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Volatile compounds from Chétoui olive oil and variations induced by growing area

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Abstract

Fruits from the same variety of *Olea europaea* L., grown under different environmental conditions in the north of Tunisia, were harvested at the same ripening degree and immediately processed. The volatile profile of virgin olive oils was established using solid phase, micro-extraction (SPME) and gas chromatography-mass spectrometry (GC-MS). Compounds belonging mainly to the following chemical classes characterised the volatile profiles: esters, aldehydes, ketons, aliphatic alcohols and hydrocarbons. Significant differences in the proportions of volatile constituents from oils of different geographical origins were detected and the major volatile in approximately 50% of the oil samples was the aldehyde (E)-2-hexenal. The results suggest that, beside the genetic factor, environmental conditions influence the volatile formation.

Keywords: Virgin olive oil; Chétoui cultivar; *Olea europaea*; Production area; Volatile compounds; SPME

1. Introduction

Virgin olive oil has a characteristic flavour that distinguishes it from other edible vegetable oils. After its extraction from the fruit of *Olea europaea* L., extra virgin olive oil can be consumed without any refining process and it preserves its typical aroma (Boskou, 1996; Fedeli, 1977a, 1977b). Flavour and aroma of virgin olive oil are generated by a number of volatile constituents that are present at extremely low concentrations (Kiritsakis, 1998a, 1998b; Kiritsakis & Christie, 2000).

The volatile fraction of virgin olive oils has been previously studied, and includes saturated, unsaturated, aromatic and terpenic hydrocarbons, as well as aldehydes, alcohols, ketones, esters, ethers, furans and other compounds (Aparicio & Morales, 1998; Fedeli, 1977a, 1977b; Flath, Forrey, & Guadagni, 1973; Kiritsakis, 1998a, 1998b; Montedoro, Bertuccioli, & Anichini, 1978).

The volatile composition of olive oil depends on the levels and activity of the enzymes involved in the various pathways (Angerosa, 2002; Angerosa, Basti, & Vito, 1999a) which are genetically determined (Campeol, Flamini, Chericoni, Catalano, & Cremonini, 2001). Other factors that influence the volatiles are ripening cycle of the fruit (Aparicio & Morales, 1998; Ranalli, Tombesi, Ferrante, & De Mattia, 1998; Solinas, Marsilio, & Angerosa, 1987) and the processing equipment (Angerosa, Mostallino, Basti, & Vito, 2001; Di Giovacchino, Costantini, Serraiocco, Surricchio, & Basti, 2001; Kiritsakis, 1998a, 1998b; Morales, Rios, & Aparicio, 1997; Ranalli, Costantini, De Mattia, & Ferrante, 2000; Ranalli & Angerosa, 1996; Salas & Sachez, 1999). The effects of climate and soil type have also been

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studied (Ranalli, Tombesi, De Mattia, Ferrante, & Giansante, 1997, 1999a). However, the effects of these variables on volatile profiles are ambiguous.

The Chétoui variety used in this study is the second main variety cultivated in Tunisia. It is widespread in the north of the country, occurring in plains as well in mountain regions, and shows a high capacity of adaptation to various pedo-climatic conditions. It covers an area of 176,000 ha and accounts for more than 20% of the olive oil produced in Tunisia. The fruit is medium to large with a fat yield of about 20–30% of fresh weight and the oil is characterised by good content of total phenols, *o*-diphenols, tocopherols and good resistance to oxidation.

The aim of this work is to make a contribution to the characterization of this cultivar of *O. europaea* by means of the analyses of the volatiles obtained from virgin oil and to determine changes of the volatile composition of Chétoui olive oil due exclusively to the environmental factor.

