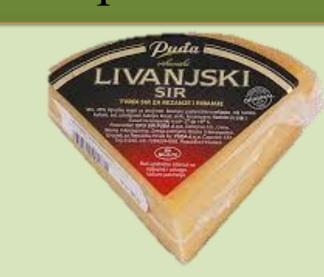


VOLATILE COMPOUNDS OF FLAVOURED CHEESES

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Cheese is a widespread and valuable food that is used in everyday life. Cheese made from a mixture of cow's and sheep's milk is traditionally produced on farms and in cheese factories throughout Croatia. Considering the production technology, it belongs to the group of hard cheeses.





The flavour of cheese is one of the main quality parameter of a certain type of cheese. Flavouring improves the sensory properties, but also the preservation of food and the satisfaction of gourmets. Cheeses are flavoured with different aromatic plants such as lavander, sage, basil and many others. In addition to nutritional properties, supplements are also useful due to their antimicrobial value.

Extractant phase leadspace Sample

Livanjski sir, a full-fat hard cheese made from a mixture of cow's and sheep's milk, served as the base. Three herb species were used for the flavouring of cheese: lavender (*Lavandula officinalis* L.), basil (*Ocimum basilicum* L.,) and sage (*Salvia officinalis* L.). Each herb species was added in two concentrations: 0.05 % and 0.25 %.

Isolation of volatile compounds was performed by solid phase microextraction (HS-SPME) using DVB/CAR/PDMS fiber at a temperature of 60 °C (n = 3). All samples were analysed by gas chromatography-mass spectrometry using a non-polar column and the results were compared. The results are presented as means ± standard deviations.

