J. Essent. Oil Res., **11**, 482-488 (Jul/Aug 1999)

Essential Oil Composition of *Citrus sinensis* (L.) Osbeck cv. Maltese

Alessandra Trozzi
Facoltà di Farmacia, Università di Catanzaro, Catanzaro, Italy
Antonella Verzera*
Instituto di Industrie Agrarie, Facoltà di Agraria, Università di Catania, Catania, Italy
Giuseppe Lamonica
Dipartimento Farmaco-chimico, Facoltà di Farmacia, Università di Messina, Messina, Italy

Abstract

The composition of the volatile fraction and of the non-volatile residue of the Maltese sweet orange oil (laboratory-prepared) was studied by GC, GC/MS and HPLC. Sixty-two components were identified in the volatile fraction. The main component was limonene (92,6%); moreover, a high content of carbonyl compounds was also found. Six polymethoxylated flavones were identified in the non-volatile residue: 5,6,7,8,4'-pentamethoxyflavone (tangeretin), 3,5,6,7,8,3',4'-heptamethoxyflavone, 5,6,7,8,3',4'-hexamethoxyflavone (nobiletin), 5,6,7,4'-tetramethoxyflavone (tetra-O-methylscutellarein), 3,5,6,7,3',4'-hexamethoxyflavone and 5,6,7,3'4'-pentamethoxyflavone (sinensetin). The Maltese oil composition was compared to that of different cultivars of sweet orange previously analyzed.

Key Word Index

Citrus sinensis cv. Maltese, Rutaceae, sweet orange, essential oil composition, polymethoxylated flavones, GC, GC/MS, HPLC.

Introduction

In Italy, sweet orange (*Citrus sinensis* L. Osbeck) cultivars are distinguished as "blond" and "blood," as previously described (1).

There are some blond cultivars characterized by a sweet pulp (without acids) which can be related to orange "Douceatre" (France), to "Sucrena" or "Canamiel" or "Grana de Oro" or "Imperial" (Spain), to "Meski" (North Africa and Middle East), to "Moghrabi" (Middle East), to "Cok Kum" or "Tounisi" (Turkey), to "Sucari" (Egypt), to "Orange the Nice" (France, Cote d'Azur), to "Lima" or "Piralima" (Brazil), to "Mosambi" (India) (2,3). In Italy, these oranges are known as "Maltese" or "Vaniglia;" they ripen from October to February and they are reserved exclusively for the fresh fruit market. Maltese orange fruit has a sweetish taste, neither sour, nor pungent. The production is very limited and it decreases continuously, because of the characteristic taste of the fruit, it is less requested on the market. The size of the fruits ranges from medium to large (180-250 g), the form from spherical to ovoid, the rind is rough and very thick. At the time of ripeness the color is dark orange.

Most of the papers pertinent to sweet orange oil composition were reviewed by Lawrence (4). A large

*Address for correspondence

Received: March 1998
Revised: June 1998
Accepted: July 1998

1041-2905/99/0004-0482$04.00/0

Table I. Samples analyzed

Sample no.	Harvest period	Ripeness	Fruit color
1	10/28/96	unripe	yellow-green
2	10/30/96	"	yellow-green
3	11/04/96	"	yellow-green
4	11/10/96	"	yellow-green
5	12/02/96	not very ripe	yellow
6	12/09/96	not very ripe	yellow
7	01/13/97	ripe	yellow-orange
8	01/17/97	ripe	yellow-orange
9	02/02/97	ripe	yellow-orange

number of papers deal with the composition of the volatile fraction of sweet orange oil and in many of them its differences in relation to the cultivars are reported with particular references to the different content of aliphatic aldehydes and linalool (5-16). Few papers are, besides, pertinent to the non-volatile residue (17-20).

In the literature, no information could be found about "Maltese" sweet orange oil composition. Only one paper deals with the composition of "Piralima" oil, a cultivar from Brazil (21).

In previous papers, we analyzed the volatile fraction and non-volatile residue composition of industrial Pelatrice and FMC sweet orange oils (22-23) and of laboratory-prepared Italian sweet orange oils from different cultivars: Biondo comune, Navelina, Washington navel, Valencia late, Ovale, Tarocco, Moro, Sanguinello (24).

