PAPER

EXTRACTION TECHNOLOGY AND LEMON OIL COMPOSITION

LA TECNOLOGIA DI ESTRAZIONE E LA COMPOSIZIONE DELL'OLIO DI LIMONE

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ABSTRACT

The influence of the extraction technology on cold-pressed lemon essential oil composition was studied. The volatile and oxygen heterocyclic fractions of cold-pressed lemon essential oils were studied by HRGC, HRGC/MS and normal-phase HPLC. The genuine lemon oils were obtained in the 1997/98 season using the following industrial machines: "Pelatrice", "Sfumatrice", "FMC" and "Torchi". Sixty-four compounds were identified and quantified. Limonene was the main compound. In the oxygen heterocyclic fraction two coumarins (5-geranyloxy-

RIASSUNTO

È stata studiata l'influenza della tecnologia di estrazione sulla composizione dell'olio essenziale di limone. La composizione della frazione volatile e la frazione dei composti eterociclici ossigenati sono state analizzate mediante HRGC, HRGC/MS e HPLC in fase normale. Gli oli essenziali analizzati sono stati ottenuti nella stagione produttiva 1997/1998 usando le seguenti tecnologie industriali: "Sfumatrice", "Pelatrice", "FMC" e "Torchi". Nella frazione volatile sono stati identificati e quantizzati 64 componenti. Il componente principale è il limonene. Nella frazione

- Key words: *Citrus limon* (L.) Burm., coumarins, HRGC, HRGC/MS, HPLC, lemon oil, psoralens, volatile fraction -

7-methoxycoumarin, citropten) and five psoralens (bergamottin, 8-geranyloxypsoralen, oxypeucedanin, byakangelicol, 5-isopent-2'-enyloxy-8-(2',3'-epoxyisopentyloxy)psoralen were identified and quantified. Bergamottin and 5-geranyloxy-7-methoxycoumarin were the main compounds. It was found that the technology does not influence the qualitative composition of the oil but there were quantitative differences. "FMC" produced an oil with olfactory notes and a quantitative composition similar to "Sfumatrice" oil, considered the best quality oil. "Pelatrice" oils gave the highest amount of volatile oxygenated compounds as well as coumarins and psoralens.

degli eterocicli ossigenati sono stati identificati e quantizzati due cumarine (5-geranilossi-7-metossicumarina, citroptene) e cinque psoraleni (bergamottina, 8-geranilossipsoralene, 5-isopent-2'-enilossi-8-(2',3',-epossiisopentilossi)-psoralene, ossipeuce- danina, biacangelicolo). I componenti principali sono bergamottina e 5-geranilossi-7-metossicumarina. L'influenza della tecnologia di estrazione sulla composizione dell'olio è legata a variazioni quantitative che riguardano sia la frazione volatile che il residuo non volatile. In particolare, gli oli ottenuti con macchine FMC mostrano una composizione quantitativa simile a quelli prodotti con macchine "Sfumatrice", considerati gli oli di migliore qualità. Gli oli ottenuti con macchine "Pelatrice" presentano il maggior contenuto di composti ossigenati, citral, cumarine e psoraleni.

INTRODUCTION

Sicilian lemon oil is considered a very high quality product especially for its high content of citral which characterises its olfactory notes.

In previous studies (DUGO *et al.*, 1983a; 1983b; 1983c; 1993; 1998; COTRONEO *et al.*, 1983; 1986a; 1986b; LICANDRO *et al.*, 1984; VERZERA *et al.*, 1987; 1996; MONDELLO *et al.*, 1999) aimed at protecting this product, we analysed 364 samples of Italian industrial lemon oil. Numerous studies have appeared in the literature, but few regard lemon oils from countries other than Italy (SHAW, 1979; CAPPELLO *et al.*, 1981; STOROSCIK and WILSON, 1982; KOKETSU *et al.*, 1983; BOELENS and JIMENEZ, 1989; LANCAS and CAVICCHIOLI, 1990; BENINCASA *et al.*, 1990; CHAMBLEE *et al.*, 1991; BLANCO TIRADO *et al.*, 1995; USAI *et al.*, 1996; AYEDOUM *et al.*, 1996; CIERI, 1969; MADSEN and LATZ, 1970; CALABRÒ and CURRÒ, 1976; GLANDIAN *et al.*, 1978; McHALE and SHERIDAN, 1988).

