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Volatile Compounds of Swiss Processed Cheeses

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Introduction

Processed cheese is manufactured by heating a mixture of cheese, water, emulsifying salts and optional other ingredients. Mix constituents and processing conditions are chosen to give the desired structure, flavour, colour and storage stability at an acceptable cost. Typical emulsifying salts include sodium citrates, sodium orthophosphates and sodium polyphosphates (1). The mix is heated in a batch cooker or in a continuous process to 70–145 °C. Typically the hot processed cheese is filled into the desired packages such as slices or wedges. Thereafter the packages are sealed and the products are cooled down.

In the United States, processed cheese is one of the most popular cheese products. However, its flavour is one of the less studied subjects (2–4). Only pyrazines as «volatiles» or «aroma compounds» seem to have been reported in processed American cheese (5).

The first aim of this work is to list the volatile compounds identified in 11 commercial Swiss processed cheese varieties and to determine their relative distribution. Since their «matrices» are similar, a direct comparison of these different processed cheese types can be made using dynamic headspace analysis. The second aim of this work is to determine the influence of the heat treatment on the occurrence and stability of these volatile compounds.

Experimental part

Sampling

Eleven commercial processed cheese types were supplied by Tiger Käse AG (CH-3550 Langnau i.E.): Toast extra, Fettine Emmental, ¼ fett mild, Glarissa (with herbs), Gruyère with ham (medium fat), Salami, Emmental, Gruyère, Appenzell, Tartine extra-fin and Fondicrem. The first 3 products were supplied in slices (approximately 18 g), the others as wedges (approximately 30 g) packaged in an aluminium foil.

In order to study the thermal behaviour of volatiles in different processed cheeses, samples were taken from the line before and after the heat treatment of the cheese mix. The heat treatment was a continuous direct UHT processing (2–4 s at 140 °C). The coding of the samples was the following: E1 = Emmental, G1 = Gruyère, T1 = Tilsiter, S1 = Schabziger (= Glarissa) and a mixture of them (E1+T1+S1) before heat processing, and E2 = Emmental, G2 = Gruyère, T2 = Tilsiter, S2 = Schabziger and a mixture of them (E2+T2+S2) after heat processing.

Sample preparation

Before analysis, samples were manually grated in a deep frozen state using a domestic rasp (i.e. Bircher rasp or equivalent), in a refrigerated room at 7–8 °C. A representative sample of approximately 10 g was weighed and introduced into a 25 ml sparger before extraction of the volatiles.

Extraction of volatiles

The Purge and Trap system LSC 2000 (Tekmar, Cincinnati, OH, USA) included a 25 ml non-fritted sparger (Schmidlin Co, part no. 14-2333-4SL, CH-6345 Neuheim), a trap (no. 8, containing a mixture of Carbosieve SIII (0.05 g) and Carbopack B60/80 (0.2 g) as well as a cryofocusing unit. The moisture control module was not used. Operating conditions were as follows: room temperature; purge gas: nitrogen; purge flow (vent): 30 ml/min; prepurge: 1 min; purge: 10 min; dry purge: 10 min; cap cool-down: –125 °C; desorb preheat to 210 °C; desorb: 4 min at 220 °C; inject: within 1.5 min from –125 to 200 °C; bake: 5 min at 260 °C; 6-port valve: 150 °C; line: 150 °C; capillary union heater (= transfer line from purge and trap to gas chromatograph): 150 °C.