In this paper, following our research on the composition of Italian sweet orange oils, we analyzed the composition of Maltese sweet orange oils. This research focuses on the identification of new flavors that could have use in the perfumery and food industries.

Experimental

The research was carried out on nine samples of laboratory prepared Maltese orange oils. The fresh fruits were collected near Taurianova (Reggio Calabria, Calabria), during the period October 1996-February 1997. Information about the samples analyzed are reported in Table I. The fruits were cold-pressed by applying a manual pressure on the rind to release the oil which was collected on a watch glass, transferred to a test tube, centrifuged and analyzed.

GC: Volatile fraction was analyzed by GC on a Carlo Erba Gas Chromatograph 5160 Mega series, equipped with a Shimadzu data processor C-R3A using the following, previously described (25), experimental conditions: fused silica SE-52 column, 30 m x 0.32 mm; film thickness, 0.40-0.45 μm [Mega, Legnano (MI), Italy]; column temperature, 45°C (6 min) to 180°C at 3°C/min; injection mode, split; detector, FID; injector and detector temperature, 280°C; carrier gas, He 95 kPa; injected volume, 1 μL of neat oil.

GC/MS: Samples were analyzed by GC/MS (EI) on a Fisons MD 800 (Milan, Italy) system coupled with Adams' library (26) and FFC banks (27); GC conditions were: a DB-5 fused silica column, 30 m x 0.25 mm, film thickness, 0.25 μm; column temperature, 60°C (6 min) to 240°C at 3°C/min; injector temperature, 250°C; injection mode, split; split ratio, 1:30; volume injected, 1 μL of a solution 1/100 in pentane of the oil; carrier gas, He 61.6 kPa; linear velocity 33.5 mL/min; interface temperature, 250°C; detector 1.5 kV; acquisition mass range, 41-300 amu; solvent cut, 2 min.

HPLC: All samples were analyzed by normal-phase HPLC, using a Waters Associates (W.A.) equipment composed of a model 519 pump; a 600 E gradient controller, a Rheodyne 9125 injector and a photo diode array detector model 996. Peak integration and quantitative calculations were performed by Millenium 2010 (W.A.) system using a calibration curve obtained for each standard component against a coumarin standard (23). The column was a Zorbax silica column (25 cm x 4.6 mm, particle size

Table II. Percentage composition of single components and of classes of substances for the Maltese sweet orange oils analyzed