The machines used to obtain lemon oil by the Italian citrus industries are "Pelatrice", "Sfumatrice", "FMC" and "Torchi". Recently, the industrial technologies have been improved, especially concerning the recycling of the water used to carry away the released essential oil and, therefore, a study on the composition of the oils produced with these technologies is of interest.

The volatile and the oxygen heterocyclic fractions of the oil were studied. The results relative to the volatile fraction and to the non-volatile residue, taken together, can best describe the Sicilian lemon oil composition and they can be useful for evaluating the influence of the technology.

This information will also be important for judging the quality of unknown lemon oil and for detecting adulteration and possible contamination.

MATERIALS AND METHODS

This study was carried out on 364 samples of genuine industrial cold-pressed Italian lemon oils produced during the 1997/98 season. The oils were obtained using "Pelatrice", "Sfumatrice", "FMC" and "Torchi" machines (Table 1).

HRGC analysis

Each oil was analysed using a 5160 Mega series Carlo Erba Gas Chromatograph (Fisons Instrument, Milan, Italy) equipped with a C-R3A Shimadzu data processor using the following 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 250°C at 3°C/min; injection mode, split; split ratio 1:100; detector, FID; injector and detector temperature, 250°C; carrier gas, He 100 kPa; injected volume, 1 µL of net whole oil. The quantitative composition was obtained by peak area normalization and the response factor for each component was considered to equal one.

HRGC-MS analysis

Samples were analysed by GC/MS (EI) on a Fisons MD 800 (Fisons Instrument, Milan, Italy) system coupled with Adams' library (ADAMS, 1995). Two different columns were used: a DB5, fused silica column, 30 m x 0.25 mm, film thickness, 0.25 µm (J & W Folston, CA, USA) and a Megawax, fused silica column, 30 m x 0.32 mm; film thickness, 0.40-0.45 µm (Mega, Legnano (MI), Italy); GC conditions for the two columns: oven 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 90 kPa; interface temperature, 250°C; acquisition mass range, 41-300 amu; solvent cut, 2 min. Compound identification was

Table 1 - Number of lemon essential oils analysed according to production period and extraction technology.

	Total	Sfumatrice	Pelatrice	FMC	Torchi
November '97	30	10	4	10	6
December	42	20	2	4	16
January '98	55	20	15	3	17
February	35	9	8	11	7
March	51	14	10	14	13
April	59	20	9	18	12
May	49	7	11	21	10
June	11	-	-	11	-
July/August	7	-	-	7	-
September	18	-	-	14	-
October	11	-	-	11	-
Total	364	100	59	124	81

confirmed by comparing mass spectra of the compounds with published spectra and of retention indices with published index data.

HPLC analysis

All samples were analysed by normal-phase HPLC, using Waters Associates (Milan, Italy) equipment consisting of a model 519 pump, a model 600 E gradient controller, a model 9125 Rheodyne injector; a model 996 photodiode array (PDA) detector. Peak integration and quantitative calculations were performed using a Waters Millenium 2010 system, using a calibration curve obtained for each previously isolated standard compound against a coumarin standard (DUGO *et al.*, 1998). The column was a 15 cm x 3.9 mm i.d. µ-Porasil (Waters Associates, MI, Italy); particle size of 10 µm. Mobile phases were: eluent A: hexane:ethyl acetate, 92:8; eluent B: hexane:ethanol, 90:10. Eluent A was pumped for 15 min, then a linear gradient to eluent B in 5 min, with a final hold for 10 min. Flow rate 1.25 mL/min; column pressure 14.1 bar (204 psi); column temperature 30°C; injection volume was 20 µL of a solution obtained by diluting about 90 mg, accurately weighed, of essential oil with 0.80 mL of hexane:ethyl acetate, 75:25 and adding 0.1 mL of coumarin solution (0.99 mg/mL) as internal standard. Detection was by UV absorbance at 315 nm. The UV spectra of eluting peaks were monitored with the PDA detector in the 220-400 nm region.

RESULTS AND DISCUSSION

Volatile fraction

Tables 2-5 report the average composition and standard deviation for the single compounds and for the classes of substances for the oils analysed according to the various extraction technologies. The values reported refer to the volatile fraction and they do not include the non-volatile residue, which constituted about 2-4% of the oil.

Sixty-four compounds were identified and quantified in each oil sample, constituting more than 99% of the volatile fraction. The qualitative composition as single compounds and classes of substances is in agreement with that of Sicilian lemon oils previously analysed (VERZERA *et al.*, 1996).

