VOLATILE COMPOUNDS IN AROMATISED OILS WITH BASIL, OREGANO AND THYME

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Abstract

During processing of aromatized rapeseed oils, volatiles from the added spices migrate in oil and give specific taste and aroma to oils, but there are no investigations about volatile compound migration in oil. The aim of this research was to determine volatile compounds in oils aromatized with basil, oregano, and thyme. In basil 43 volatile compounds, in oregano – 39, and in thyme – 37 volatile compounds were identified. In oil aromatized with basil – 8, in oil aromatized with oregano – 20, and in oil aromatized with thyme 11 volatile compounds were identified. From the total amount of identified compounds in spices, 23-30 % of volatiles found in thyme migrated in oil aromatized with thyme, from oregano in oil migrated 12-15% of volatiles, but from basil in oil aromatized with basil – only 5% of volatiles. More volatile compounds as camphene, α -pinene, and α -thujene migrated in oil better than less volatile compounds like methyl chavicol, and thymol. Migration of the same compound in oil from various spices differed. It could be explained by the location of the volatile compound in plant structure.

Key words: volatile compounds, basil, oregano, thyme, migration, aromatized oil.

Introduction

Basil, oregano and thyme are popular aromatic herbs growing in many regions of the world. Many authors reported the content of volatile compounds in basil (Lee et al., 2005; Barbieri et al., 2004; Diaz-Maroto et al., 2004; Pojjanapimol et al., 2004; Grayer 1996), oregano (Rodrigues et al., 2004; Juliani et al., 2002; Russo et al., 1998), and thyme (Kaloustian et al., 2005; Lee et al., 2005; Santos et al., 2005; Loziene et al., 2003; Pereira et al., 2000). A number of studies showed that basil, oregano and thyme are very variable in chemical composition of essential oils depending on geographical origin, vegetative stage, and used treatment. Spices and herbs are used to enhance the sensory features of food. Aromatization of oils could be one of the ways to expand use of the spices and vegetable oils and also to vary the assortment of oil products. During processing of aromatized rapeseed oils, volatiles from the added spices migrate in oil and give specific taste and aroma to oils.

For detection of volatile compounds in the spices and oils, different methods have been used, e.g. hydrodistillation (Kaloustian et al., 2005; Lee et al., 2005; Loziene et al., 2003), Soxlet extraction (Antonelli et al., 1998), simultaneous distillation-extraction (Barbieri et al., 2004; Diaz-Maroto et al., 2002), and supercritical fluid extraction (Rodrigues et al., 2004; Diaz-Maroto et al., 2002). But some compounds change during those types of extractions, for example, monoterpenes during hydrodistillation and removal of solvent (Diaz-Maroto et al., 2002; Yang et al., 1999). Headspace techniques could be a good option for qualitative analyses, but extraction yields are lower than those achieved using solid extraction. One of the headspace method used for extraction of volatiles in spices and oil is solid phase microextraction (Guillen et al., 2005; Kanavouras et al., 2005; Mildner-Szkudlarz et al., 2003; Jelen et al., 2000). This method was chosen for further research because it was possible to use the same method for spices and aromatized oils.

The aim of this research was to determine volatile compounds in oils aromatized with basil, oregano and thyme.

Materials and Methods

Dried basil, thyme and oregano were purchased from 'Santa Maria' (Tule, Estonia). Aromatized oils were prepared using refined rapeseed oil 'Risso'. Processing of aromatized oils is shown in Figure 1. Two concentrations of spices in oil were tested: 30 g kg⁻¹ and 60 g kg⁻¹.

Extraction of volatiles from spices and oils was made using solid phase microextraction (SPME). 1.2 g of basil and oregano, 1.5 g of thyme and 5 g of aromatised oils were weighed in 20 ml vials and capped with a septum (Gerstel). SPME extraction and injection were performed by an MP-2 autosampler (Gerstel). Divinylbenzene/Carboxen/ Polydimethylsiloxane (DVB/Car/PDMS) fiber (Supelco Inc., Bellefonte, PA, USA) was used for headspace SPME sampling. SPME parameters: incubation time - 2 min, extraction temperature – 30 °C, extraction duration – 60 min, rotating speed – 250 rpm, agitator on time (s) – 1, agitator off time (s) – 10, desorbtion – 5 min, 250 °C. For the analysis of the spices and aromatised oils, a Hewlett-Packard 6890 GC Plus coupled with a HP 5973 MSD (Mass Selective Detector-Quadrupole type) equipped with a CIS-4 PTV (Programmed Temperature Vaporization) Injector (Gerstel, Mulheim-an-der-Ruhr, Germany), and a EC5-MS capillary column (30 m x 0.25 mm i.d.; coating thickness 0.25 gm) was used. Working conditions were: injector - 250 °C; transfer line to MSD – 260 °C; oven temperature start – 35 °C, hold -5 min, programmed from 35 to 60 °C at 2 °C min⁻¹ and from



Figure.1. Processing of aromatised oils.