No.	Components	10/28/96	10/30/96	11/04/96	11/10/96	12/02/96	12/09/96	01/13/97	01/17/97	02/02/97	$\bar{X}$	s
1.	α-thujene	0.02	0.01	0.03	0.03	0.01	0.01	0.01	0.01	0.01	0.01	0.001
2.	α-pinene	0.49	0.58	0.59	0.61	0.52	0.51	0.49	0.52	0.69	0.55	0.067
3.	camphene	0.01	t	0.01	0.01	t	t	t	t	t	t	0.002
4. 5.	sabinene β-pinene }	1.44	1.69	2.95	3.32	1.66	1.46	1.56	1.63	1.63	1.93	0.696
6.	myrcene	1.94	2.10	2.06	2.21	1.88	1.87	1.83	1.87	1.83	1.95	0.136
7.	octanal	0.69	0.74	0.81	0.72	0.84	0.79	0.64	0.66	0.23	0.68	0.183
8.	α-phellandrene	0.08	0.08	0.09	0.08	0.09	0.09	0.07	0.07	0.03	0.08	0.020
9.	δ-3-carene	0.03	0.03	0.02	0.03	0.03	0.02	0.04	0.04	0.03	0.03	0.006
10.	α-terpinene	0.03	t	0.02	0.02	t	t	t	t	t	0.01	0.012
11.	p-cymene	0.01	t	t	t	t	t	t	t	t	t	-
12.	limonene	93.12	92.64	91.05	90.06	92.68	93.15	93.42	93.29	93.74	92.57	1.287
13.	(Z)-β-ocimene	0.01	t	0.01	0.01	t	t	t	t	t	t	-
14.	(E)-β-ocimene	0.05	0.05	0.09	0.09	0.08	0.08	0.06	0.03	0.04	0.06	0.002
15.	γ-terpinene	0.04	0.02	0.02	0.03	t	t	t	t	t	0.01	0.014
16.	cis-sabinene hydrate	0.04	0.05	0.08	0.08	0.04	0.04	0.04	0.04	0.02	0.05	0.020
17.	octanol	t	t	0.02	0.01	0.01	0.01	0.01	0.01	t	0.01	0.006
18.	terpinolene	0.07	0.01	0.03	0.03	0.01	t	0.01	0.01	0.01	0.02	0.020
19.	trans-sabinene hydrate	t	t	0.01	0.01	t	t	t	t	t	t	-
20.	linalool	0.39	0.38	0.45	0.70	0.70	0.58	0.40	0.39	0.21	0.46	0.164
21.	nonanal	0.11	0.11	0.10	0.11	0.12	0.11	0.09	0.09	0.06	0.10	0.019
22.	heptyl acetate	t	t	0.01	0.02	0.01	t	t	t	0.01	0.01	0.006
23.	cis-limonene oxide	t	t	0.01	0.03	t	t	0.01	0.01	0.02	0.01	0.001
24.	trans-limonene oxide	0.01	t	0.01	0.02	0.01	0.01	0.01	0.01	0.03	0.01	0.009
25.	citronellal	0.03	0.03	0.03	0.04	0.03	0.02	0.04	0.04	0.04	0.03	0.001
26.	terpinen-4-ol	0.01	t	0.01	0.02	t	t	t	t	t	0.01	0.004
27.	α-terpineol	0.10	0.10	0.13	0.13	0.09	0.08	0.08	0.08	0.04	0.09	0.029
28.	decanal	0.36	0.35	0.35	0.37	0.43	0.39	0.40	0.39	0.29	0.37	0.039
29.	octyl acetate	t	t	0.01	t	t	t	t	t	t	0.01	0.001
30.	cis-carveol	t	t	0.02	0.02	0.01	0.01	0.02	t	0.01	0.01	0.007
31.	nerol	0.01	0.01	0.02	0.03	t	0.01	0.01	0.01	0.03	0.02	0.011
32.	neral	0.15	0.15	0.14	0.17	0.12	0.11	0.11	0.11	0.07	0.13	0.029
33.	carvone	t	t	t	t	0.01	t	t	t	t	t	-
34.	geraniol	t	t	0.01	t	t	t	0.01	0.01	0.01	0.01	0.001
35.	(E)-2-decenal	t	t	t	t	t	t	t	t	t	t	-
36.	geranial	0.25	0.23	0.22	0.24	0.19	0.17	0.18	0.17	0.10	0.19	0.046