Headspace volatile compounds of flavoured cheeses

Compound	در (min)	Peak area (%) ± STD
		lavander 0,05%	lavander 0,25%
ethanol	1,445	$4,24 \pm 2,118498$	3,97 ± 0,510033
acetic acid	1,557	$8,77 \pm 0,525579$	7,03 ± 1,540433
propanoic acid	1,901	$9,84 \pm 0,35171$	7,31 ± 1,160014
methyl isothiocyanate	2,234	$1,50 \pm 0,513907$	1,10 ± 0,401123
butanoic acid	2,602	$13,58 \pm 1,338108$	11,45 ± 1,591446
ethyl butamoate	2,651	1,08	0,89 ±,374477
3-methylbutanoic acid**	3,050	$0,54 \pm 0,285132$	1
2-methylbutanoic acid**	3,172	0,36	/
pentanoic acid	3,633	$0,36 \pm 0,15308$	0,29 ± 0,045092
heptan-2-one	3,917	$2,65 \pm 0,633114$	2,67 ± 0,348186
heptan-2-ol	4,063	$1,18 \pm 0,244404$	0,95 ± 0,410041
benzaldehyde	5,484	$0,23 \pm 0,026458$	0,19 ± 0,023094
hexanoic acid	6,253	$22,26 \pm 1,445902$	19,92 ± 0,28746
limonene	7,375	$5,67 \pm 1,891278$	3,57 ± 1,852278
<i>trans-</i> β-ocimene	7,624	$0,53 \pm 0,200749$	0,40 ± 0,217945
2-phenylacetaldehyde	7,844	0.38 ± 0.095349	0,69 ± 0,098489
cis-β-ocimen	7,955	I .	0.10 ± 0.01
γ-terpinen	8,351	I	0,09 ± 0,026458
octan-1-ol*	8,680	Į.	0,11 ± 0,025166
cis-linalool oxide	8,818	1	0,39 ± 0,132791
heptanoic acid	8,877	0.33 ± 0.028868	1
non-8-en-2-one	9,159	$0,15 \pm 0,040415$	0,27 ± 0,078102
trans-linatool oxide	9,373	Į.	0,25 ± 0,04
nonan-2-one	9,471	$1,42 \pm 0,337244$	1,94 ± 0,25
linalool	9,766	$0,56 \pm 0,087369$	8,97 ± 0,810021
nonanal	9,921	$0,14 \pm 0,045826$	1
2-phenylethanol	10,257	0,11	1
camphor	11,476	I .	0,20 ± 0,017321
borneol	12,309	$0,15 \pm 0,025166$	1,65 ± 0,109697
terpinen-4-ol	12,760	I	3,65 ± 0,105987
octanoic acid	13,120	$14,99 \pm 3,694121$	11,79 ± 6,377
o-terpineol	13,302	1	0,69
ethyl octanoate	13,576	$0,63 \pm 0,092376$	0,80 ± 0,110151
dodecane	13,651	$0,24 \pm 0,040415$	0,21 ± 0,043589
linalyl acetate	15,985	1	2,15 ± 0,101527
nonanoic acid	16,598	0.13 ± 0.01	0,06 ± 0,005774
lavandulyl acetat	17,452	1	0,10 ± 0,02
undecan-2-one	17,551	$0,15 \pm 0,015275$	0,19 ± 0,07
hexyl tiglate**	19,098	7	0,11
decanoic acid	20,840	$4,68 \pm 1,236986$	3,30 ± 1,091238
ethyl decanoate	21,802	$0,20 \pm 0,040415$	0,22 ± 0,025166
tetradecane	21,933	$0,10 \pm 0$	1
caryophyllene	22,693	1	0,12 ± 0,015275
coumarine	23,278	1	0,10 ± 0,02
β-famesene*	24,249	1	0,09 ± 0
δ-decalaktone	25,732	$0,33 \pm 0,06245$	0,26 ± 0,017321
dodecanoic acid	28,438	$0,20 \pm 0,161967$	0,15 ± 0,051316
δ-dodecalactone	_	0.11 ± 0.030551	0,10 ± 0,005774
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Compound	t_{κ} (min)	Peak are	ea (%) ± STD
		basil 0,05%	basil 0,25%
ethanol	1,445	$4,32 \pm 0,405832$	$3,29 \pm 1,081403$
acetic acid	1,557	$6,93 \pm 1,985925$	$6,76 \pm 1,140234$
propanoic acid	1,901	$8,38 \pm 2,93869$	$9,20 \pm 0,592312$
methyl isothiocyanate	2,234	$1,43 \pm 0,226789$	$1,45 \pm 0,28006$
butanoic acid	2,602	$9,43 \pm 1,617354$	$10,38 \pm 0,727942$
ethyl butanoate	2,651	0.94 ± 0.39	$0,61 \pm 0,092916$
heptan-2-one	3,917	$4,27 \pm 1,019035$	$3,21 \pm 0,497896$
heptan-2-ol	4,063	$1,44 \pm 0,511566$	0.57 ± 0.250067
benzaldehyde	5,484	0.71 ± 0.286182	$0,69 \pm 0,232451$
sabinene	5,801	1,88	1,80
hexanoic acid	6,253	$16,13 \pm 2,730513$	$13,32 \pm 3,620331$
ethyl hexanoate	6,423	$2,97 \pm 1,13697$	$8,20 \pm 5,798209$
limonene	7,375	$5,96 \pm 3,345211$	$5,04 \pm 3,408269$
1,8-cineole	7,464	$0,18 \pm 0,077675$	$0,45 \pm 0,092916$
trans-β-ocimene	7,624	0.59 ± 0.312623	0.50 ± 0.350428
2-phenylacetaldehyde	7,844	0.49 ± 0.233524	0.98 ± 0.22053
cis-β-ocimene	7,955	0,09	0,11
γ-terpinene	8,351	0,10	0,11
heptanoic acid	8,877	0.21 ± 0.02	0.28 ± 0.032146
non-8-en-2-one	9,159	0.26 ± 0.047258	0.29 ± 0.096437
nonan-2-one	9,471	$2,06 \pm 0,451331$	$2,01 \pm 0,144338$
linalool	9,766	$1,95 \pm 0,545008$	$5,05 \pm 1,247117$
nonanal	9,921	0,25	0.15 ± 0.07
o-thujon	10,014	0.14 ± 0.055076	1
alloocimene*	10,864	$0,10 \pm 0,026458$	0,11
camphor	11,476	0,09	1
octanoic acid	13,120	$19,69 \pm 14,04437$	$17,49 \pm 9,076145$
α-terpineol	13,302	I	0,35
ethyl octanoate	13,576	$1,64 \pm 0,162583$	$1,13 \pm 0,041633$
dodecane	13,651	0.29 ± 0.110151	0.29 ± 0.091652
nonanoic acid	16,598	0.04 ± 0	0,08
bornyl acetate	17,223	I	0.10 ± 0.020817
undecan-2-one	17,551	$0,16 \pm 0,047258$	$0,24 \pm 0,02$
eugenol	20,170	0.12 ± 0.011547	$0,49 \pm 0,047258$
decanoic acid	20,840	$3,35 \pm 0,547753$	$4,31 \pm 1,669341$
β-elemene	21,611	1	0,06
ethyl decanoate	21,802	0.39 ± 0.098658	0.38 ± 0.020817
∞-bergamoten*	23,372	ſ	0.08 ± 0.020817
germacrene D	25,169	ſ	0,04
5-decalactone	25,732	0,26 ± 0,025166	$0,34 \pm 0,060277$
γ-cadinene*		0,13	
dodecanoic acid		0,11 ± 0,037859	
δ-dodecalactone	33,709	0,09	0,10 ± 0,064291
	-	-	

