*)-2-nonenal, 2-methylbutanoic acid and/or 3-methylbutanoic acid, (*E,Z*)-2,4-heptadienal, (*E*)-2-octenal, 5-ethyl-2,3-dimethylpyrazine and/or 2-ethyl-3,5-dimethylpyrazine, nonanal, and tentatively 2-methylpyrrole were found to be the most important odour active compounds of French fries.

2-Methylpropanal, 3-methylbutanal and 2-methylbutanal were perceived by almost all assessors. It was difficult for the assessors to notice a difference between

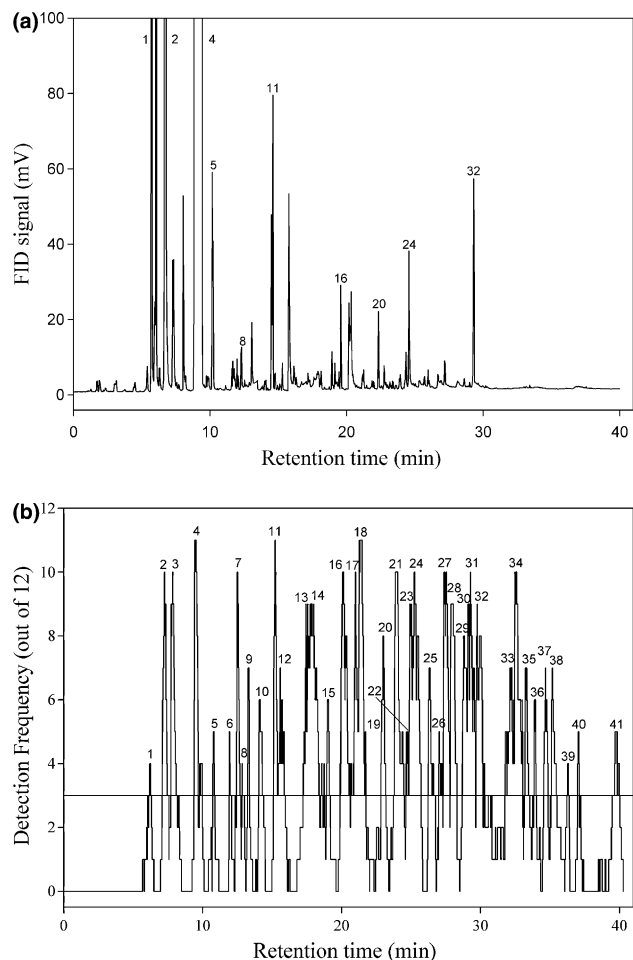


Fig. 1. Aromagram of dynamic headspace samples. FID response (a) and detection frequency of assessors at sniffing port (b). Horizontal line in (b) represents noise level.

3- and 2-methylbutanal, resulting in a large peak with a small shoulder. The odour is usually referred to as chocolate or malty, but some assessors described it as sweaty, which corresponds to what Berdague et al. (1991) found. Pyrazines were responsible for eleven peaks in the aromagram and resulted in the dominant fried potato note. C2- and C4-substituted pyrazines eluted closely after each other making it difficult to identify the one responsible for peak 13 and peaks 29–31, respectively. 2,3-Butanedione and to a lesser extent 2,3-pentanedione contributed to the caramel or buttery note. Fatty aldehydes, of which hexanal, heptanal, octanal, (*E*)-2-nonenal, and nonanal were the most important representatives, were perceived as green odours. These compounds are however known to cause a rancid off-flavour (Brewer et al., 1999; Whitfield, 1992). We confirm that alkadienals, such as (*E,E*)-2,4-decadienal, (*E,E*)-2,4-nonadienal, and (*E,Z*)-2,4-heptadienal, contribute to the deep-fried note (Wagner & Grosch, 1997). Dimethyl trisulfide, phenylacetaldehyde, and possibly 2-methylpyrrole cause a

Table 3
Detection frequency and odour description of odour active compounds from French fries