Gas chromatography

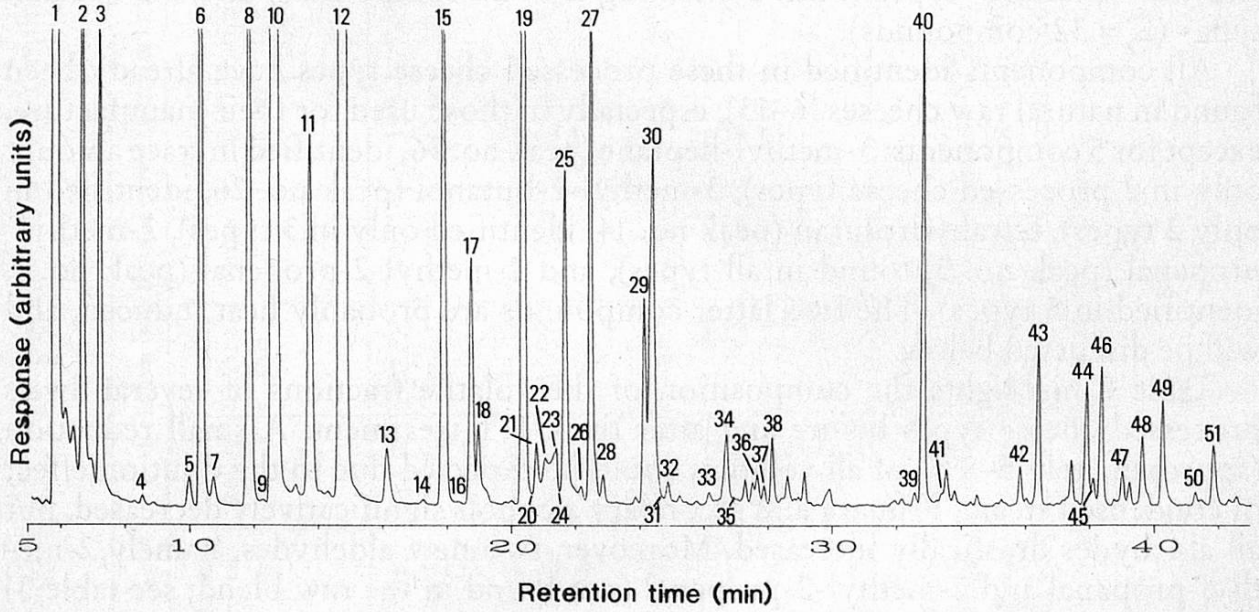
A Hewlett-Packard (HP) 5890, Series II was used. Operating conditions were as follows: carrier gas: helium; inlet pressure 40 kPa; flow: approx. 1.6 ml/min, injection at 45 °C; transfer line (from GC to MS): 280 °C; interface: direct inlet;

temperature program: 13 min at 45 °C, heating rate: 5 °C/min to 240 °C, and 5 min at 240 °C; capillary column: SPB1 (Supelco), 30 m × 0.32 mm id., film thickness: 4 µm.

Detection

Two detectors were mounted in parallel by splitting the flow at the end of the capillary column (split ratio: approx. 1:1 at 45 °C), i.e. a Hewlett-Packard flame ionisation detector (FID) and a mass-sensitive detector (MSD model HP 5972), operating in the scan mode (TIC) from 19 to 250 amu at 2.9 scan/s, ionisation by EI at 70 eV by autotuning; MS-Scan after 3.5 min. The MSD was used for the identification of the volatile (flavour) compounds (signal/noise ratio = 3), the FID for determination of their relative quantities (6). All determinations were performed twice.

Figure 1 shows a typical GC/FID chromatogram of a processed cheese.



Caption: The peak numbering is indicated in table 1–7. Some peaks, visible on the GC/FID chromatogram, were not considered: they were below the detection limit for identification in the corresponding GC/MSD chromatogram. Others, identified in other processed cheese types but not visible on this figure, are nevertheless indicated as retention time (i.e. peaks no. 20, 24, 31 and 35). Further peaks, due to contaminants present in the blank, were not considered

Fig. 1. Typical GC/FID chromatogram of volatile compounds found in the Salami-type processed cheese

Results and Discussion

Using the MSD, 51 volatile compounds were identified from the 11 commercial Swiss processed cheeses studied. These compounds are grouped into alcohols (table

1), ketones (table 2), aldehydes (table 3), esters (table 4), hydrocarbons (table 5), terpenes (table 6) and miscellaneous components (table 7) according to their chemical functionality.

Each table indicates the frequency Σ_i ($1 \leq i \leq 11$) of occurrence of a given volatile component i in the various processed cheese types investigated. Most volatiles occur in most cheese varieties but at different concentrations (the detection limit, defined by a signal/noise ration of 3, corresponds to 500 arbitrary units for the FID). Several components such as 2-methyl-propanal, 2-methyl- and 3-methyl-butanal, ethanol, 2-propanol, 2-propanone, 2,3-butanedione, 2-butanone, 2-pentanone, 2-heptanone occurred in all the 11 cheese varieties. Many others occurred frequently.

Table 7 also shows the total number of volatiles Σ_j contained in a given processed cheese type j. The cheese variety with the least number of volatiles was the «¼ Fett» ($\Sigma_j = 18$ compounds identified) followed by the «Fondicream» cheese type ($\Sigma_j = 19$ compounds). The cheeses with the highest number of volatiles were the «Gruyère» and the «Glarissa» types (both containing $\Sigma_j = 29$ compounds) and the «Salami type» ($\Sigma_j = 32$ compounds).