Table II. Continued

No.	Components	10/28/96	10/30/96	11/04/96	11/10/96	12/02/96	12/09/96	01/13/97	01/17/97	02/02/97	$\bar{X}$	s
37.	bornyl acetate	0.01	0.01	0.01	0.01	0.02	0.01	0.02	0.02	0.01	0.01	0.004
38.	undecanal	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.01	0.01	0.02	0.002
39.	nonyl acetate	t	t	0.01	t	t	t	t	t	t	0.01	-
40.	α-terpinyl acetate	t	t	t	t	t	t	t	t	t	t	-
41.	citronellyl acetate	t	t	t	t	t	t	0.01	t	t	t	-
42.	neryl acetate	0.01	0.01	0.01	0.01	t	0.01	0.01	0.01	0.01	0.01	0.001
43.	α-copaene	0.03	0.03	0.02	0.02	0.02	0.02	0.02	0.02	0.03	0.02	0.001
44.	geranyl acetate	0.01	0.01	0.01	0.01	t	t	0.01	0.01	0.01	0.01	0.001
45.	β-cubebene + β-elemene	0.03	0.03	0.02	0.02	0.02	0.02	0.02	0.02	0.03	0.02	0.001
46.	dodecanal	0.05	0.05	0.04	0.06	0.05	0.05	0.07	0.05	0.06	0.05	0.007
47.	β-caryophyllene	t	t	t	0.01	0.01	0.01	0.01	0.01	0.02	0.01	0.005
48.	β-gurjunene	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	t	0.01	0.003
49.	α-humulene	0.01	0.01	0.03	0.01	t	t	t	t	0.01	0.01	0.008
50.	(E)-β-farnesene	0.03	0.03	0.04	0.06	0.02	0.02	0.03	0.02	0.02	0.03	0.014
51.	germacrene D	0.02	0.02	0.02	0.02	t	t	0.01	0.01	0.02	0.01	0.007
52.	valencene	0.01	0.01	0.01	0.01	0.02	0.02	0.03	0.02	0.08	0.02	0.024
53.	bicyclogermacrene	t	t	t		0.01	0.01	0.01	t	0.01	0.01	0.004
54.	(E,E)- α-farnesene	0.03	0.03	0.06	0.07	0.03	0.02	0.03	0.02	0.02	0.03	0.016
55.	tridecanal	t	t	t	t	t	t	t	t	0.01	t	-
56.	δ-cadinene	0.04	0.04	0.02	0.04	0.03	0.03	0.03	0.02	0.03	0.03	0.006
57.	(E)-nerolidol	t	t	t	t	t	t	t	t	t	t	-
58.	tetradecanal	t	t	t	t	t	t	t	t	t	t	-
59.	β-sinensal	0.06	0.06	0.05	0.08	0.04	0.04	0.05	0.04	0.03	0.05	0.016
60.	α-sinensal	0.07	0.08	0.08	0.12	0.05	0.05	0.05	0.04	0.04	0.06	0.025
61.	nootkatone	t	t	0.01	0.01	t	t	0.01	t	0.01	0.01	0.002
	Hydrocarbons	97.50	97.40	97.18	96.80	97.14	97.36	97.69	97.64	98.27	97.44	0.41
	Monoterpenes	97.30	97.21	96.95	96.53	96.97	97.19	97.49	97.47	98.01	97.24	0.41
	Sesquiterpenes	0.20	0.20	0.22	0.27	0.17	0.16	0.20	0.17	0.26	0.21	0.04
	Oxygenated compounds	2.41	2.40	2.65	3.04	2.79	2.52	2.26	2.19	1.35	2.40	0.48
	Carbonyl compounds	1.81	1.82	1.84	1.94	1.89	1.75	1.64	1.60	0.94	1.69	0.30
	Alcohols	0.56	0.55	0.74	1.01	0.86	0.72	0.56	0.53	0.31	0.65	0.20
	Esters	0.03	0.03	0.05	0.06	0.04	0.03	0.05	0.04	0.04	0.04	0.01
	Aliphatic aldehydes	1.24	1.27	1.32	1.28	1.46	1.36	1.21	1.19	0.65	1.22	0.23
	Terpene aldehydes	0.56	0.55	0.51	0.65	0.42	0.39	0.43	0.41	0.27	0.46	0.11

7 μm); mobile phase, hexane: ethyl alcohol, 95:5; flow rate, 1.6 mL/min; injection volume, 20 μL of a solution obtained by diluting about 50 mg of each oil and 0.1 mL of a coumarin solution of known concentration in 1 mL of hexane: ethyl acetate (75:25). Detection was by UV absorbance at 315 nm. The UV spectra of eluting peaks were monitored with the PDA detector in the region 200-400 nm.

Results and Discussion

Volatile Fraction Composition: Sixty-two components were identified in each oil. For each sample we calculated the quantitative composition as a relative percentage of the peak area, as well as, the total amount of hydrocarbons, monoterpenes, sesquiterpenes, oxygenated compounds, alcohols, esters, carbonyl compounds, aliphatic aldehydes and terpene aldehydes. The average percentage ($\bar{X}$) and the standard deviation (s) of the single components and of classes of substances were also calculated. These data are reported in Table II. With respect to a previous research on sweet orange oil (24) the following additional components were identified: β-gurjunene and bicyclogermacrene. On the contrary, α-muurolene and γ-muurolene were not identified.