Peak no.	Retention time on GC–O	RI ^a	Detection frequency	Odour group ^b	Compound
1	6.19	<500	4	5, 6, 11	Ethanol
2	7.25	550	10	7, 5, 9, 11	2-Methylpropanal
3	7.83	585	10	7	2,3-Butanedione
4	9.44	660	11	11, 9, 5, 7	3-Methylbutanal and 2-methylbutanal
5	10.76	700	5	7	2,3-Pentaandion
6	11.92	730	5	6	2,5-Dihydro-3,4-dimethylfuran tentative
7	12.51	745	10	11	Pyridine
8	12.84	755	4	11, 6, 1	Pyrrrole
9	13.26	760	7	6	Methyl 2-butenate, (<i>E</i>)-
10	14.07	775	6	11, 9	2-Methylpropanoic acid
11	15.21	800	11	6, 8	Hexanal
12	15.57	810	7	1, 10	1-Ethylpyrrole tentative
13	17.42	845	9	9, 3	2-Methylpyrrole tentative
14	17.75	855	9	11, 9	3-Methylbutanoic acid and/or 2-methylbutanoic acid
15	18.97	880	6	3	Unknown
16	20.07	900	10	8, 3, 6	Heptanal
17	21.00	920	10	3, 1, 12	2,5-Dimethylpyrazine, 2,6-dimethylpyrazine and/or ethylpyrazine
18	21.30	925	11	2, 4	2,3-Dimethylpyrazine
19	21.70	935	5	1, 2	Vinylpyrazine
20	22.97	960	8	3	2-Heptenal, (<i>E</i>)-
21	23.87	980	10	9, 10, 2, 12	Dimethyl trisulfide
22	24.67	995	5	6	2-Pentylfuran
23	24.90	1000	9	6, 1	2,4-Heptadienal, (<i>E,Z</i>)-
24	25.23	1005	10	6, 1	Octanal
25	26.29	1025	7	2, 12, 4	2-Methyl-5-vinylpyrazine
26	27.01	1045	5	3, 11	1-Methyl-2-pyrrolidinone tentative
27	27.36	1050	10	3, 12, 6, 8	Phenylacetaldehyde
28	27.87	1060	9	8, 11, 10, 7	2-Octenal, (<i>E</i>)-
29	28.67	1080	8	3, 11, 2, 10	3-Ethyl-2,5-dimethylpyrazine, 2,3-diethylpyrazine and/or 2,6-diethylpyrazine
30	29.09	1090	9	6, 2, 10	5-Ethyl-2,3-dimethylpyrazine and/or 2-ethyl-3,5-dimethylpyrazine
31	29.27	1095	10	6, 3, 12, 11	2,5-Diethylpyrazine
32	29.76	1105	9	1, 8	Nonanal
33	32.11	1155	7	3, 6, 9	3,5-Diethyl-2-methylpyrazine and/or 2,3-diethyl-5-methylpyrazine
34	32.48	1170	10	8	2-Nonenal, (<i>E</i>)-
35	33.22	1180	7	2, 4	Pyrazine, methylpropenyl isomer
36	33.89	1200	6	8, 6, 3, 12	Pyrazine, dimethylisobutyl isomer
37	34.67	1215	7	8	Decanal
38	35.16	1230	7	2, 1, 4, 11	2,4-Nonadienal, (<i>E,E</i>)-
39	36.26	1255	4	8, 2, 3	Pyrazine, isopentylmethyl isomer
40	37.02	1275	5	6, 7, 2, 12	2-Decenal, (<i>E</i>)-
41	39.68	1330	5	1	2,4-Decadienal, (<i>E,E</i>)-

^a Retention index estimated by 30–40 s delay between FID-signal and flavour perception.

^b See Table 1.

spicy note. Based on retention index and mass spectrum peak 13 could be 2-methylpyrrole, however no information about the odour description could be found in literature. Although the chemical odour from pyridine, and sweaty odour from 3-methylbutanoic acid and 2-methylbutanoic acid were clearly noticed by the panel, they do not seem to give a distinct note, but may influence the perceived aroma as a whole. 2-Methylbutanoic acid and 3-methylbutanoic acid had a very broad peak and that is the reason why the re-

tention index is higher than the literature value in Table 2. As these compounds were dominating the mass spectra around peak 15, it was not possible to identify the compound responsible for the potato or earthy odour.

