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Real-Time Flavor Release from French Fries Using Atmospheric Pressure Chemical Ionization−Mass Spectrometry

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Flavor release from French fries was measured with atmospheric pressure chemical ionization−mass spectrometry (APCI-MS) using both assessors (in vivo) and a mouth model system (in vitro). Several volatiles measured with APCI were identified with MS-MS. The effect of frying time, salt addition, and an alternative process using superheated steam was determined on I_{max} (maximum intensity of compounds) and on t_{max} (time of maximum intensity). In vitro a "chewing" frequency of 0.60 Hz caused an increased t_{max} for low molecular weight compounds compared to the other frequencies tested. Above 0.93 Hz further increase in the frequency did not affect t_{max}. Trends observed with in vivo experiments could be verified with in vitro experiments. I_{max} correlated well with frying time. Addition of salt resulted in a decreased t_{max}, suggesting a salting-out effect. The alternative process caused a layer of oil on the surface, and this resulted in a higher t_{max}, but no effect on I_{max} was found. This phenomenon may be critical for the sensory quality and would not have been observed with static volatile measurements, demonstrating the value of flavor release measurements.

KEYWORDS: Atmospheric pressure chemical ionization−mass spectrometry; flavor release; mouth model system; French fries

INTRODUCTION

Since the introduction of fast-food restaurants, French fries have become a popular food all over the world. Apart from texture, flavor is one of the most important quality aspects. The flavor of potato has received much attention by researchers, and more than 500 volatiles have been identified (*1*). The flavor composition changes considerably with different processing methods (*2*). Enzymatic degradation of unsaturated fatty acids by lipoxygenase is important for raw potato flavor. Enzymes are inactivated during cooking, and methional, formed in the Maillard reaction, is a key compound of boiled potato flavor (*3*). In oven-baked potatoes, where higher temperatures are applied, an increased number of Maillard reaction products have been found (*4*). Microwave-baked potatoes have a composition of volatile compounds between those of boiled and oven-baked potatoes (*5*). The amount of heterocyclic compounds from the Maillard reaction is the highest in deep-fried potato products due to the high heat transition (*6*). Additionally, frying in oil yields flavor compounds derived from lipid degradation and from interactions between lipids and Maillard reaction products (*7*).

Several authors have studied the flavor of French fries. A dissertation on this subject (*8*) focused on the identification of volatiles, but no information on the odor impact was given. Wagner and Grosch (*9*, *10*) identified 48 odorants of French fries by application of both aroma extraction dilution analysis and GC−olfactometry (GC-O). Van Loon et al. (*11*) used a purge and trap method mimicking mouth conditions to extract volatile compounds. GC-FID, GC-MS, and GC-O were used to select and identify odor active compounds. On the basis of relative peak areas, 85% were found to originate from sugar degradation and/or Maillard reaction, whereas 15% were lipid-derived. With GC-O 50 odor active compounds were found, of which 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, and (*E*)-2-nonenal were found to be the most important compounds.

With the introduction of new mass spectrometric methods it is possible to sample volatile compounds in the nose space of assessors during eating (*12*, *13*). Atmospheric pressure chemical ionization−mass spectrometry (APCI-MS) has been used both on model systems (*14*) and on food systems (*15*). This method has been proven to be suitable to measure flavor release in real time, and good correlations with sensory perception were found. Mouth model systems have been designed to mimic eating dynamics in vitro (*16*). In general, the food is blended and volatiles are trapped on an adsorbent. Although these devices usually do not include inhaling and swallowing, they have the

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advantage of causing less variation than assessors (*17*). Furthermore, mouth model systems allow examination of individual oral physiological factors on flavor release.

Although the composition of French fry flavor has received some attention by researchers and key aroma compounds have been identified, to our knowledge no attention has been paid to flavor release. Flavor release could provide information about flavor perception during the consumption of French fries. Furthermore, no previous study was undertaken to show the effects of process conditions on French fry flavor. Recently, a new energy-efficient process for the production of French fries was proposed (*18*). Therefore, the aim of the present study was to investigate how this new process affects in vivo and in vitro flavor release from French fries in real time. A novel approach was chosen to identify aroma compounds from real food systems by APCI-MS-MS. Also, the effects of frying time and salt addition on flavor release were evaluated.