Compound	t_{κ} (min)	Peak area (%) ± STD	
		sage 0,05%	-
ethanol	1,445	$1,99 \pm 0,720625$	-
acetic acid	1,557		
propanoic acid	1,901	$10,43 \pm 1,556931$	
methyl isothiocyanate	2,234		
butanoic acid	2,602		
ethyl butanoate	2,651	0,52	0.79 ± 0.299555
heptan-2-one	3,917	$4,88 \pm 0,921466$	
heptan-2-ol	4,063	0.89 ± 0.288271	$0,67 \pm 0,20502$
α-pinene	4,893	0.29 ± 0.159478	
camphene	_	0.47 ± 0.225389	$1,40 \pm 0,104403$
benzaldehyde	5,484	0.18 ± 0.047258	0.15 ± 0.07
sabinene	5,801	$1,46 \pm 0,315119$	$1,03 \pm 0,408207$
hexanoic acid	6,253	$15,39 \pm 1,31371$	$10,65 \pm 0,875386$
ethyl hexanoate	6,423	0.82 ± 0.005774	$1,63 \pm 0,72148$
α-terpinene	6,958	1	0.08 ± 0.02
p-cymen	7,258	0.17 ± 0.051316	0.51 ± 0.133167
limonene	7,375	7.41 ± 1.594125	$6,32 \pm 2,952643$
1,8-cineole	7,464	0.67 ± 0.165025	$2,03 \pm 0,500233$
trans-β-ocimene	7,624	0.78 ± 0.250599	0.56 ± 0.37072
2-phenylacetaldehyde	7,844	0.88 ± 0.266896	$1,15 \pm 0,388887$
cis-β-ocimene	7,955	0.10 ± 0.015275	0,09
γ-terpinene	8,346	0.11 ± 0.015275	0.21 ± 0.036056
heptanoic acid	8,877	0.20 ± 0.037859	$0,16 \pm 0,065064$
non-8-en-2-one	9,159	0.70 ± 0.159478	0.28 ± 0.045826
α-terpinolene	9,349	1	0.30 ± 0.073711
nonan-2-one	9,471	$4,65 \pm 0,822131$	$2,08 \pm 0,517333$
linzlool	9,766	0.63 ± 0.037859	0.80 ± 0.132035
nonanal	9,921	0.13 ± 0.015275	0,13
othujone	10,014	$2,73 \pm 0,896456$	$8,10 \pm 1,571189$
β-thujone	10,416	0.32 ± 0.147309	$1,01 \pm 0,196977$
alloocimene*	10,864	0.10 ± 0.035119	0,09
camphor	11,476	$2,00 \pm 0,461122$	$5,67 \pm 0,837397$
borneol	12,309	0.34 ± 0.085049	$1,02 \pm 0,176163$
octanoic acid	13,120	$13,63 \pm 2,302803$	$11,20 \pm 7,295768$
α-terpineol	13,302	T.	0,21
ethyl octanoate	-	1,13 ± 0,590028	-
dodecane		0,31 ± 0,060828	
nonanoic acid		0,13 ± 0,092376	
bornyl acetate	-	0,26 ± 0,086603	-
undecan-2-one		0,30 ± 0,060277	
decanoic acid		3,02 ± 0,975551	
ethyl decanoate		0,16 ± 0,017321	
tetradecane	21,933	-	/
caryophyllene	22,693	-	0,09
o-humulene		0,23 ± 0,135769	
δ-decalactone	-	0.23 ± 0.045092	-
	_		
dodecanoic acid viridiflorol	28,438 89,435	0,14	0,15 0,12
ethyl dodecanoate	29,632	r	0,06
1	-	0.00	
δ-dodecalactone	33,709	0,09	0,12