Although the potato variety used was the same, the results were different from what Wagner and Grosch (1997) found. They did not find chemical or sweaty odours such as pyridine and 3-methylbutanoic acid, and that we did not find methional and methanethiol. This

can be explained by using different methods for sample preparation and extraction. Other factors such as storage conditions of the potatoes (Duckham et al., 2002), the type of frying oil (Brewer et al., 1999), and the frying temperature (Maga & Sizer, 1978; Martin & Ames, 2001b) may play a role as well.

4. Conclusion

Isolation of French fries flavour by purge-and-trap using conditions during consumption yielded 122 identified volatiles of which 85% of the relative area originated from sugar degradation and/or Maillard reaction. 2-Methylpropanal, 2-methylbutanal, 3-methylbutanal, and 26 pyrazines were the main representatives. 15% of the volatiles were lipid-derived and ethanol, 2-propanol, hexanal, and nonanal showed the highest relative areas of this group. About 50 odour active compounds were, due to coelution, responsible for 41 odours perceived by the panel. The compounds with the highest detection frequencies caused a strong malty note and fried potato note, combined with caramel/buttery, green, spicy, and deep-fried notes. Also chemical and sweaty odours were observed.

References

- Adams, R. P. (2001). *Identification of essential oil components by gas chromatography/quadrupole mass spectroscopy*. Carol Stream, IL: Allured.
- Arvisenet, G., Voilley, A., & Cayot, N. (2002). Retention of aroma compounds in starch matrices: Competitions between aroma compounds toward amylose and amylopectin. *Journal of Agricultural and Food Chemistry*, *50*, 7345–7349.
- Berdague, J.-L., Denoyer, C., Le Quéré, J.-L., & Semon, E. (1991). Volatile components of dry-cured ham. *Journal of Agricultural and Food Chemistry*, *39*, 1257–1261.
- Brewer, M. S., Vega, J. D., & Perkins, E. G. (1999). Volatile compounds and sensory characteristics of frying fats. *Journal of Food Lipids*, *6*, 47–61.
- Buttery, R., Guadagni, D. G., & Ling, L. C. (1973). Volatile components of baked potatoes. *Journal of the Science of Food and Agriculture*, *24*, 1125–1131.
- Carlin, J. T. (1983). *French fry flavor project*. New Brunswick, NJ: Rutgers University.
- Carlin, J. T., Ho, C.-T., Chang, S. S., Velluz, A., & Pickenhagen, W. (1990). Analysis of French fried potato flavor: Identification of 3-(methylthio)alkanal. *Lebensmittel Wissenschaft und Technologie*, *23*, 276.
- Carlin, J. T., Jin, Q. Z., Huang, T.-C., Ho, C.-T., & Chang, S. S. (1986). Identification of alkyloxazoles in the volatile compounds from French-fried potatoes. *Journal of Agricultural and Food Chemistry*, *34*, 621–623.
- Coleman, E. C., & Ho, C.-T. (1980). Chemistry of baked potato flavor. I. Pyrazines and thiazoles identified in the volatile flavor of baked potato. *Journal of Agricultural and Food Chemistry*, *28*, 66–68.
- Coleman, E. C., Ho, C.-T., & Chang, S. S. (1981). Isolation and identification of volatile compounds from baked potatoes. *Journal of Agricultural and Food Chemistry*, *29*, 42–48.
- Doyen, K., Carey, M., Linforth, R. S. T., Marin, M., & Taylor, A. J. (2001). Volatile release from an emulsion: Headspace and in-mouth studies. *Journal of Agricultural and Food Chemistry*, *49*, 804–810.
- Duckham, S. C., Dodson, A. T., Bakker, J., & Ames, J. M. (2001). Volatile flavour components of baked potato flesh. A comparison of eleven potato cultivars. *Nahrung*, *45*(5), 317–323.
- Duckham, S. C., Dodson, A. T., Bakker, J., & Ames, J. M. (2002). Effect of cultivar and storage time on the volatile flavor components of baked potato. *Journal of Agricultural and Food Chemistry*, *50*, 5640–5648.