MATERIALS AND METHODS

Materials. In this study two different processes to prepare French fries were compared: the conventional process and a new, energy-efficient process, in which superheated steam was used (*18*). For both processes potatoes of the variety Agria were used, and potato strips were cut to a size of 10 × 10 mm. Conventional par-fried, frozen French fries were kindly provided by Boots Frites BV (Purmerend, The Netherlands). In the alternative process potato strips were first blanched according to the conventional process. Subsequently, potato strips were treated with superheated steam for 25 min at 0.47 bar, with a steam temperature of 110 °C and a steam flow of 115 kg h^{-1}. Cooling and freezing were performed according to the conventional process. Previous research showed that the French fries of the alternative process had a 30% reduction in oil content after finish-frying (in oil) compared to the conventional product (*18*).

Partially hydrogenated vegetable oil (Remia, Den Dolder, The Netherlands) was used for finish-frying. French fries of ~6 cm length were selected and fried individually in a Princess Classic household fryer (Princess Household Appliances, Middelharnis, The Netherlands) at 180 °C.

Artificial saliva was prepared in demineralized water according to the method of Van Ruth et al. (*19*) and consisted of K_2PO_4 (1.37 g L^{-1}), KCl (0.45 g L^{-1}), $CaCl_2 \cdot H_2O$ (0.44 g L^{-1}), NaCl (0.88 g L^{-1}), $NaHCO_3$ (5.2 g L^{-1}), porcine stomach mucine (2.16 g L^{-1}, type II M2378, Sigma, Steinheim, Germany), and α-amylase from *Aspergillus oryzae* (10.5 g L^{-1}, type X-A, 500 000 units, Sigma). NaN_3 (0.08 g L^{-1}) was used for preservation.

General Setup. After finish-frying, a French fry was allowed to cool for 3 min to reach an acceptable temperature for consumption. This was timed with a stopwatch. Next, flavor release was measured by APCI-MS, both in exhaled breath of assessors (in vivo) and in a mouth model system (in vitro). The maximum intensity (I_{max}) and time of maximum intensity (t_{max}) of released flavor compounds were determined to evaluate the effect of the alternative process for the production of French fries, the effect of frying time (2, 4, 6, and 8 min), and the effect of salt addition (0.1 g per French fry). The amount of salt was based on preliminary experiments. All salt particles automatically stuck to the oily surface of the French fries.

Identification of Released Flavor Compounds. Flavor compounds were measured on-line by an atmospheric pressure chemical ionization gas-phase analyzer attached to a VG Quattro II mass spectrometer (Micromass UK Ltd., Manchester, U.K.) (*13*). Compounds were ionized by a 3.0 kV discharge (source and probe temperatures were 80 °C) and scanned for *m*/*z* 40−250. *m*/*z* values of observed compounds were selected and fragmented with argon for identification. In further experiments compounds were monitored in selected ion mode with 0.08 s of dwell time on each ion, resulting in a cycle time of 0.88 s.

In Vivo Flavor Release in Exhaled Breath. Flavor release was measured in exhaled breath of three experienced assessors in triplicate. Assessors breathed in and out through the nose. One nostril was placed over a plastic tip that was connected to a transfer line (0.53 mm i.d., heated to 100 °C), from which continuously 80 mL min^{-1} of air was sampled directly into the APCI-MS. A strict protocol was followed during the experiments. After putting one French fry in the mouth, assessors immediately started chewing at a rate of about one chewing movement per second. The sample was swallowed after 30 s, and chewing movements were continued until 60 s (*14*). Between samples the mouth was rinsed with water. Blank experiments were recorded with water following the same protocol. Acetone, present in human breath, was measured at *m*/*z* 58.8 (19 V) as an indicator for the breathing pattern (*14*). The background noise was negligible to the signal.

Table 1. Compounds Detected with APCI-MS and Identified by MS-MS

m/*z*	cone (V)	compound	lit.[c]
69	20	unknown (fragmentation pattern: *m*/*z* 69, 100%; *m*/*z* 41, 65%; *m*/*z* 39, 5%; *m*/*z* 29, 9%)[a,b]	
73	20	methylpropanal[a,b]	*1*, *4*, *9*
75	23	unknown (fragmentation pattern: *m*/*z* 75, 100%; *m*/*z* 57, 6%; *m*/*z* 43, 23%; *m*/*z* 29, 8%; *m*/*z* 15, 5%)[a]	
87	16	2- and/or 3-methylbutanal[a,b]	*1*, *4*, *5*, *9*
91	10	unknown (fragmentation pattern: *m*/*z* 91, 100%; *m*/*z* 73, 111%; *m*/*z* 58, 9%; *m*/*z* 55, 44%; *m*/*z* 45, 22%; *m*/*z* 43, 10%; *m*/*z* 31, 14%; *m*/*z* 29, 12%)[a]	
95	20	methylpyrazine[a,b]	*1*, *4*, *5*
109	30	C2-pyrazine (dimethyl-, ethyl-)[a,b]	*1*, *4*, *5*
113	20	2-heptenal[a]	*1*, *3*, *5*
123	37	C3-pyrazine (ethylmethyl-)[a]	*1*, *4*, *5*
137	37	C4-pyrazine (ethyldimethyl-, diethyl-)[a]	*1*, *5*, *9*
153	21	2,4-decadienal[a]	*1*, *3*, *5*, *9*