60 to 250 °C at 20 °C min⁻¹, hold 5 min; carrier gas (He) -1 ml min⁻¹; splitless; ionization El -70 eV. Acquisition parameters in full scan mode: scanned m/z 40-200 (0-20 min), 40-400 (>20 min).

Compounds were identified by comparison of their mass spectra with mass spectral libraries (Nist 98 and Wiley 6th), and by calculation of linear retention indexes and comparison with literature data (Adams, 1995). All analyses were made in triplicate. Compounds in tables are shown in the sequence of retention times.

Results and Disscusion

Volatile compounds of basil, oregano and thyme were determined. In the headspace of basil – 43, in oregano – 39, and in thyme 37 compounds were detected (Table 1). The major volatile compounds migrated from spices to aromatized oil: in oil with basil – 8, with oregano – 20, and with thyme 11 volatile compounds were detected (Table 2). Some spice compounds were not identified in aromatized oil, generally the ones forming less than 5% of the total spice volatile composition.

Table 1

Compound	LRI ^a	LRI	Composition (%) \pm SD			
Compound	calculated	literature ^b	Basil	Oregano	Thyme	
α-Thujene	925	931	0.12 ± 0.01	$\boldsymbol{1.38\pm0.08}$	0.61 ± 0.04	
α-pinene	932	939	0.40 ± 0.03	0.43 ± 0.01	$\boldsymbol{0.78 \pm 0.02}$	
Camphene	947	953	n.d.	n.d.	$\textbf{0.47} \pm \textbf{0.02}$	
Benzaldehyde	960	961	0.63 ± 0.02	n.d.	n.d.	
Sabinene	971	976	$\textbf{0.44} \pm \textbf{0.03}$	$\textbf{3.27} \pm \textbf{0.08}$	0.22 ± 0.02	
β-pinene	975	980	0.65 ± 0.04	0.35 ± 0.01	0.33 ± 0.02	
1-Octen-3-ol	979	978	n.d.	0.15 ± 0.01	0.99 ± 0.03	
3-Octanone	985	986	n.d.	n.d.	0.28 ± 0.02	
Myrcene	989	991	0.66 ± 0.05	$\textbf{2.69} \pm \textbf{0.05}$	$\textbf{2.88} \pm \textbf{0.25}$	
α-phellandrene	1005	1005	n.d.	$\boldsymbol{0.48 \pm 0.00}$	0.23 ± 0.01	
Δ -3-carene	1008	1011	0.31 ± 0.02	$\textbf{0.55} \pm \textbf{0.01}$	0.46 ± 0.03	
α-terpinene	1016	1018	n.d.	$\textbf{2.07} \pm \textbf{0.05}$	1.48 ± 0.09	
p-cymene	1024	1026	1.81 ± 0.17	19.62 ± 0.12	37.27 ± 1.34	
Limonene	1028	1031	1.64 ± 0.08	3.27 ± 0.08	3.05 ± 0.11	