All samples analyzed showed similar composition characteristics. The most representative class of substances was that of monoterpenes. The oils revealed a high content of limonene ($\bar{X}$ = 92.57%) which is typical for the orange oils; for other monoterpenes only sabinene, β-pinene and myrcene showed a percentage higher than 1%. Among the oxygenated compounds, the most representative classes were the carbonyl compounds ($\bar{X}$ = 1.69%) and alcohols ($\bar{X}$=0.65%). For alcohols, the main component was linalool ($\bar{X}$ = 0.46%), while octanal, nonanal and decanal represented more than 70% of carbonyl compound fraction with the most representative monoterpene aldehydes being neral (0.13%) and geranial (0.19%). The content of α-sinensal ($\bar{X}$ = 0.06%) was always higher than β-sinensal ($\bar{X}$ = 0.05%). Moreover, this oil was characterized, as the other cultivars of sweet orange, by the presence of the δ-3-carene ($\bar{X}$ = 0.03%), by a large number of sesquiterpenes and by a low content of esters ($\bar{X}$ = 0.04%).

Variation in the Composition of the Volatile Fraction during the Productive Season: During the productive season, quantitative differences in the composition of the oils were observed.

Monoterpenes showed the highest values in the final period of the production season. Limonene showed a similar behavior. Carbonyl compounds showed a slight decrease during the production season, from October to January, and a clear reduction in February. Alcohols reached the highest values in November, then decreased, showing the lowest values at the end of the production season. Esters and sesquiterpenes also showed the highest values in November even if the variations during the production season were not very large.

Comparison between the Volatile Fraction of the cv. Maltese Oil and "Blond" and "Blood" Cultivar Oils of Sweet Orange: Table III is compared to the average percentage composition in classes of substances of Maltese sweet orange oils to that of oils obtained from the previously analyzed cultivars: Moro, Tarocco, Sanguinello (blood oranges), Navelina, Washington Navel, Biondo comune, Ovale, Valencia late (blond oranges) (24).

The monoterpene content in the Maltese sweet orange oil was similar to that of oils obtained from Biondo comune, Navelina, Ovale, Valencia late, while the sesquiterpene content was similar to that found in Ovale and Valencia sweet orange oils. The aliphatic and terpene aldehyde content was clearly higher than in other sweet orange cultivars, this was due to the content of each single aldehyde. For example, the Maltese sweet orange oil showed an average percentage of octanal equal to 0.68%; while the octanal highest content in oils of the other cultivars of sweet orange oils was 0.52% (Navelina oil) (24). Neral and geranial, together, never exceeded 0.2% in the sweet orange cultivar oils, previously analyzed (24). In Maltese sweet orange oil they reached 0.41% in November and had an average percentage composition of 0.32%.

The high content of neral and geranial is probably a characteristic of oils obtained from cultivars with sweet pulp without acids since a similar behavior is shown by the sweet orange "Piralima" oil from Brazil (21). Piralima, in fact, possessed a higher content of carbonyl compounds, in particular of neral

Table III. Composition in classes of substances for Maltese "blond" and "blood" cultivars of sweet orange oils

	Maltese $\bar{X}$	Biondo comune $\bar{X}$	Navelina $\bar{X}$	Ovale $\bar{X}$	Valencia late $\bar{X}$	Washington navel $\bar{X}$	Moro $\bar{X}$	Sanguinello $\bar{X}$	Tarocco $\bar{X}$
Hydrocarbons	97.44	97.72	97.77	97.70	97.60	98.39	98.42	98.20	98.62
Monoterpenes	97.24	97.46	97.52	97.50	97.41	98.06	98.03	97.69	98.37
Sesquiterpenes	0.21	0.26	0.25	0.20	0.19	0.32	0.38	0.51	0.25
Oxygenated compounds	2.40	2.04	2.06	2.05	2.08	1.37	1.43	1.58	1.20
Carbonyl compounds	1.69	1.15	1.46	0.96	1.32	0.85	0.65	0.80	0.62
Alcohols	0.65	0.81	0.53	0.99	0.66	0.43	0.70	0.67	0.51
Esters	0.04	0.04	0.06	0.08	0.07	0.07	0.06	0.08	0.05
Aliphatic aldehydes	1.22	0.79	1.09	0.63	0.95	0.61	0.38	0.54	0.42
Terpene aldehydes	0.46	0.36	0.35	0.31	0.36	0.22	0.25	0.23	0.19

Table IV. Content (mg/100 g) of polymethoxylated flavones in Maltese sweet orange oil and in industrial Italian sweet orange oils