The extraction technology did not influence the qualitative composition of the oil, but there were quantitative differences. In fact, "Pelatrice" oils had the highest content of oxygenated and carbonyl compounds, and the lowest content of monoterpene hydrocarbons. "FMC" and "Sfumatrice" oils had similar amounts of carbonyl compounds. Even though "Sfumatrice" oils had a slightly lower amount of carbonyl compounds, such as neral and geranial, than "Pelatrice" oils, they had the best and most appreciated olfactory notes. In fact "Sfumatrice" oils have a fine fresh lemon aroma; on the average they are 15% more expensive than "Pelatrice" oils which have a poorer, grassy odor ("Sfumatrice" machines give a lower yield and more costly oil than "Pelatrice"). The grassy note, typical of "Pelatrice" oils, is probably due to a high content of chlorophyll that is extracted in a larger amount from the fruit peels using rasping machines. "FMC" oils have olfactory notes similar to "Sfumatrice" oils. "FMC" technology is the most commonly used because it is a good compromise between cost, yield and oil quality.

"Torchi" machines, which work on the peel of lemon fruits, produce oils called "Torchi". In this study, "Torchi" oils were obtained by processing the peels which had been previously cold-pressed by "Sfumatrice" machines. These oils had a different composition. The content of carbonyl compounds and alcohols was lower than in the other oils because of

Table 2 - Percentage composition (mean values $\bar{x}$ with standard deviations s) as single compounds and as classes of compounds for "Sfumatrice", "Pelatrice", "FMC" and "Torchi" oils. Numbers in parentheses refer to the number of oils analysed.

	Sfumatrice (100)		Pelatrice (59)		FMC (124)		Torchi (81)	
	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s
tricyclene	0.01	0.001	0.01	0.001	0.01	0.001	0.01	0.001
α-thujene	0.48	0.050	0.47	0.050	0.47	0.041	0.46	0.042
α-pinene	2.21	0.228	2.15	0.206	2.23	0.189	2.11	0.187
camphene	0.07	0.006	0.07	0.008	0.07	0.008	0.06	0.006
sabinene + β-pinene	16.56	1.779	16.74	1.798	18.15	2.293	16.04	1.730
6-methyl-5-hepten-2-one	0.01	0.005	0.01	0.003	0.01	0.004	0.01	0.005
myrcene	1.62	0.167	1.57	0.158	1.52	0.138	1.60	0.155
octanal	0.11	0.032	0.11	0.030	0.08	0.060	0.08	0.021
α-phellandrene	0.04	0.020	0.04	0.019	0.08	0.039	0.04	0.012
δ-3-carene	tr	-	tr	-	tr	-	tr	-
α-terpinene	0.19	0.032	0.17	0.035	0.16	0.041	0.17	0.035
p-cymene	0.12	0.086	0.16	0.087	0.16	0.110	0.12	0.074
limonene	61.84	3.866	61.13	3.610	60.18	3.687	63.31	3.360
(Z)-β-ocimene	0.07	0.017	0.08	0.017	0.07	0.017	0.07	0.016
(E)-β-ocimene	0.13	0.026	0.15	0.025	0.13	0.023	0.13	0.021
γ-terpinene	10.26	1.059	10.42	1.056	10.10	0.844	10.25	0.968
cis-sabinene hydrate	0.03	0.005	0.06	0.049	0.05	0.00	0.01	0.004
octanol	tr	-	tr	-	tr	-	tr	-
terpinolene	0.40	0.043	0.39	0.051	0.36	0.049	0.39	0.043
trans-sabinene hydrate	0.02	0.005	0.04	0.010	0.04	0.010	0.01	0.002
linalool	0.12	0.016	0.12	0.013	0.13	0.017	0.07	0.012
nonanal	0.15	0.027	0.16	0.021	0.16	0.022	0.14	0.017
cis-limonene oxide	tr	-	tr	-	tr	-	tr	-
trans-limonene oxide	tr	-	tr	-	0.01	0.004	tr	-
camphor	tr	-	tr	-	tr	-	tr	-
citronellal	0.09	0.020	0.10	0.017	0.10	0.022	0.08	0.016
borneol	0.01	0.005	0.01	0.002	0.01	0.006	tr	-
terpinen-4-ol	0.07	0.016	0.04	0.009	0.05	0.013	0.04	0.011
α-terpineol	0.18	0.028	0.21	0.022	0.23	0.035	0.10	0.020
decanal	0.05	-	0.05	0.013	0.06	0.016	0.05	0.014
octyl acetate	tr	-	tr	-	tr	-	tr	-
nerol	0.03	0.007	0.04	0.020	0.07	0.043	0.02	0.009
neral	0.95	0.151	1.00	0.153	0.86	0.151	0.68	0.117
piperitone	tr	-	tr	-	tr	-	tr	-
geraniol	0.02	0.007	0.04	0.013	0.02	0.009	0.02	0.005
geranial perillaldehyde	1.64	0.210	1.74	0.264	1.44	0.301	1.22	0.182
bornyl acetate	tr	-	tr	-	tr	-	tr	-
undecanal	0.03	0.008	0.03	0.006	0.04	0.033	0.03	0.007
nonyl acetate	tr	-	tr	-	0.01	0.002	tr	-
methylgeranoate	tr	-	tr	-	tr	-	tr	-
citronellyl acetate	0.03	0.010	0.03	0.010	0.03	0.009	0.03	0.009
neryl acetate	0.40	0.067	0.42	0.069	0.49	0.141	0.43	0.078
geranyl acetate	0.39	0.132	0.45	0.113	0.50	0.141	0.41	0.128
dodecanal	0.01	0.002	0.01	0.003	0.01	0.004	0.009	0.001
decyl acetate	tr	-	0.01	0.002	0.01	0.009	0.005	0.002
(E)-caryophyllene	0.23	0.034	0.23	0.028	0.24	0.044	0.244	0.029