2. Materials and methods

2.1. Oil sample extraction

The olives were collected, at the same stage of maturity, from 14 different farms in the north of the country and the same laboratory mill was used to prepare the olive oil samples. The olives were picked by hand from the tree. Only drupes not damaged, fresh and healthy were selected. After harvesting, the olive fruit samples were immediately transported to the laboratory mill where they were transformed into oil within 24 h. The olives were washed and deleafed, crushed with a hammer crusher, and the paste mixed at 25 °C for 30 min, centrifuged without addition of warm water and then transferred into dark glass bottles, and stored in the dark at 4 °C until the moment of analysis.

2.2. Oil sample analyses

Determinations of free acidity, peroxide value, UV absorption characteristics, and fatty acid composition were carried out following the analytical methods described in Regulations ECC/2568/91 and ECC/1429/92 of the Commission of the European Union (1992).

Total phenols and *ortho*-diphenols were quantified colorimetrically (Ranalli, Ferrante, De Mattia, & Costantini, 1999b). Phenolic compounds were isolated by triple extraction of a solution of oil in hexane with a water–methanol mixture (60:40). The Folin–Ciocalteau reagent (Merck) was added to a suitable aliquot of the combined extracts, and the absorption of the solution at 725 nm was measured. Values are given as milligrammes of caffeic acid per kilogramme of oil (Gutfinger, 1981; Vasquez, 1978).

Ortho-diphenols were measured colorimetrically at 370 nm after adding 5% (wt/vol) sodium molybdate in 50% ethanol to the extract (Gutfinger, 1981; Vasquez, 1978). Results are as milligrammes of caffeic acid per kilogramme of oil.

Oxidative stability was evaluated by the Rancimat method, because it is a fast and reliable analytical procedure (Gutiérrez, 1989). Stability was expressed as the oxidation induction time (hours), measured with the Rancimat 743 apparatus (Metrohm Co., Basel, Switzerland), using an oil sample of 3.5 g warmed to 100 °C, and an air flow of 10 l/h. With this well-established methodology, the volatile oxidation products were stripped from the oil and dissolved in cold water, whose conductivity increased progressively. The time taken to reach a fixed level of conductivity was measured.

2.3. Analysis of volatile compounds

2.3.1. General

The volatile profile of virgin olive oils was established using SPME and gas chromatography with mass spectrometry.

2.3.2. Extraction

Solid phase micro-extraction was used as a technique for headspace sampling of Chétoui virgin olive oils. For SPME analyses, Supelco SPME devices coated with poly-dimethylsiloxane (PDMS, 100 μm) were used to sample the headspace of 3 ml of virgin olive oil inserted into a 5 ml glass vial and allowed to equilibrate for 30 min. After the equilibration time, the fibre was exposed to the headspace for 50 min at room temperature. Once sampling was finished, the fibre was withdrawn into the needle and transferred to the injection port of the GC-MS system. The splitless injection mode was used.

2.3.3. Identification

Gas chromatography with mass spectrometry was used for the identification of volatile compounds extracted from Chétoui virgin olive oils.

GC analyses were accomplished with an HP-5890 series II instrument equipped with HP-Wax and HP-5 capillary columns (30 m × 0.25 mm, 0.25 μm film thickness), working with the following temperature programme: 60 °C for 10 min, ramp of 5 °C/min to 220 °C; injector and detector temperatures, 250 °C; carrier gas, nitrogen (2 ml/min); detector, dual FID; split ratio, 1:30; injection, 0.5 μl. The identification of the components was performed, for both columns, by comparison of their retention times with those of pure authentic samples and by means of their linear retention indices (LRI) relative to the series of *n*-hydrocarbons.