No.	Components	Maltese sweet orange oils 10/28/96	10/30/96	11/4/96	11/10/96	12/2/96	12/9/96	1/13/97	1/17/97	2/2/97	$\bar{X}$	Industrial sweet orange oils $\bar{X}$
1.	Tangeretin	83	90	92	109	95	83	98	129	71	94	48
2.	3, 5,6,7,8,3',4'-Heptamethoxyflavone	79	81	64	86	106	92	100	125	52	87	84
3.	Nobiletin	77	78	85	86	77	80	86	107	56	81	52
4.	tetra-*O*-Methylscutellarein	58	60	62	68	61	52	58	70	32	58	31
5.	5,6,7,8,3'4'-Hexamethoxyflavone	14	12	9	16	12	11	12	13	7	12	13
6.	Sinensetin	8	8	7	11	t	5	6	6	4	7	9

(0.17-0.34%) and geranial (0.13-0.25%) than other cultivar oils of sweet orange produced in Brazil.

Alcohols, in Maltese oil, were similar to those found in oils of Valencia late, Moro and Sanguinello. In conclusion, the Maltese sweet orange oil can be distinguished from other cultivars oil by its high content of carbonyl compounds.

Non-Volatile Residue: In the Maltese sweet orange oils analyzed, six polymethoxylated flavones were identified and quantified: tangeretin, 3,5,6,7,8,3',4'-heptamethoxyflavone, nobiletin, tetra-*O*-methylscutellarein, 3,5,6,7,3',4'-hexamethoxyflavone and sinensetin. Table IV reports the content of each polymethoxyflavone in Maltese sweet orange oils; the same table compares these results with those of industrial Italian sweet orange oils, previously analyzed (23). As can be seen tangeretin, 3,5,6,7,8,3',4'-heptamethoxyflavone and nobiletin showed similar average values for the Maltese oils. From a comparison of these results with those of the industrial sweet orange oils (23), it can be seen that Maltese sweet orange oil possesses slightly higher levels of tangeretin, nobiletin and tetra-*O*-methylscutellarein, while the values of other polymethoxylated flavones were similar to sweet orange oils encountered in industry.

References

1. A. Di Giacomo and B. Mincione, *Gli olii essenziali agrumari in Italia*. Laruffa Editore, Reggio Calabria (1994).
2. G. Cutuli, E. di Marino, V. Lo Giudice, L. Pennisi, G. Raciti, F. Russo, A. Scuderi and P. Spina, *Trattato di agrumicoltura*. Ed. Agricole, Bologna (1985).
3. J. C. Praloran, *Les Agrumes*. G. P. Maisonneur E Larose, Paris (1971).