[a] Detected in vitro. [b] Detected in vivo. [c] Previously found in potato or potato products.

In Vitro Flavor Release in the Mouth Model System. Dynamic headspace measurements were carried out in triplicate with a mouth model system developed by Van Ruth et al. (*20*). The mouth model consists of a double-wall glass housing with an inner volume comparable to the human mouth (70 mL) in which a plunger moves up and down and rotates simultaneously to simulate chewing. Water of 37 °C is pumped through the double wall. One French fry was put in the mouth model system, and 3.5 mL of artificial saliva was added directly. The volume of artificial saliva was determined in a preliminary experiment by having the panelists chew a French fry for 30 s and measure the weight gain. Immediately after saliva addition, the mouth model system was closed and "chewing" and sampling started. Five chewing frequencies of the mouth model system (0.60, 0.93, 1.27, 1.60, and 1.93 Hz) were tested for optimization. An air flow of 80 mL min^{-1} was sampled directly into the APCI-MS. Flavor release was monitored for 5 min after chewing started.

Statistical Analysis. SPSS 10.0.7 was used for statistical evaluation of the data. Linear regression was used for the effect of frying time and chewing frequency. MANOVA with General Linear Model followed by a post hoc test according to Tukey was used to calculate the effect of salt addition and the alternative process. A significance level of $\alpha = 0.05$ was used throughout the study.

RESULTS AND DISCUSSION

Identification of Detected Compounds. Release of a total of 11 compounds was observed in vitro, 5 of which were also found in vivo (**Table 1**). A similar observation was reported by Deibler et al. (*17*). Only part of the breath was sampled with in vivo experiments, whereas all volatiles that were released in vitro entered the APCI-MS. Therefore, in vivo the signal did not reach the detection limit for some compounds. Identification of compounds detected during flavor release is a difficult task, because it puts high demands on MS equipment. The technique

Table 2. t_{max} (Seconds) in Vitro of Compounds Released from French Fries at Different "Chewing" Frequencies

	chewing frequency				
m/z	0.60 Hz	0.93 Hz	1.26 Hz	1.60 Hz	1.93 Hz
69	63 (8)[a]	33 (8)	28 (3)	28 (6)	34 (4)
73	55 (8)	19 (7)	29 (7)	25 (3)	30 (9)
75	69 (1)	34 (10)	32 (15)	29 (4)	39 (9)
87	62 (11)	27 (14)	29 (8)	24 (2)	30 (11)
91	56 (25)	24 (8)	29 (8)	28 (4)	34 (5)
95	107 (8)	60 (18)	63 (8)	73 (13)	73 (30)
109	229 (35)	204 (38)	242 (15)	198 (104)	248 (39)
123	232 (37)	204 (38)	242 (15)	198 (104)	248 (39)
137	270 (28)	249 (52)	275 (44)	262 (37)	292 (10)
153	299 (2)	292 (7)	242 (54)	262 (12)	251 (43)

[a] Standard deviations are given in parentheses.

has to be fast and sensitive, it has to be capable of handling air and water, and it must allow the simultaneous detection of compounds. Techniques involving ionization based on proton transfer (APCI, PTR) followed by MS can overcome these problems (*21*). Fragmentation of the compounds is generally necessary for conclusive identification. However, these soft ionization techniques mainly produce single ions from compounds. In the present study selected ions were fragmented with argon and analyzed in a second MS. This elegant, direct method made unequivocal identification of several compounds possible. In accordance with the literature (*4*), the observed compounds originated from either the Maillard reaction or lipid degradation. The highest signal was obtained for 2- and/or 3-methylbutanal, which corresponds to previous findings (*11*). Methylpropanal, 3-methylbutanal, and 2-methylbutanal are Strecker aldehydes from valine, leucine, and isoleucine, respectively (*7*), and pyrazines are known Maillard reaction products as well (*6*, *22*). 2(*E*)-Heptenal and 2(*E*),4(*E*)-decadienal are formed from autoxidation of linoleic acid (*23*, *24*). *m/z* values of 69, 75, and 91 could not be identified. As the fragmentation pattern of *m/z* 87 showed a high peak of *m/z* 69 and similar fragments were found for both *m/z* values, it is possible that *m/z* 69 is a fragment of 2- and/or 3-methylbutanal. For the pyrazines it was not possible to make a conclusive identification. *m/z* 137, for example, could be 2,3-diethylpyrazine, 2-ethyl-3,5-dimethylpyrazine, or another isomer. A mixture of two or more isomers is possible as well.