All components identified in these processed cheese types have already been found in natural raw cheeses (6–13), especially in those used for their manufacture, except for 5 components: 3-methyl-heptane (peak no. 36, identified in trace amount only in 2 processed cheese types), 3-methyl-2-butanol (peak no. 26, identified in only 2 types), tetrahydrofuran (peak no. 14, identified only in 3 types), 2-methyl-propanal (peak no. 5, found in all types), and 2-methyl-2-propenal (peak no. 7, identified in 5 types). The two latter compounds are probably heat induced, and will be discussed below.

Table 8 highlights the composition of the volatile fractions of several Swiss processed cheese types before and after the UHT treatment. A small reduction (approximately 5–8%) of all volatiles could be expected due to the dilution effect of condensed steam. Primary and secondary alcohols significatively decreased, but all aldehydes drastically increased. Moreover, two new aldehydes, namely 2-methyl-propanal and 2-methyl-2-propenal (not found in the raw blend; see table 3) occurred in most processed cheeses. This general trend could be related to the β-oxidation of the unsaturated fatty acids or originate from the corresponding amino acids. Any new compounds could also be originally present as conjugates, sulphates or glucuronidates, from which they are liberated by heat. Many ketones decreased in concentration, but some others increased. All esters decreased. This behaviour was also observed with the aromatic hydrocarbons and terpenes. These various losses could be explained by the volatility of these compounds.

A small amount of α-pinene occurred in the «Glarissa» processed cheese which contains alpine herbs («Ziegerklee») (14). Nine further terpenes were identified, mostly in trace amounts below the detection limit in the Total Ion Current mode (TIC) in the Salami type using the MSD in Selected Ion Monitoring (SIM) mode and the retention indices of these terpenes. They originate from the added spices and not from the cheese base used.

Table 1. Alcohols identified (with MSD) and whose relative quantities were determined with FID in various Swiss processed cheese types

Peak No	Compounds	Σ_i	Relative height measured in different cheeses (arbitrary units**)											
			Emmentaler	Gruyère	Appenzeller	Tartine (extra-fin)	Fondicrem	Toast extra (Slices)	fettine Emmentaler (Slices)	¼ fett mild (Slices)	Glarissa (with alpine herbs)	Gruyère with ham	Salami	Reference number related to other cheese varieties
1	ethanol	11	36888	54057	81421	33128	18451	60352	27422	51512	335552	70041	98150	(7,9,11)
6	1-propanol*	10	3606	3623	4456	2651	729	4111	3071		107042	3333	4397	(7,9,11)
13	2-methyl-1-propanol*	5		579	771						978	4816	608	(7,9,11)
18	1-butanol*	7	611	1080	1056			1021			177218	727	502	(7,9,11)
29	3-methyl-1-butanol*	10	3262	3704	4504	1058	909	2230	2024		2355	3348	3039	(7,9,11)
31	2-methyl-1-butanol*	4	1912	2246	2150				1596					(7)
33	1-pentanol*	2								1749	2387			(7,9,11)
3	2-propanol*	11	5752	5877	9735	1865	1313	3516	2973	755	4409	4656	7652	(7,9,11)
4	2-methyl-2-propanol	3				782						1145	510	(7,9,11)
11	2-butanol*	8	652	775	2984	781		5416			2827	1159	2814	(7,9,11)
25	2-pentanol	7	3156	2587	5568	783		1911				2156	2921	(7,9,11)
26	3-methyl-2-butanol	2							4298		1367			

Caption: blank = not detected (detection limit: 500 arbitrary units).
trace = trace amount (below the detection limit, found with MSD in SIM mode).
* = confirmed by comparison of retention times and MS spectra of authentic substances.
** = the extraction rate of the different compounds using the dynamic headspace analysis is unknown.
Σ_i = number of cheese varieties containing a given component i.

Table 2. Ketones identified (with MSD) and whose relative quantities were determined with FID in various Swiss processed cheese types