4. B. M. Lawrence, *Progress in essential oils.* Perfum. Flavor., **1**(1), 1-5 (1976); **4**(6), 31-36 (1980); **9**(4), 37-48 (1984); **9**(6), 61-71 (1985); **12**(3), 58-70 (1987); **15**(6), 45-64 (1990); **17**(5), 131-146 (1992); **19**(4), 35-37 (1994).
5. A. Lifshitz, W. L. Stanley and Y. Stepak, *Composition of Valencia essenzial oil from California, Florida and Israel.* J. Food Sci., **35**, 547-548 (1970).
6. R. Huet and M. C. Murail, *L'huile essentielle d'orange de type "Guinée."* Fruits, **27**, 297-301 (1972).
7. P. E. Shaw and R. L. Coleman, *Quantitative composition of cold-pressed orange oils.* J. Agric. Food Chem., **22**, 785-787 (1974).
8. J. W. Kesterson and R. J. Braddock, *Total peel oil content of the major Florida citrus cultivars.* J. Food Sci., **40**, 931-933 (1975).
9. C. W. Wilson and P. E. Shaw, *Quantitation of individual and total aldehydes in citrus cold-pressed oils by fused silica capillary gas-cromatography.* J. Agric. Food Chem., **32**, 399-401 (1984).
10. H. Boelens and R. Jimenez, *The chemical composition of some Mediterraneam Citrus oils.* J. Essent. Oil Res., **1**, 151-159 (1989).
11. M. Ziegler, H. Brandauer, E. Ziegler and G. Ziegler, *A different aging model for orange oil: deterioration products.* J. Essent. Oil Res., **3**, 209-220 (1991).
12. H. Sugisawa, M. Takeda, R. Hua Yang and N. Takagi, *The comparison of odor quality of volatiles in peel oils of five kinds of Navel oranges.* Nippon Shokuhin Kogyo Gakkaishi, **38**, 668-674 (1991).
13. M. Usai, G. Arras and F. Fronteddu, *Effect of cold storage on essential oil of peel of Thompson Navel Orange.* J. Agric. Food Chem., **40**, 271-275 (1992).
14. A. Baaliouamer and B.-Y. Meklati, *The chemical composition of some cold-pressed Citrus oils produced in Algeria.* J. Essent. Oil Res., **4**, 251-258 (1992).
15. C. Blanco Tirado, E. E. Stashenko, M. Y. Combariza and J. R. Martinez, *Comparative study of Colombian citrus oils by high-resolution gas chromatography and gas chromatography-mass spectrometry.* J. Chromatogr., A, **697**, 501-513 (1995).
16. L. R. Rouseff, J. W. Grosser, H. E. Nordby and H. D. Powell, *Comparison of flavor volatiles in a somatic hybrid from West Indian Lime (Citrus aurantifolia) and Valencia orange (C. sinensis) with its parents.* Spec. Publ. R. Soc. Chem., **197**, 78-81 (1996).
17. J. P. Bianchini and E. M. Gaydou, *Role of water in qualitative and quantitative determination of polymethoxylated flavones by straight-phase high-performance liquid chromatography: application to orange peel oils.* J. Chromatogr., **211**, 61-78 (1981).
18. E. M. Gaydou, J. P. Bianchini and R. P. Randriamiharisoa, *Orange and mandarin peel oils differentiation using polymethoxylated flavone composition.* J. Agric. Food Chem., **35**, 525-529 (1987).
19. D. McHale and J. B. Sheridan, *The oxygen heterocyclic compounds of Citrus peel oils.* J. Essent. Oil Res., **1**, 139-149 (1989).
20. E. M. Gaydou, T. Berahia, J. C. Wallet and J. P. Bianchini, *Gas chromatography of some polymethoxylated flavones and their determination in orange peel oils.* J. Chromatogr., **549**, 440-445 (1991).
21. M. Koketsu, M. T. Magalhaes, U. C. Wilberg and M. G. R. Donalisio, *Oleos essenciais de frutos citricos cultivados no Brazil.* Boletin de Pesquita, **7**, 3-21 (1983).
22. G. Dugo, A. Verzera, I. Stagno d'Alcontres, A. Cotroneo, A. Trozzi and L. Mondello, *On the genuineness of Citrus essential oils. Part XLIII. The composition of the volatile fraction of italian sweet orange oils.* J. Essent. Oil Res., **6**, 101-137 (1994).
23. P. Dugo, L. Mondello, E. Cogliandro, I. Stagno d'Alcontres and A. Cotroneo, *On the genuineness of citrus essential oils. Part XLVI. Polymethoxylated flavones of the non volatile residue of italian sweet orange and mandarin essential* oils. Flav. Fragr. J., **9**, 105-111 (1994).
24. A. Verzera, A. Trozzi, I. Stagno d'Alcontres and A. Cotroneo, *On the genuineness of citrus essential oils. Part XLVIII. The composition of the volatile fraction of some varieties of sweet orange oils.* J. Essent. Oil Res., **8**, 159-170 (1996).
25. A. Verzera, L. Mondello, A. Trozzi and P. Dugo, *On the genuineness of citrus essential oils. Part LII. Chemical characterization of essential oil of three cultivars of Citrus clementine Hort.* Flav. Fragr. J., **12**, 163-172 (1997).
26. R. Adams, *Identification of essential oil components by gas chromatography/mass spectroscopy.* Allured Publ. Corp., Carol Stream, IL (1995).
27. L. Mondello, P. Dugo, A. Basile, G. Dugo and K. D. Bartle, *Interactive use of linear retention indices, on polar and apolar columns, with a MS-library for reliable identification of complex mixture.* J. Microcol. Sep., **7**, 581-591 (1995).