Effect of Chewing Frequency on t_{max} for in Vitro Experiments. To optimize the procedure for in vitro measurements, t_{max} was determined for a number of chewing frequencies (**Table 2**). Lower molecular weight compounds (*m/z* 69−95) reached t_{max} at ∼60 s, whereas higher molecular weight compounds (*m/z* 109−153) reached t_{max} only after 3−5 min. With MANOVA it was shown that the lowest frequency (0.60 Hz) caused the lower molecular weight compounds to release significantly more slowly than the other frequencies. From 0.93 Hz t_{max} did not further decrease upon a higher frequency. No significant effect of chewing frequency on the t_{max} of *m/z* values 109, 123, 137, and 153 was found. In other words, the t_{max} of fast-releasing compounds was affected by a low chewing frequency, whereas slow-releasing compounds were not additionally slowed. For in vitro experiments a chewing frequency of 1.60 Hz was chosen, because it did not influence the release of any of the selected compounds. Furthermore, it was considered to be a normal chewing rate for humans.

Effect of Frying Time on I_{max} and t_{max}. Average aroma release curves of *m/z* 87 in vitro and in vivo after different frying times are shown as an example in **Figure 1**, panels **A** and **B**,

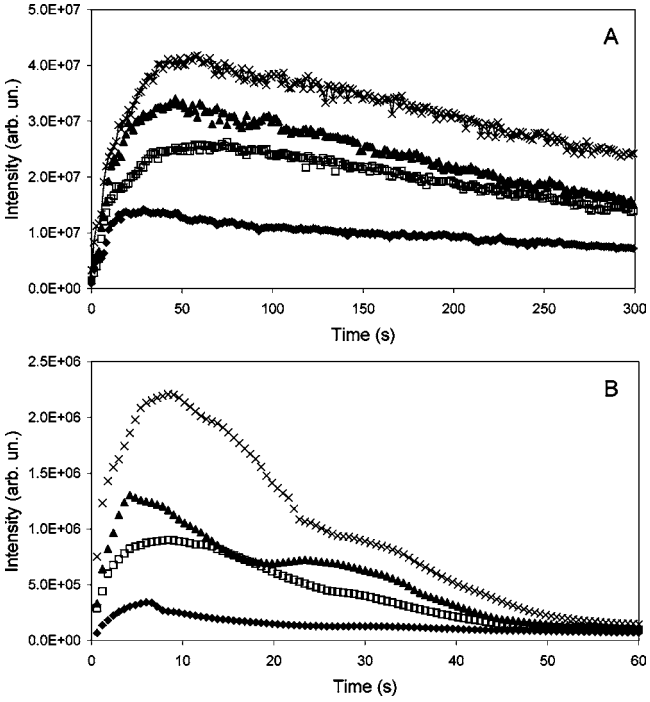

Figure 1. Example of a set of typical aroma release curves in vitro (**A**) and in vivo (**B**) of *m/z* 87 from French fries finish-fried for 2 (◆), 4 (□), 6 (▲), and 8 min (×) at 180 °C.

respectively. I_{max} and t_{max} values of all compounds are listed in **Table 3**. For all compounds I_{max} increased linearly with frying time. As frying proceeds, surface moisture evaporates, and the temperature gradually increases. A high temperature and reduced moisture content are favorable conditions for the Maillard reaction (*25*). This is in accordance with the observation that the color became darker as the frying time increased. *m/z* 113 (2-heptenal) and *m/z* 153 (2,4-decadienal) increased to a lesser extent than the other compounds, because these compounds originated from lipid oxidation. The highest I_{max} value was reached for *m/z* 87 (2- and/or 3-methylbutanal). Martin and Ames (*6*) reported high concentrations of 2- and 3-methylbutanal in fried potato model systems compared to pyrazines. I_{max} was higher in vitro than in vivo, because, as already mentioned, the amount of sample that entered the APCI-MS from the mouth model system was higher. The correlation coefficient of linear regression between I_{max} and frying time was higher in vitro (0.60−0.85) than in vivo (0.25−0.55). This may be explained by differences among assessors such as geometry of the mouth, saliva production, and chewing behavior.