(Continue)

Table 2 - Continue.

trans-α-bergamotene	0.36	0.056	0.38	0.049	0.39	0.065	0.383	0.050
α-humulene	0.02	0.003	0.02	0.005	0.02	0.006	0.017	0.005
(E)-β-farnesene	0.03	0.007	0.03	0.008	0.03	0.008	0.034	0.007
β-santalene	0.01	0.003	0.01	0.002	0.01	0.003	0.013	0.002
γ-muurolene	0.01	0.006	0.01	0.002	0.01	0.002	0.043	0.008
germacrene D	tr	-	0.01	0.002	0.01	0.004	0.007	0.002
valencene	0.02	0.011	0.02	0.015	0.02	0.015	0.026	0.028
bicyclogermacrene	0.07	0.014	0.06	0.017	0.06	0.015	0.068	0.013
(Z)-α-bisabolene	0.04	0.007	0.04	0.006	0.04	0.009	0.043	0.008
β-bisabolene	0.54	0.090	0.56	0.083	0.58	0.098	0.560	0.078
(E)-α-bisabolene	0.01	0.002	0.01	0.003	0.01	0.003	0.015	0.004
tetradecanal	tr	-	0.01	0.002	tr	-	0.009	0.002
2,3-dimethyl-3-(4-methyl--3-pentenyl)-2-norbornanol	0.02	0.006	0.02	0.007	0.02	0.008	0.019	0.006
campherenol	0.02	0.005	0.02	0.003	0.02	0.004	0.020	0.003
α-bisabolol	0.02	0.005	0.02	0.005	0.02	0.005	0.024	0.004
nootkatone	tr	-	tr	-	tr	-	tr	-
hydrocarbons	95.29	0.631	94.84	0.820	95.04	0.628	96.21	4.882
monoterpenes	93.93	0.815	93.44	0.934	93.61	0.813	94.77	3.651
sesquiterpenes	1.36	0.204	1.41	0.166	1.43	0.230	1.44	0.231
carbonyl compounds	3.07	0.371	3.25	0.416	2.81	0.455	2.35	0.407
esters	0.84	0.201	0.92	0.175	1.06	0.285	0.90	0.232
alcohols	0.54	0.074	0.62	0.080	0.67	0.118	0.34	0.079
oxygenated compounds	4.49	0.666	4.80	0.511	4.54	0.512	3.60	0.720

tr = <0.01;
- = not detected.

Table 3 - Amount (mg/kg) of coumarins and psoralens in lemon oil obtained using "Sfumatrice", "Pelatrice", "FMC" and "Torchi" machines (mean values $\bar{x}$ and standard deviations s). Numbers in parentheses refer to the number of oils analysed.