Composition of the volatile compounds of spices

	1					
1,8-cineole	1031	1033	4.52 ± 0.18	$\textbf{2.05} \pm \textbf{0.04}$	0.95 ± 0.08	
Z-β-ocimene	1036	1040	n.d.	0.77 ± 0.02	n.d.	
E-β-ocimene	1047	1050	n.d.	0.16 ± 0.00	n.d.	
γ-terpinene	1058	1062	0.13 ± 0.02	$0.13 \pm 0.02 \qquad 4.51 \pm 0.09$		
cis-sabinene hydrate	1069	1068	0.35 ± 0.01	3.62 ± 0.01	0.89 ± 0.03	
cis-linalool oxide	1071	1074	0.56 ± 0.02 n.d.		n.d.	
trans-linalool oxide	1086	1088	0.50 ± 0.01	n.d.	n.d.	
Terpinolene	1087	1088	n.d.	$\boldsymbol{0.77 \pm 0.02}$	0.25 ± 0.01	
p-cymenene	1089	1089	n.d.	0.30 ± 0.00	0.37 ± 0.01	
trans-sabinene hydrate	1102	1097	n.d.	n.d.	5.07 ± 0.24	
Linalool	1102	1098	19.02 ± 0.35	$\textbf{7.29} \pm \textbf{0.14}$	n.d.	
trans-p-menth-2-en-1-ol	1124	1121	n.d.	0.93 ± 0.01	n.d.	
cis-p-menth-2-en-1-ol	1142	1140	n.d.	0.70 ± 0.01	n.d.	
Camphor	1146	1143	0.37 ± 0.01	n.d.	0.53 ± 0.01	
Borneol	1170	1165	0.25 ± 0.00	n.d.	0.82 ± 0.07	
terpin-4-ol	1180	1177	0.99 ± 0.02	$\textbf{8.13} \pm \textbf{0.05}$	1.3 ± 0.09	
α-terpineol	1194	1189	0.67 ± 0.00	1.94 ± 0.02	0.3 ± 0.02	
Methyl chavicol	1202	1195	43.11 ± 0.29	0.8 ± 0.08	0.78 ± 0.10	
trans-piperitol	1208	1205	n.d.	0.27 ± 0.00	n.d.	
Thymol, methyl ether	1231	1235	n.d.	4.66 ± 0.05	3.18 ± 0.09	
Carvacrol, methyl ether	1241	1244	n.d.	8.11 ± 0.12	2.96 ± 0.12	
Carvone	1245	1242	n.d.	0.08 ± 0.00	n.d.	
Linalool acetate	1251	1257	0.16 ± 0.00	10.63 ± 0.11	n.d.	
Z-anethole	1253	1251	0.42 ± 0.02	n.d.	n.d.	
Bornyl acetate	1285	1285	0.50 ± 0.01	n.d.	n.d.	
<i>E</i> -anethole	1287	1283	2.13 ± 0.16	0.31 ± 0.03	0.45 ± 0.02	
Thymol	1290	1290	0.49 ± 0.05	5.27 ± 0.48	21.07 ± 1.21	
Carvacrol	1299	1298	0.24 ± 0.02	0.51 ± 0.05	1.46 ± 0.10	
Z-Methyl cinnamate	1303	1301	1.96 ± 0.12	n.d.	n.d.	
Eugenol	1354	1356	1.93 ± 0.11	n.d.	n.d.	
α-copaene	1379	1376	0.34 ± 0.01	0.19 ± 0.01	0.24 ± 0.01	
<i>E</i> -Methyl cinnamate	1386	1379	4.72 ± 0.43	0.46 ± 0.03	0.36 ± 0.04	
β-elemene	1393	1391	0.50 ± 0.02	n.d.	n.d.	
Methyl Eugenol	1401	1401	0.40 ± 0.03	n.d.	n.d.	
<i>E</i> -caryophyllene	1423	1418	0.64 ± 0.00	2.27 ± 0.06	2.55 ± 0.06	
trans-α-bergamontene	1436	1436	5.23 ± 0.02	n.d.	n.d.	
α-guaiene	1439	1439	0.37 ± 0.01	n.d.	n.d.	
α-humulene	1459	1454	0.21 ± 0.00	0.19 ± 0.00	n.d.	
cis-β-guaiene	1492	1490	0.19 ± 0.01	n.d.	n.d.	
α-selinene	1498	1494	0.23 ± 0.01	n.d.	n.d.	
α-bulnesene	1505	1505	0.20 ± 0.02	n.d.	n.d.	
γ-cadinene	1517	1513	1.38 ± 0.04	n.d.	0.19 ± 0.02	
Δ - cadinene	1523	1524	n.d.	n.d.	0.26 ± 0.01	
cis-calamenene	1525	1521	0.31 ± 0.02	n.d.	n.d.	
Spathulenol	1563	1576	n.d.	0.28 ± 0.03	n.d.	
Caryophyllene oxide	1588	1581	n.d.	0.52 ± 0.03	0.19 ± 0.00	
Cadinol	1639	1640	0.30 ± 0.01	n.d.	n.d.	

Table 1 continuation

 $^{\mbox{\tiny \alpha}}$ – LRI – linear retention index

^b – Adams. 1995

n.d. - not detected

 \boldsymbol{bold} – volatile compounds identified in oil aromatized with correspondent spice