- Ho, C.-T., & Coleman, E. C. (1980). Chemistry of baked potato flour: Further identification of heterocyclic compounds in the volatile flavor of baked potato. *Critical Reviews in Food Science and Nutrition*, *45*, 1094–1095.
- Josephson, D. B., & Lindsay, R. C. (1987). c4-Heptenal: An influential volatile compound in boiled potato flavor. *Journal of Food Science*, *52*(2), 328–331.
- Kondjoyan, N., & Berdague, J.-L. (1996). *A compilation of relative retention indices for the analysis of aromatic compounds*. Dijon: Laboratoire Flaveur INRA.
- Maarse, H. (1991). *Volatile compounds in food and beverages*. New York: Marcel Dekker Inc..
- Macku, C., & Shibamoto, T. (1991). Volatile sulfur-containing compounds generated from the thermal interaction of corn oil and cysteine. *Journal of Agricultural and Food Chemistry*, *39*, 1987–1989.
- Maga, J. A. (1994). Potato flavor. *Food Reviews International*, *10*(1), 1–48.
- Maga, J. A., & Sizer, C. E. (1978). Flavour preferences for potato chips as influenced by time and temperature of chipping and total pyrazine concentration. *Lebensmittel Wissenschaft und Technologie*, *11*, 181–182.
- Martin, F. L., & Ames, J. M. (2001a). Comparison of flavor compounds of potato chips fried in palmolein and silicone fluid. *Journal of the American Oil Chemists Society*, *78*(8), 863–866.
- Martin, F. L., & Ames, J. M. (2001b). Formation of strecker aldehydes and pyrazines in a fried potato model system. *Journal of Agricultural and Food Chemistry*, *49*, 3885–3892.
- Morales, F. J., & Jimenez-Perez, S. (2001). Free radical scavenging capacity of Maillard reaction products as related to colour and fluorescence. *Food Chemistry*, *72*, 119–125.
- Nursten, H. E., & Sheen, M. R. (1974). Volatile flavour components of cooked potato. *Journal of the Science of Food and Agriculture*, *25*, 643–663.
- Oruna-Concha, M. J., Bakker, J., & Ames, J. M. (2002a). Comparison of the volatile components of eight cultivars of potato after microwave baking. *Lebensmittel Wissenschaft und Technologie*, *35*, 80–86.
- Oruna-Concha, M. J., Bakker, J., & Ames, J. M. (2002b). Comparison of the volatile components of two cultivars of potato cooked by boiling, conventional baking and microwave baking. *Journal of the Science of Food and Agriculture*, *82*, 1080–1087.
- Oruna-Concha, M. J., Duckham, S. C., & Ames, J. M. (2001). Comparison of volatile compounds isolated from the skin and flesh of four potato cultivars after baking. *Journal of Agricultural and Food Chemistry*, *49*, 2414–2421.
- Petersen, M. A., Poll, L., & Larsen, L. M. (1998). Comparison of volatiles in raw and boiled potatoes using a mild extraction technique combined with GC odour profiling and GC-MS. *Food Chemistry*, *61*(4), 461–466.
- Sigma-Aldrich, 2003. *Flavors and fragrances. International Edition*, Milwaukee, USA.
- Van Ruth, S. M., Roozen, J. P., & Cozijnsen, J. L. (1995). Changes in flavour release from rehydrated diced bell peppers (*Capsicum annuum*) by artificial saliva components in three mouth model systems. *Journal of the Science of Food and Agriculture*, *67*, 189–196.

- Wagner, R. K., & Grosch, W. (1997). Evaluation of potent odorants of French fries. *Lebensmittel Wissenschaft und Technologie*, 30(2), 164–169.
- Wagner, R. K., & Grosch, W. (1998). Key odorants of french fries. *Journal of the American Oil Chemists Society*, 75(10), 1385–1392.
- Whitfield, F. B. (1992). Volatiles from interactions of Maillard reactions and lipids. *Critical Reviews in Food Science and Nutrition*, 31(1/2), 1–58.
- Whitfield, F. B., & Last, J. H. (1991). Tuber vegetables. In H. Maarse (Ed.), *Volatile compounds in foods and beverages* (pp. 222–231). New York: Marcel Dekker.