	Sfumatrice (100)		Pelatrice (59)		FMC (124)		Torchi (81)	
	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s
bergamottin	2,635	326	2,955	423	2,877	517	2,973	500
5-geranyloxy--7-methoxycoumarin	2,453	648	2,711	749	2,845	838	2,656	844
citropten	1,360	284	1,495	206	1,473	326	659	239
8-geranyloxypsoralen	399	69	437	116	454	154	440	71
5-isopent-2'-eniloxy-8-(2',3'-epoxyisope-ntyloxy)psoralen	275	58	324	66	204	62	260	53
oxypeucedanin	1,556	379	2,200	413	1,909	449	863	266
byakangelicol	992	292	1,640	261	1,536	427	555	216
Total	9,648	1,027	11,801	1,330	11,267	1,888	8,388	1,753

Table 4 - Amount (mg/kg) of coumarins and psoralens for 1997/98 "Sfumatrice" oils (mean values $\bar{x}$ and standard deviations s). Numbers in parentheses refer to the number of oils analysed.

	November (10)		December (20)		January (20)		February (9)		March (14)		April (20)		May (7)	
	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s
bergamottin	2,486	107	2,518	137	2,526	111	2,428	130	2,704	436	2,975	410	2,768	378
5-geranyloxy-7-methoxycoumarin	1,942	115	2,088	107	2,074	120	2,070	123	2,780	781	3,340	193	2,951	513
citropten	1,458	333	1,694	89	1,492	111	1,261	68	1,200	175	1,097	172	971	129
8-geranyloxypsoralen	500	37	456	50	388	41	344	32	362	57	365	59	351	47
5-isopent-2'-enyloxy-8-(2',3'-epoxyisopentyloxy)psoralen	252	13	287	32	309	31	301	25	324	51	195	49	208	26
oxypeucedanin	1,418	342	1,788	207	1,934	104	1,643	166	1,483	354	1,167	165	962	80
byakangelicol	944	226	1,225	209	1,258	104	982	107	842	295	746	144	559	70
Total	9,000	814	10,066	537	9,871	438	9,028	471	9,647	1,601	9,853	1,259	8,740	1,187

Table 5 - Amount (mg/kg) of coumarins and psoralens for 1997/98 "Pelatrice" oils (mean values $\bar{x}$ and standard deviations s). Numbers in parentheses refer to the number of oils analysed.

	November (4)		December (2)		January (15)		February (8)		March (10)		April (9)		May (11)	
	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s	$\bar{x}$	s
bergamottin	2,668	234	2,664	113	2,826	190	2,779	250	2,993	384	3,200	422	3,509	795
5-geranyloxy-7-metoxycoumarin	1,970	208	1,922	151	2,280	187	2,307	224	3,145	664	3,435	394	3,840	980
citropten	1,485	298	1,746	172	1,581	110	1,473	175	1,526	158	1,348	48	1,272	389
8-geranyloxypsoralen	586	107	476	32	416	47	379	42	388	63	414	65	460	118
5-isopent-2'-enyloxy-8-(2',3'-epoxyisopentyloxy)psoralen	274	52	307	34	352	36	356	38	368	44	209	20	293	71
oxypeucedanin	2,037	254	2,383	136	2,417	166	2,410	213	2,472	209	1,554	235	1,675	407
byakangelicol	1,780	297	1,945	67	1,660	127	1,590	131	1,676	348	1,467	102	1,424	360
Total	11,355	111	11,442	535	11,533	575	11,293	931	12,568	1,512	11,581	1,083	12,652	2,826

Table 6 - Amount (mg/kg) of coumarins and psoralens for 1997/98 "FMC" oils (mean values $\bar{X}$ and standard deviations s). Numbers in parentheses refer to the number of oils analysed.

	November (10)		December (4)		January (3)		February (11)		March (14)		April (18)	
	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s
bergamottin	2,692	93	2,513	149	2,590	245	2,547	122	2,920	682	2,949	296
5-geraniloxy-7-metoxycoumarin	2,032	66	2,026	142	2,074	300	2,149	172	3,083	934	3,171	307
citropten	1,992	127	1,957	322	1,434	126	1,401	80	1,381	279	1,229	130
8-geranyloxypsoralen	583	46	414	53	390	14	354	45	394	94	375	36
5-isopent-2'-enyloxy-8-(2',3'-epoxyisopentyloxy) psoralen	223	13	216	16	224	58	244	9	252	64	151	26
oxypeucedanin	2,435	178	2,351	108	2,157	369	2,111	107	2,021	263	1,401	159
byakangelicol	2,086	405	1,795	168	1,495	48	1,400	121	1,370	355	1,212	155
Total	12,043	789	11,270	641	10,363	1,109	10,206	438	11,421	2,316	10,363	960