Peak No	Compounds	Σ_i	Relative height measured in different cheeses (arbitrary units**)											
			Emmentaler	Gruyère	Appenzeller	Tartine (extra-fin)	Fondicrem	Toast extra (Slices)	fettine Emmentaler (Slices)	¼ fett mild (Slices)	Glarissa (with alpine herbs)	Gruyère with ham	Salami	Reference number related to other cheese varieties
2	2-propanone*	11	18136	20805	16776	4546	6281	20631	12948	5541	18313	10871	9099	(7,8,9)
8	2,3-butanedione*	11	4839	5925	6289	19114	13236	7161	8504	31874	7515	5684	5210	(7,8,12)
10	2-butanone*	11	25164	22549	33801	46637	73201	8136	1557	1750	56020	64953	95748	(7,10,11)
16	3-methyl-2-butanone	1						712						(4,5)
19	2-pentanone*	11	20933	21179	28854	6884	6491	17832	12539	2715	19561	1371	12427	(7,10,11)
20	3-methyl-2-pentanone*	1									1420			(7,9,10)
21	2,3-pentadienone	7		1250	2010		707	1207	1450			17298	1045	(7)
24	3-hydroxy-2-butanone	2				508				1022				(7,8)
30	4-methyl-2-pentanone*	6				4945	3281	1893			5203	5813	3749	(7,11)
40	2-heptanone	11	13540	1233	14412	5408	4594	9094	12327	2798	10824	10934	7486	(7,8,11)
51	2-nonanone	10	1987	9865	1499	1014	851	1342	2785		677	1010	938	(7,8,11)

Caption: See table 1

Table 3. Aldehydes identified (with MSD) and whose relative quantities were determined with FID in various Swiss processed cheese types

Peak No	Compounds	Σ_i	Relative height measured in different cheeses (arbitrary units**)											
			Emmentaler	Gruyère	Appenzeller	Tartine (extra-fin)	Fondicrem	Toast extra (Slices)	fettine Emmentaler (Slices)	¼ fett mild (Slices)	Glarissa (with alpine herbs)	Gruyère with ham	Salami	Reference number related to other cheese varieties
5	2-methyl-propanal	11	2495	2182	1444	470	533	2974	941	4016	918	656	644	
7	2-methyl-2-propenal	5	848	802		559		1008		870				
9	butanal	1						trace			1742			(7,10)
15	3-methyl-butanal*	11	11092	14190	14511	9834	7126	13096	5577	16338	19590	4949	9096	(7,8,12)
17	2-methyl-butanal*	11	5212	5212	4715	2481	1884	6575	2200	9168	7605	1990	2042	(7,8,12)
22	pentanal	10	987	807	1189	957	882	1284	1315	8613	2404	1079		(7,9,10)
37	hexanal	3	531	531						1028				(7,9,10)

Caption: See table 1

Table 4. Esters identified (with MSD) and whose relative quantities were determined with FID in various Swiss processed cheese types

Peak No	Compounds	Σ_i	Relative height measured in different cheeses (arbitrary units**)											
			Emmentaler	Gruyère	Appenzeller	Tartine (extra-fin)	Fondicrem	Toast extra (Slices)	fettine Emmentaler (Slices)	¼ fett mild (Slices)	Glarissa (with alpine herbs)	Gruyère with ham	Salami	Reference number related to other cheese varieties
12	ethyl acetate*	10	3785	3942	4898	65043	48424	2930	2455		48426	73116	81941	(7,9,10)
27	ethyl propionate	10	7961	6906	8745	2628	1421	6965	6560		2785	7063	6138	(7,9,10)
38	ethyl butanoate*	8	750	580	751			651	671		2241	686	806	(7,9,11)
41	propyl butanoate	1									1231			(7,9,10)

Caption: See table 1

Table 5. Hydrocarbons identified (with MSD) and whose relative quantities were determined with FID in various Swiss processed cheese types

Peak No	Compounds	Σ_i	Relative height measured in different cheeses (arbitrary units**)											
			Emmentaler	Gruyère	Appenzeller	Tartine (extra-fin)	Fondicrem	Toast extra (Slices)	fettine Emmentaler (Slices)	¼ fett mild (Slices)	Glarissa (with alpine herbs)	Gruyère with ham	Salami	Reference number related to other cheese varieties
28	heptane	2		714									620	(7)
34	methyl benzene*	10	788	951	1114	737		6708	1863	10179	2210	1885	2045	(7)
36	3-methyl-heptane	2										1096	734	
39	1,3-dimethyl-benzene	1	760											(7,9,10)

Caption: See table 1

Table 6. Terpenes identified (with MSD) and whose relative quantities were determined with FID in various Swiss processed cheese types