The main compounds in basil were methyl chavicol (43 %), followed by linalool (19%), trans- α -bergamontene (5.2%), *E*-Methyl cinnamate (4.7%), and 1.8-cineole (4.5%). Peak areas of other compounds were found in less than 2% of total area. Grayer et al. (1996) reported a similar content of basil, rich in linalool and methyl chavicol. In oil aromatized with basil, main compounds were linalool (35%), methyl chavicol (27%), and 1.8-cineole. The content of volatiles in basil and in oil aromatised with basil differ significantly. Percentage of methyl chavicol in basil was 17% higher than in oil aromatised with basil, whereas percentage of 1.8-cineole was 20% higher in aromatized oil. The major volatile compounds in oregano were p-cymene (19.6%) and trans-sabinene hydrate (12.8%), followed by linalool acetate (9.9%), carvacrol methyl ether, and terpin-4-ol (each 7.5%). In oil aromatised with oregano, p-cymene and trans-sabinene hydrate were the main volatile compounds too, but their percentage was higher than in oregano. The two main compounds of thyme were pcymene (36.2%) and thymol (20.5%), followed by γ terpinene (6.6%) and linalool (4.9%). Other compounds were found in less than 3% from the total. These data are similar to literature, and thyme analysed could belong to the p-cymene chemotype containing 32% of p-cymene, 21% of thymol, 9.5% of γ -terpinene, and 2.8% of linalool (Kaloustian, 2005). In oil aromatized with thyme, the main compounds were p-cymene (66%) and γ -terpinene (14.5%). Percentage of thymol in thyme was 20%, but in oil aromatised with thyme only – 2.5-3.6%. Percentages of volatile compounds in basil, oregano, thyme and aromatised oils made using these spices differ significantly (Tables 1, 2). It could be explained by the various migration levels of volatile compounds in oil.

The migration of each compound from spices in oil was expressed by calculating the percentage of volatile compound amount in the aromatized oil versus the volatile

Table 2

	Compos	ition of aromatised oils (%, from total volatile compounds)					
Compounds	Oil wit	h basil	Oil with	oregano	Oil with thyme		
	30 g kg^{-1}	60 g kg^{-1}	30 g kg^{-1}	$60 \mathrm{g \ kg}^{-1}$	30 g kg^{-1}	60 g kg^{-1}	
α-Thujene	n.d.	n.d.	4.43 ± 0.24	4.25 ± 0.05	0.99 ± 0.12	0.82 ± 0.05	
α-pinene	2.39 ± 0.18	2.42 ± 0.09	1.04 ± 0.15	1.00 ± 0.06	1.17 ± 0.04	1.23 ± 0.01	
camphene	n.d.	n.d.	n.d.	n.d.	1.02 ± 0.08	0.96 ± 0	
sabinene	1.55 ± 0.02	1.45 ± 0.03	6.85 ± 0.12	6.37 ± 0.24	n.d.	n.d.	
β-pinene	3.16 ± 0.05	3.05 ± 0.08	0.52 ± 0.09	0.55 ± 0.07	n.d.	n.d.	
Myrcene	n.d.	n.d.	1.61 ± 0.19	1.56 ± 0.10	3.11 ± 0.1	2.98 ± 0.29	
α -phellandrene	n.d.	n.d.	1.07 ± 0.16	0.94 ± 0.10	n.d.	n.d.	
Δ -3-carene	n.d.	n.d.	0.59 ± 0.08	0.62 ± 0.05	n.d.	n.d.	
α-terpinene	n.d.	n.d.	4.79 ± 0.09	4.37 ± 0.12	2.99 ± 0.04	2.69 ± 0.27	
p-cymene	2.97 ± 0.19	3.1 ± 0.14	32.29 ± 0.26	30.39 ± 0.50	66.06 ± 0.85	65.87 ± 3.08	
limonene	3.8 ± 0.24	3.6 ± 0.12	3.28 ± 0.65	3.30 ± 0.09	1.68 ± 0.1	1.85 ± 0.21	
1,8 - cineole	23.95 ± 0.2	21.83 ± 1.22	4.43 ± 0.04	6.55 ± 1.87	1.75 ± 0.19	1.78 ± 0.15	
γ-terpinene	n.d.	n.d.	8.38 ± 0.18	7.37 ± 0.15	14.47 ± 0.44	14.33 ± 1.23	
cis-sabinene							
hydrate	n.d.	n.d.	4.09 ± 0.21	4.38 ± 0.34	n.d.	n.d.	
terpinolene	n.d.	n.d.	1.07 ± 0.04	1.08 ± 0.05	n.d.	n.d.	
linalool	35.35 ± 0.76	35.09 ± 0.86	n.d.	n.d.	2.72 ± 0.1	2.59 ± 0.43	
trans-sabinene							
hydrate	n.d.	n.d.	17.99 ± 0.93	18.42 ± 1.05	n.d.	n.d.	
terpin-4-ol	n.d.	n.d.	4.58 ± 0.03	5.26 ± 0.10	n.d.	n.d.	
α-terpineol	n.d.	n.d.	0.52 ± 0.08	0.74 ± 0.02	n.d.	n.d.	
Methyl chavicol	26.85 ± 0.56	29.46 ± 0.32	n.d.	n.d.	n.d.	n.d.	
Thymol, methyl							
ether	n.d.	n.d.	0.55 ± 0.09	0.66 ± 0.02	n.d.	n.d.	
Carvacrol, methyl							
ether	n.d.	n.d.	1.03 ± 0.07	1.19 ± 0.03	n.d.	n.d.	
linalool acetate	n.d.	n.d.	0.90 ± 0.02	1.00 ± 0.01	n.d.	n.d.	
Thymol	n.d.	n.d.	n.d.	n.d.	2.56 ± 0.28	3.57 ± 0.52	