	May (21)		June (11)		July/August (7)		September (14)		October (11)	
	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s
bergamottin	3,258	734	3,549	535	3,156	547	2,738	137	2,649	340
5-geraniloxy-7-metoxycoumarin	3,569	911	4,162	325	3,513	470	2,818	190	2,336	713
citropten	1,336	342	1,523	142	1,672	199	1,506	66	1,434	166
8-geranyloxypsoralen	439	117	548	75	972	159	700	146	517	132
5-isopent-2'-enyloxy-8-(2',3'-epoxyisopentyloxy) psoralen	191	47	58	-	tr	-	tr	-	tr	-
oxypeucedanin	1,553	474	2,262	434	2,029	448	1,978	67	1,676	98
byakangelicol	1,418	401	1,815	171	2,331	441	1,977	224	1,438	89
Total	11,282	2,523	11,417	2,193	13,674	2,265	11,717	664	9,870	1,527

Table 7 - Amount (mg/kg) of coumarins and psoralens for 1997/98 "Torchi" oils (mean values $\bar{X}$ and standard deviations s). Numbers in parentheses refer to the number of oils analysed.

	November (6)		December (16)		January (17)		February (7)		March (13)		April (12)		May (10)	
	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s	$\bar{X}$	s
bergamottin	2,715	152	2,786	167	2,777	114	2,660	70	2,953	518	3,348	488	3,546	546
5-geraniloxy-7-metoxycoumarin	2,034	135	2,176	141	2,171	125	2,088	95	2,975	884	3,600	592	3,511	280
citropten	786	212	776	212	557	101	544	78	565	142	680	254	544	82
8-geranyloxypsoralen	522	26	482	58	432	28	378	43	396	71	415	58	426	92
5-isopent-2'-enyloxy-8-(2',3'-epoxyisopentyloxy) psoralen	232	9	268	29	280	26	281	10	294	49	203	40	204	38
oxypeucedanin	716	139	848	190	784	135	801	129	773	193	1,038	286	922	399
byakangelicol	493	66	613	189	516	95	519	78	478	112	533	265	602	22
Total	7,498	409	7,948	706	7,518	364	7,271	318	8,394	1,760	9,786	1,539	9,606	1,648

the prolonged contact of the oil with the aqueous phase, typical of this technology, causing the loss of these components. Because of their composition and, therefore, of their olfactory notes "Torchi" oils, are not separately marketed, but they are added to oils of better quality.

Comparing these results with those previously obtained for Sicilian lemon oils we observed that the oils produced in the 1997/98 season had a higher content of oxygenated compounds (VERZERA *et al.*, 1996). This could be due to the weather conditions during this season as well as to the improved technology that reduced the loss of oxygenated compounds which are soluble in water.

Oxygen heterocyclic fraction

Two coumarins: citropten and 5-geranyloxy-7-methoxycoumarin and five psoralens: bergamottin, 8-geranyloxypsoralen, byakangelicol, oxypeucedanin and 5-isopent-2'-enyloxy-8-(2',3'epoxyisopentyloxy)psoralen were identified and quantified. Table 3 reports the average content of coumarins and psoralens for all the oils analysed. Bergamottin and 5-geranyloxy-7-methoxycoumarin were the principal compounds of this fraction, followed by oxypeucedanin, byakangelicol and citropten.

Even though the "Pelatrice", "Sfumatrice" and "FMC" oils had a similar amount of these compounds, "Pelatrice" oils had the highest amount of total coumarins and psoralens (1,1801 mg/kg), followed by "FMC" oils (1,1267 mg/kg). The higher amount of these compounds in "Pelatrice" oils is due to the efficacy of breaking the utricles and releasing the essential oil. "Torchi" oils had a lower amount of citropten, oxypeucedanin and byacangelicol than the oils obtained with the other technologies.

Tables 4-7 report the data of all the samples analysed according to the month of production and the extraction technology. The values of the coumarins and psoralens were quite constant during the production season. For each technology, from November to February, the amount of bergamottin was higher than that of 5-geranyloxy-7-methoxycoumarin; the opposite happened from March onward. This fact, together with the variation in the quantitative composition of the volatile fraction, could be useful for evaluating the quality of an unknown lemon oil.

In comparison with our previous results (DUGO *et al.*, 1998), we found a higher amount of 5-geranyloxy-7-methoxycoumarin, 8-geranyloxypsoralen and oxypeucedanin in the oils produced in the 1997-98 season than in those produced in the 1995 season.

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