The relative proportions of the constituents were obtained by FID peak area normalization. GC-EIMS analyses were performed with a Varian CP-3800 gas chromatograph equipped with a DB-5 capillary column (30 m × 0.25 mm; coating thickness = 0.25 μm) and a Varian Saturn 2000 ion trap mass detector. Analytical conditions: injector and transfer line temperatures, 220 and 240 °C, respectively; oven temperature programmed from 60 to 240 °C at 3 °C/min; carrier gas, helium at 1 ml/min; injection, 0.2 μl (10% hexane solution); split ratio, 1:30. Identification of the constituents was based on comparison of the retention times with those of authentic samples, by means of their LRI relative to the series of *n*-hydrocarbons, and on computer matching against commercial (NIST 98 and ADAMS) and home-made library mass spectra built from pure substances and components of known oils and literature data (Massada, 1976; Jennings & Shibamoto, 1980; Swigar & Silverstein, 1981; Davies, 1990; Adams, 1995). Moreover, the molecular weights of all the identified substances were confirmed by GC-EIMS, using MeOH as CI ionizing gas. All of the reference compounds were obtained by Sigma–Aldrich (Sigma, Aldrich, and Fluka catalogues) with the exception of (E, E)-α-farnesene (TCI America), (Z)-3-hexenyl benzoate (ABCR Product List), and (E)-2-decenal which originated from (Lancaster Synthesis Ltd).

3. Results and discussion

3.1. Free acidity, UV spectrophotometric indices, peroxide index

For the majority of the Chétoui virgin olive oils analysed, the values of the analytical parameters fell within the ranges established for the highest quality category 'extra virgin' olive oil. As shown in Table 1, these analytical parameters are practically unaffected by the region of olive cultivation.

In all studied samples, the free fatty acid content was much lower than the upper limit of 1% established for the best commercial quality olive oil, designated extra virgin (Regulations EEC 2568/91) (Table 1).

The peroxide value evaluates hydroperoxides content and offers a measure of lipid oxidation. In the samples studied, the peroxide values of the oils were below the limit of 20 meq of oxygen/kg of oil, which is accepted as the limit for 'extra' quality virgin olive oil. They are ranged from 2.29 to 12.9 meq O_2/kg oil (Table 1).

To evaluate the oxidation level of oil samples the parameter K_{270} has been used, data are shown in Table 1. The values of this parameter were all good in all analysed oils and below the limits established for 'extra' virgin olive oils (EEC Regulations).

These results are consistent with the findings of other authors (Kiritsakis, 1998a, 1998b; Ranalli & Angerosa, 1996). They reported that, cultivar or origin area had no significant influence on these analytical parameters, which are basically affected by factors causing damage to the fruits (e.g. olive fly attacks or improper systems of harvesting, transport and storage of olives).

3.2. Fatty acid composition

The major fatty acids present as glycerides in ''Chétoui'' olive oil are oleic (C18:1), linoleic (C18:2), palmitic (C16:0), and stearic acid (C18:0) (Table 1). Oleic acid is the main monounsaturated fatty acid in olives and is present in higher concentrations (64.2–72.8%) than other acids. Palmitic acid content varies between 9.5% and 12.2% according to the zones of plantation. Polyunsaturated fatty acids are very important for human nutrition as they are considered essential. Linoleic acid was the dominant polyunsaturated fatty acid in ''Chétoui'' olives ranging from 13.3% to 20.5%, while linolenic acid (Cl8:3) ranged from 0.65% to 0.82%.

Variations in oleic and linoleic acid contents observed in olive oil samples obtained from Chétoui cultivar (Table 1) are probably related to both genetic factors and environmental conditions during the development and the maturity of the fruit (Fedeli, 1977b; Lavee & Wodner, 1995). These results are in agreement with the findings of other authors (Bruni, Cortesi, & Fiorino, 1994; Deidda et al., 1994; Rana & Ahmed, 1981; Osman, Metzidakis, Girasopoulos, & Kiritsakis, 1994; Schiratti, 1999). They reported that several agronomic parameters can modify the fatty acid composition of olive oil. The most studied aspects include cultivar and origin, fruit ripening, harvest period and pedoclimatic conditions of production.

3.3. Total phenols and o-diphenols

Olive oil is the only vegetable oil which contains appreciable amounts of phenolic compounds (which were represented basically by *o*-diphenols) acting as antioxidant substances and conferring to it a greater stability against oxidation during storage (Argenson, 1999).