Peak No	Compounds	Σ_i	Relative height measured in different cheeses (arbitrary units**)											
			Emmentaler	Gruyère	Appenzeller	Tartine (extra-fin)	Fondicrem	Toast extra (Slices)	fettine Emmentaler (Slices)	¼ fett mild (Slices)	Glarissa (with alpine herbs)	Gruyère with ham	Salami	Reference number related to other cheese varieties
42	α-thujene*	1											605	(13)
43	α-pinene*	1									trace		2083	(13)
44	sabinene*	1											2015	(13)
45	β-myrcene*	0											trace	(13)
46	β-pinene*	1											1951	(13)
47	α-phellandrene*	0											trace	(13)
48	δ-3-carene*	1										trace	739	(13)
49	dl-limonene*	1											1498	(13)
50	γ-terpinene*	0											trace	(13)

Table 7. Miscellaneous compounds identified (with MSD) and whose relative quantities were determined with FID in various Swiss processed cheese types

Peak No	Compounds	Σ_i	Relative height measured in different cheeses (arbitrary units**)											
			Emmentaler	Gruyère	Appenzeller	Tartine (extra-fin)	Fondicrem	Toast extra (Slices)	fettine Emmentaler (Slices)	¼ fett mild (Slices)	Glarissa (with alpine herbs)	Gruyère with ham	Salami	Reference number related to other cheese varieties
14	tetrahydrofuran	3						1754	675	2013				
23	propanoic acid	6	767	1051		826	952		2834		1716			(7,8,12)
32	dimethyl disulfide*	7	789	4004	14816			10802	847	1604	trace	541		(7,11)
35	butanoic acid	1									4047			(7,8,12)
	Total number of components contained in a given cheese variety j (Σ j)	Σ j	27	29	25	24	19	27	24	18	29	27	32	

Caption: See table 1

Table 8. Distribution of volatiles in various processed cheese varieties before and after heat treatment

Compounds	Relative peak height (arbitrary unit)							
	Emmentaler E1	Emmentaler E2	Greyerzer G1	Greyerzer G2	Tilsiter T1	Schabziger S1	Schabziger Mixture S1+T1+E1	Schabziger Mixture S2+T2+E2
ethanol	154 046	84 197	149 259	56 574	219 202	1 086 234	1 133 168	775 986
1-propanol	13 718	4 796	7 460	5 580	–	113 870	223 173	123 702
2-methyl-1-propanol	1 446	590	–	–	1 200	–	1 719	1 455
1-butanol	1 792	623	6 079	1 896	–	496 176	491 340	311 740
2-methyl-1-butanol	4 546	2 518	1 203	665	–	–	2 530	1 637
3-methyl-1-butanol	7 862	3 987	1 915	906	–	10 577	7 483	5 988
1-pentanol	–	–	–	–	–	27 627	12 219	8 535
2-propanol	13 065	4 194	4 278	2 697	4 568	–	17 524	4 421
2-butanol	1 500	1 419	1 719	512	–	–	31 061	29 484
3-methyl-2-butanol	–	–	1 990	653	3 065	–	5 089	–
2-pentanol	7 468	4 534	–	–	–	–	4 288	–
3-penten-2-ol	–	–	–	–	–	14 752	–	–
2-heptanol	679	452	–	–	–	–	–	–
2-methyl-propanal	–	2 864	547	2 456	–	–	–	1 056
2-methyl-propenal	–	1 427	–	1 298	–	–	–	–
butanal	–	–	–	–	–	–	6 712	2 833
3-methyl-butanal	1 162	17 046	2 475	11 848	1 409	–	1 287	20 376
2-methyl-butanal	2 808	5 988	1 524	5 188	–	–	746	7 399
pentanal	–	2 215	–	–	–	–	–	2 246
hexanal	345	1 880	–	–	–	–	–	–