Headspace volatile compounds of Livanjski sir

Compound	t _{s.} (min)	Peak area (%) ± STD
ethanol	1,445	$3,30 \pm 1,063689$
acetic acid	1,557	6,99 ± 0,502096
propanoic acid	1,901	9,46 ± 0,433128
methyl isothiocyznate	2,234	$1,48 \pm 0,571664$
butanoic acid	2,602	$13,25 \pm 0,321299$
pentanoic acid	3,633	$0,26 \pm 0,083267$
heptan-2-one	3,917	3,97 ± 0,10116
heptan-2-ol	4,063	1,59 ± 0,600528
benzaldehyde	5,484	$0,29 \pm 0,115902$
sabinene	5,809	1,69 ± 0,642106
hexanoic acid	6,253	20,56 ± 1,431049
ethyl hexanoate	6,423	0,80 /
limonene	7,375	6,59 ±2,710615
<i>trans</i> -β-ocimene	7,624	$0,64 \pm 0,295014$
2-phenylacetaldehyde	7,844	$0,25 \pm 0,025166$
γ-terpinene	8,346	0.09 ± 0.015275
heptanoic acid	8,877	$0,26 \pm 0,047258$
non-8-en-2-one	9,159	0,36 ± 0,043589
nonan-2-one	9,471	$2,77 \pm 0,182483$
linalool	9,757	$0,51 \pm 0,089629$
nonanal	9,921	0.12 ± 0.034641
alloocimene*	10,864	0,10 /
octanoic acid	13,120	$18,66 \pm 8,7669$
ethyl octanoate	13,561	$0,44 \pm 0,015275$
dodecane	13,638	0.29 ± 0.060828
nonanoic acid	16,598	0,06 /
undecan-2-one	17,551	0.17 ± 0.026458
decanoic acid	20,840	$3,12 \pm 0,756395$
ethyl decanoate		0,07 /
δ -decalactone	25,732	0.30 ± 0.046188
dodecanoic acid	28,438	0.10 ± 0.030551
$\delta\text{dodecalactone}$	33,709	0.09 ± 0.015275

Symbols in tables:

 $t_{\rm R}$ = retention time on HP-5MS column;

STD = standard deviation

/ =not detected; tr = traces (< 0,1 %);

* = correct isomer is not identified;

^a = compound identified only by mass spectra comparison with Wiley9 and/or NIST17 mass spectral libraries

In all samples, the most abundant compounds are carboxylic acids, especially the fatty acids butanoic, hexanoic and octanoic acid as well as propanoic and acetic acid. Terpenes were also identified in all the cheeses studied. A higher number of terpenes was identified in the flavoured cheeses than in the cheese without additives. For all flavoured cheeses: the higher the proportion of a particular aromatic plant in the cheese, the higher the number and proportion of terpenes, with the terpenes specific to a particular plant standing out.