With MANOVA no significant effect of frying time on t_{max} was found (**Table 3**). There was, however, a distinct difference between in vivo and in vitro experiments. For in vivo experiments all compounds had a t_{max} of <10 s, whereas in vitro the fastest releasing compounds had a t_{max} of ∼60 s. Apparently, the mouth model system was less effective in releasing flavor compounds than mastication in the human mouth. Furthermore, the difference in volatility may have caused higher molecular weight compounds such as pyrazines to release more slowly than lower molecular weight aldehydes.

Effect of Salt Addition on I_{max} and t_{max}. The effect of salt addition on I_{max} and t_{max} of all compounds is shown in **Table 4**. A trend was observed for both in vitro and in vivo experiments that I_{max} decreased when salt was added, but the effect was not significant. Salt and increased saliva production (in vivo) may have had an effect on the partition coefficient of some compounds. There was, however, a significant decrease

Table 3. I_{max} and t_{max} of Compounds Released from French Fries at Different Frying Times

	I_{max} ($\times 10^5$ arbitrary units)				t_{max} (s)			
m/z	2 min	4 min	6 min	8 min	2 min	4 min	6 min	8 min
				In Vitro				
69	110 (28)[a]	166 (35)	217 (14)	273 (58)	44 (31)	78 (18)	46 (21)	53 (12)
73	149 (44)	245 (43)	353 (67)	457 (130)	35 (25)	51 (38)	39 (8)	55 (7)
75	24 (4)	72 (2)	121 (19)	142 (35)	47 (18)	70 (7)	64 (4)	92 (29)
87	146 (44)	243 (65)	348 (37)	438 (86)	36 (26)	63 (22)	48 (20)	55 (10)
91	27 (6)	45 (7)	63 (11)	80 (22)	44 (32)	54 (41)	53 (21)	57 (9)
95	17 (2)	22 (0.4)	25 (1)	32 (9)	110 (33)	116 (17)	137 (8)	194 (56)
109	11 (1)	14 (3)	17 (2)	33 (12)	245 (90)	273 (27)	265 (37)	295 (10)
113	16 (1)	22 (1)	24 (1)	23 (2)	69 (34)	84 (29)	116 (22)	99 (27)
123	5 (0.6)	6 (2)	8 (2)	14 (4)	284 (21)	276 (12)	285 (14)	302 (4)
137	3 (0.3)	4 (1)	5 (1)	8 (2)	267 (36)	262 (9)	302 (2)	296 (5)
153	8 (0.5)	8 (0.4)	9 (0.3)	9 (0.8)	275 (14)	286 (17)	298 (5)	300 (5)
				In Vivo				
69	3 (0.3)	9 (2)	11 (2)	20 (5)	5 (3)	7 (4)	8 (5)	6 (2)
73	5 (0.3)	22 (3)	31 (5)	53 (14)	5 (3)	6 (4)	5 (3)	6 (2)
87	4 (0.7)	11 (2)	15 (3)	24 (6)	5 (3)	7 (3)	6 (3)	6 (3)
95	0.7 (0.1)	1 (0.1)	1 (0.2)	2 (0.2)	7 (2)	7 (4)	8 (7)	7 (2)
109	0.7 (0.1)	0.7 (0.1)	0.8 (0.1)	1 (0.2)	6 (4)	5 (3)	5 (4)	5 (4)

[a] Standard deviations are given in parentheses.

Table 4. I_{max} and t_{max} of Compounds Released from French Fries with and without the Addition of Salt

	I_{max} ($\times 10^5$ arbitrary units)		t_{max} (s)	
m/z	no salt	with salt	no salt	with salt
	In Vitro			
69	209 (56)[a]	156 (46)	34 (1)	25 (6)
73	284 (63)	284 (82)	34 (1)	23 (7)
75	92 (39)	33 (19)	51 (2)	28 (7)
87	349 (105)	242 (88)	33 (1)	25 (4)
91	50 (11)	50 (2)	36 (4)	25 (4)
95	18 (2)	16 (2)	136 (14)	76 (9)
109	14 (1)	11 (5)	217 (37)	165 (33)
123	6 (0.6)	5 (2)	257 (26)	152 (45)
137	4 (0.3)	3 (1)	238 (20)	160 (45)
153	8 (0.3)	6 (0.6)	229 (17)	165 (30)
	In Vivo			
69	9 (5)	7 (3)	7 (4)	5 (3)
73	23 (18)	15 (9)	5 (3)	4 (2)
87	10 (5)	8 (4)	6 (3)	4 (3)
95	2 (0.3)	0.8 (0.4)	6 (3)	5 (4)
109	0.7 (0.2)	0.7 (0.2)	4 (3)	5 (2)

[a] Standard deviations are given in parentheses.