Composition of the volatile compounds of the aromatized oils

compound amount in an equal amount of dried spices necessary for aromatization (Table 3). From total volatile compounds identified in thyme, in oil migrated 30% (in oil with 30 g kg⁻¹ of thyme) and 23% (in oil with 60 g kg⁻¹ of thyme) of volatiles. In oil with basil, only 5% from the total amount of identified volatiles migrated in oil. It was due to low migration of the main basil volatiles - metilchavicol and linalool in oil (Table 3). From oregano in oil migrated 15 % (in oil with 30 g kg⁻¹ of oregano) and 12% (in oil with 60 g kg⁻¹ of oregano) of volatile compounds. In the tables the identified compounds are presented according to their sequence of eluation. The more volatile compounds eluate first, then follow less volatile compounds. The more volatile compounds – α -thujene α -pinene, camphene – migrated in oil better, whereas migration of less volatile compounds as methyl chavicol and thymol was very low (3-5%).

Migration of volatile compounds depended on the type of spice, for example, from thyme 40% of myrcene migrated in oil, whereas from oregano only 9% of that compound migrated in oil. It could be explained by the location of the volatile compound in plant structure that could accelerate or reduce migration of compounds in oil. The migration rate of volatile compounds from oregano and thyme in oil was lower with a higher spice additive. But the migration rate of basil volatile compounds did not change with the concentration.

Conclusions

1. In oil aromatized with basil - 8, in oil aromatized with oregano - 20, in oil aromatized with thyme 11 volatile compounds were identified.

2. From the total amount of identified compounds in spices, 23-30 % of volatiles found in thyme migrated in oil aromatized with thyme, from oregano in oil migrated 12-15% of volatiles, but from basil in oil aromatized with basil only 5% of volatiles.

3. More volatile compounds as camphene, α -pinene, and α -thujene migrated in oil better than less volatile compounds like methyl chavicol, and thymol.

4. Migration of the same compound in oil from various spices differed. It could be explained by the location of the volatile compound in plant structure.

Table 3

	% of compounds migration from spices in oil					
Compounds	Oil with basil		Oil with	oregano	Oil with thyme	
	30 g kg^{-1}	60 g kg ⁻¹	30 g kg^{-1}	60 g kg^{-1}	30 g kg^{-1}	60 g kg ⁻¹
α-Thujene	n.d.	n.d.	48	38	60	39
α-pinene	30	32	36	29	56	46
camphene	n.d.	n.d.	n.d.	n.d.	81	61
sabinene	18	17	31	24	n.d.	n.d.
β-pinene	25	25	21	19	n.d.	n.d.
Myrcene	n.d.	n.d.	9	7	40	30
α-phellandrene	n.d.	n.d.	32	24	n.d.	n.d.
∆-3-carene	n.d.	n.d.	16	14	n.d.	n.d.
α-terpinene	n.d.	n.d.	34	26	75	53
p-cymene	8	9	25	19	66	52
limonene	12	12	14	13	21	18
1,8 cineole	27	25	32	39	69	55
γ-terpinene	n.d.	n.d.	28	20	80	62
cis-sabinene hydrate	n.d.	n.d.	17	15	n.d.	n.d.
terpinolene	n.d.	n.d.	21	17	n.d.	n.d.
linalool	10	10	n.d.	n.d.	20	15
trans-sabinene						
hydrate	n.d.	n.d.	20	17	n.d.	n.d.
terpin-4-ol	n.d.	n.d.	8	8	n.d.	n.d.
α-terpineol	n.d.	n.d.	4	5	n.d.	n.d.
Methyl chavicol	72	81	n.d.	n.d.	n.d.	n.d.
Thymol, methyl ether	n.d.	n.d.	2	2	n.d.	n.d.
Carvacrol, methyl ether	n.d.	n.d.	2	2	n.d.	n.d.
linalool acetate	n.d.	n.d.	1	1	n.d.	n.d.
Thymol	n.d.	n.d.	n.d.	n.d.	5	5

Percentage of compounds migration from spices in oil

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