The contents of total phenols and *o*-diphenols of olive oils varied widely according to the production area. Amdoun oil showed the highest values in phenols and *o*-diphenols 748 and 295 mg/kg, respectively, whereas Borj El Amri oil recorded the lowest 467 and 198 mg/kg, respectively.

As reported by different authors, the amount of total phenols (200–500 mg/kg on average) shows a great variability (from 50 to 1000 mg/kg) (Boskou, 1996), depending on various factors, such as cultivar, climate and environmental factors, ripeness, processing, follow-

Table 1
Analytical and compositional characteristics of fresh virgin olive oils from Chétoui cultivar grown in 14 different geographical areas

Analytical oil parameters	Geographical area													
	Amdoun	Testour	Bouarada	Goubellat	Lakhouet	Gáafour	Amayem	Chuigui	Slouguia	Elles	Sers	Borj El Amri	Jendouba	Zaghouan
Acidity (as oleic acid, g/100 g)[a]	0.30 ± 0.10	0.48 ± 0.07	0.91 ± 0.22	0.51 ± 0.10	0.56 ± 0.21	0.40 ± 0.10	0.91 ± 0.07	0.48 ± 0.12	0.86 ± 0.38	0.25 ± 0.05	0.33 ± 0.06	0.48 ± 0.07	0.25 ± 0.05	0.70 ± 0.18
Peroxide index (meq O_2/kg)[a]	5.56 ± 1.52	9.21 ± 3.82	3.92 ± 0.96	6.40 ± 1.86	4.96 ± 0.95	4.80 ± 1.24	7.57 ± 1.65	6.16 ± 1.64	9.81 ± 0.78	2.29 ± 0.52	11.0 ± 2.79	8.40 ± 4.93	12.94 ± 1.02	6.25 ± 1.46
K_{270}[a]	0.09 ± 0.03	0.15 ± 0.05	0.14 ± 0.04	0.16 ± 0.04	0.11 ± 0.03	0.16 ± 0.06	0.13 ± 0.03	0.12 ± 0.02	0.16 ± 0.04	0.11 ± 0.03	0.19 ± 0.03	0.20 ± 0.05	0.18 ± 0.04	0.12 ± 0.03
Total phenols (as caffeic acid, mg/kg)[ab]	748 ± 39.0	526 ± 58.8	613 ± 36.1	564 ± 44.5	559 ± 54.0	673 ± 79.5	606 ± 48.4	679 ± 4.91	644 ± 23.5	714 ± 43.3	545 ± 95.8	467 ± 37.1	523 ± 38	481 ± 52.6
o-diphenols (as caffeic acid, mg/kg)[ab]	295 ± 9.26	221 ± 30.5	233 ± 17.6	247 ± 34.4	229 ± 22.7	263 ± 35.6	253 ± 17.9	274 ± 8.46	255 ± 23.7	279 ± 15	228 ± 35.3	198 ± 5.05	227 ± 26.2	209 ± 24.4
Stability (h)[a]	52.9 ± 6.90	36.6 ± 4.82	38.8 ± 4.00	35.2 ± 7.56	33.9 ± 1.58	37.0 ± 0.48	36.8 ± 5.62	41.6 ± 4.27	39.4 ± 1.03	43.3 ± 3.91	32.5 ± 2.63	23.8 ± 3.79	36.4 ± 2.83	25.3 ± 1.95
Palmitic acid (%)[ac]	9.79 ± 0.48	9.82 ± 0.30	10.2 ± 0.30	10.7 ± 0.63	10.30 ± 0.48	10.7 ± 0.77	9.79 ± 0.42	12.2 ± 0.83	10.3 ± 0.94	9.47 ± 0.53	10.63 ± 0.48	11.5 ± 0.73	9.71 ± 1.28	10.3 ± 0.48
Stearic acid (%)[ac]	2.79 ± 0.61	2.57 ± 0.25	2.67 ± 0.62	2.75 ± 0.25	3.10 ± 0.75	2.67 ± 0.31	2.79 ± 0.34	2.88 ± 0.64	2.74 ± 0.29	3.27 ± 0.26	3.13 ± 0.31	3.12 ± 0.56	3.36 ± 0.63	2.78 ± 0.17
Oleic acid (%)[ac]	71.0 ± 0.71	72.82 ± 0.66	68.1 ± 1.74	64.2 ± 0.89	67.0 ± 1.88	68.6 ± 1.58	70.97 ± 1.13	64.2 ± 1.76	66.3 ± 1.54	67.8 ± 1.28	66.4 ± 1.43	66.6 ± 1.18	71 ± 0.89	67.9 ± 0.67
Linoleic acid (%)[ac]	14.8 ± 0.95	13.3 ± 0.49	17.0 ± 1.64	20.5 ± 0.49	16.4 ± 2.42	16.4 ± 1.26	14.8 ± 1.19	18.3 ± 1.43	18.4 ± 1.05	17.4 ± 1.85	17.8 ± 1.42	17.5 ± 1.00	14.0 ± 0.46	17.2 ± 0.47
Linolenic acid (%)[ac]	0.68 ± 0.05	0.65 ± 0.05	0.75 ± 0.11	0.79 ± 0.18	0.79 ± 0.09	0.76 ± 0.08	0.68 ± 0.09	0.82 ± 0.06	0.73 ± 0.09	0.69 ± 0.11	0.74 ± 0.06	0.73 ± 0.08	0.68 ± 0.11	0.71 ± 0.06
Oleic acid/linoleic acid ratio	5. 26 ± 0.39	5.47 ± 0.23	4.04 ± 0.46	3.14 ± 0.11	4.16 ± 0.68	4.22 ± 0.37	4.83 ± 0.49	3.54 ± 0.36	3.62 ± 0.28	3.94 ± 0.46	3.76 ± 0.35	3.83 ± 0.28	5.06 ± 0.14	3.95 ± 0.10
Unsaturated fatty acids/saturated fatty acids ratio	6.15 ± 0.32	7.01 ± 0.16	6.68 ± 0.27	6.39 ± 0.34	6.33 ± 0.70	6.42 ± 0.42	6.87 ± 0.05	5.56 ± 0.53	6.55 ± 0.43	6.75 ± 0.51	6.17 ± 0.40	5.82 ± 0.37	6.61 ± 0.66	6.56 ± 0.37