Table 5. I_{max} and t_{max} of Compounds Released from French Fries Produced According to the Conventional and Alternative Processes

	I_{max} ($\times 10^5$ arbitrary units)		t_{max} (s)	
m/z	conventional	alternative	conventional	alternative
	In Vitro			
69	210 (56)[a]	215 (78)	34 (1)	83 (20)
73	281 (67)	322 (126)	34 (1)	85 (21)
75	95 (36)	27 (14)	51 (2)	80 (21)
87	349 (105)	328 (136)	33 (1)	85 (21)
91	50 (11)	57 (22)	36 (4)	83 (18)
95	18 (2)	26 (9)	96 (4)	170 (30)
109	13 (1)	31 (14)	217 (37)	268 (19)
113	17 (2)	13 (2)	58 (13)	75 (23)
123	6 (0.5)	12 (5)	204 (22)	260 (9)
137	4 (0.3)	7 (3)	204 (19)	258 (1)
153	8 (0.3)	11 (2)	203 (7)	193 (83)
	In Vivo			
69	7 (5)	7 (4)	4 (2)	6 (2)
73	16 (10)	13 (6)	5 (2)	6 (2)
87	12 (7)	9 (3)	5 (2)	6 (1)
95	1 (0.6)	0.7 (0.3)	6 (2)	7 (3)
109	1 (0.7)	0.9 (0.2)	5 (2)	5 (3)

[a] Standard deviations are given in parentheses.

of t_{max} by the addition of salt in vitro. The effect on t_{max} was not significant for in vivo experiments, although the same trend was visible. The faster release of compounds after salt addition may be explained by a salting-out effect, causing the concentration of compounds in the air phase to increase (*19*, *26*). This phenomenon has been observed for aqueous solutions only. However, water vapor escaped from the surface of the French fries continuously, and some condensation may have occurred during cooling. Therefore, it may be possible that salt particles (partly) dissolved on the surface and constituted a salting-out effect.

Effect of the Alternative Process on I_{max} and t_{max}. The effect of the alternative process for the production of French fries using superheated steam on I_{max} and t_{max} is shown in **Table 5**. The variation of experiments with assessors was higher than that of experiments with the mouth model system, and this is in accordance with the effect of frying time and the effect of salt addition. There was no significant difference in I_{max} between French fries produced with the alternative and the conventional process. However, the alternative process resulted in a higher t_{max} of released compounds than the conventional process. This may have a strong impact on the sensory quality and would not have been observed with static volatile measurements alone, demonstrating the value of flavor release measurements. The oil content of French fries produced with the alternative process was reduced by ~30% (*18*). As lipids are known to retain nonpolar flavor compounds (*19*, *27*), the t_{max} of the compounds was expected to be higher for the conventional process. However, superheated steam, which was used in the alternative process, caused a skin on the surface of the French fries. The skin hindered evaporation of water during finish-frying in oil and absorption of oil thereafter (*18*). Although the oil content was lower, the oil was concentrated in a layer on the surface, and this may have caused the release of flavor compounds to slow.

In conclusion, with APCI-MS it is possible to identify and follow the release of several flavor compounds from French

fries both with assessors and with a mouth model system. Longer frying times resulted in a higher I_{max} for all compounds. Addition of salt caused a decrease of t_{max}, whereas an increase of t_{max} was found for the release of flavor compounds from French fries produced with the alternative process. Trends observed in experiments with assessors were found to be statistically significant with the mouth model system. Therefore, a combination of in vivo and in vitro measurements gives a synergy.

ABBREVIATIONS USED

APCI, atmospheric pressure chemical ionization; FID, flame ionization detection; GC, gas chromatography; I_{max}, maximum intensity; MANOVA, multivariate analysis of variance; MS, mass spectrometry; PTR, proton-transfer reaction; t_{max}, time of maximum intensity.

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