Compounds	Relative peak height (arbitrary unit)							
	Emmentaler E1	Emmentaler E2	Greyerzer G1	Greyerzer G2	Tilsiter T1	Schabziger S1	Schabziger Mixture S1+T1+E1	Schabziger Mixture S2+T2+E2
2-propanone	31 971	16 139	20 245	22 980	88 440	–	36 546	16 852
2,3-butanedione	13 872	8 952	13 133	6 823	75 520	–	48 088	17 084
3-hydroxy-2-butanone	–	–	–	–	17 370	–	–	–
2-butanone	5 126	7 591	3 480	2 787	–	–	19 956	16 807
2,3-pentadienone	–	–	1 450	972	–	–	–	–
2-pentanone	46 656	22 951	13 973	12 492	–	–	18 184	10 807
4-methyl-2-pentanone	2 308	2 484	–	–	–	–	–	–
2-hexanone	895	549	–	–	–	–	–	–
2-heptanone	16 479	13 864	6 307	6 932	3 065	–	7 924	9 219
2-nonanone	3 306	3 062	1 094	268	–	21 806	872	1 456
ethyl acetate	7 620	5 355	2 381	7 787	1 111	–	6 196	3 599
butyl acetate	–	–	–	–	–	68 267	10 514	4 706
propyl acetate	–	–	–	–	–	–	10 068	?
ethyl propionate	23 742	13 678	4 086	2 298	–	–	21 014	4 669
propyl propanoate	560	388	–	–	–	–	–	–
butyl propionate	–	–	–	–	–	146 273	8 684	2 291
ethyl butanoate	1 317	991	761	539	–	118 330	36 971	23 776
propyl butanoate	–	–	–	–	–	56 358	6 165	3 920
butyl butanoate	–	–	–	–	–	–	8 684	6 559
methyl benzene	5 013	3 858	2 001	2 737	3 205	–	2 764	3 454
1,3-dimethylbenzene	–	–	–	–	–	–	–	691
decane	–	–	–	–	768	–	1 088	–
β-myrcene	1 304	–	–	–	–	–	–	–
β-pinene	740	–	–	–	–	–	–	–
γ-terpinene	2 311	–	–	–	–	–	–	–
dimethyl disulfide	–	–	3 923	3 276	–	–	2 065	1 171

Caption: E1: Swiss Emmentaler before heat treatment
E2: Swiss Emmentaler after heat treatment
G1: Greyerzer before heat treatment
G2: Greyerzer after heat treatment
T1: Tilsiter before heat treatment
S1: Schabziger before heat treatment
S1+T1+E1: Schabziger mixture before heat treatment
S2+T2+E2: Schabziger mixture after heat treatment
blank = not detected (detection limit: 500 arbitrary units)
? = peak overlapping

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Summary

The present study identifies and gives relative quantities for 51 volatiles found in 11 Swiss commercial processed cheese varieties using a dynamic headspace GC-MSD & FID analysis. Most of the volatiles are present in all processed cheese types but in different ratios. Most components are already preformed in the raw material before heat processing. The thermal treatment of the various processed cheese varieties generally produced a decrease in the concentration of most alcohols, ketones (with a few exceptions), esters, as well as terpenoids, due to their relatively high volatility. On the other hand, the significant increase in the concentration of all aldehydes seems to be related to the heat processing. The presence of the terpenes can be explained by the presence of herbs (Glarissa processed cheese) or spices (Salami processed cheese).

Zusammenfassung

In 11 verschiedenen schweizerischen Schmelzkäsesorten wurden 51 flüchtige Verbindungen mittels einer dynamischen Dampfraum- und GC/MSD & FID-Analysenmethode nachgewiesen. Die meisten Verbindungen wurden in sämtlichen Käsesorten gefunden, aber in sehr unterschiedlichen Konzentrationen. Im allgemeinen war die Mehrheit der Komponenten bereits im Rohmaterial (vor der thermischen Behandlung) vorhanden. Nach dem Schmelzen der Käsemasse beobachtete man normalerweise eine Gehaltsabnahme der meisten flüchtigen Stoffe wie Alkohole und Ketone (mit einigen Ausnahmen), Ester und Terpenoide, was auf deren hohe Flüchtigkeit zurückzuführen ist. Im Gegensatz dazu scheint die deutliche Konzentrationszunahme sämtlicher Aldehyde von der thermischen Behandlung verursacht zu sein. Das Vorkommen einiger Terpene in Spuren kann durch die Verwendung von Kräutern (im Glarissa-Schmelzkäse) oder Gewürzen (im Salamischmelzkäse) erklärt werden.

Résumé

Le présent travail a permis d'identifier et de quantifier de façon relative 51 composés volatils dans 11 sortes de fromages fondus suisses du commerce en utilisant une technique d'analyse d'effluves dynamique par GC/MSD & FID. La plupart de ces composants sont présents dans la majorité des sortes de fromages fondus analysés, mais dans des proportions très variables. Maints composés ont d'ailleurs déjà été trouvés dans la matière première avant la fonte. Après celle-ci, la teneur en volatils tels que alcools, cétones (à quelques exceptions près), en esters et en hydrocarbures aromatiques tendait en général à décroître en raison de leur volatilité relativement élevée. Inversement, l'augmentation significative de la teneur en aldéhydes semble clairement indiquer que ces substances sont générées par le traitement thermique appliqué. La présence de quelques composés terpéniques s'explique par l'emploi de plantes (dans la sorte «Glarissa» aux herbes) ou d'épices (dans la sorte «au salami»).

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