[a] Data are means of three independent samples.
[b] As determined by colorimetric method.
[c] Related to the total area of chromatogram.

ing storage of the oil (Alessandri, 1997; Garcia, Seller, & Perez-Camino, 1996; Gutiérrez, Arnaud, & Albi, 1999; Parlati, Perri, Palopoli, & Rizzuti, 1994; Tous & Romero, 1994).

3.4. Oxidative stability

Oxidative stability of the analyzed Chétoui olive oils varies according to growing area. It ranged from a minimum of 23.83 h to a maximum of 52.91 h (Table 1). Compared to European ones, our samples seem to have low stability values (<70 h). For example, the mean stability of Picual and Cornicabra oils, two main Spanish varieties, goes over 100 h (Aparicio, Roda, Albi, & Gutiérrez, 1999; Deiana et al., 2002; Gutiérrez, Arnaud, & Garrido, 2001). The highest stability value was registered in Amdoun oil and can be explained by its richness in phenols and, more exactly, in *o*-diphenols (Table 1).

3.5. Volatile components

Aroma is an important criterion for virgin olive oils. Consequently, the identification of the compounds contributing to this aroma is considered to be a key for quality and authentication control. In fact, volatile components of olive oil are of great interest since they are related to its quality and are used to detect adulteration (Cavalli, Fernandez, Lizzani-Cuvelier, & Loiseau, 2004).

Many analytical procedures have been used to isolate, identify and quantify the volatile components that characterize olive oil aroma (Angerosa, 2002). Among these extraction techniques, solid phase micro-extraction (SPME) is a solvent-free sample preparation technique for the extraction of volatile and non-volatile compounds, and is also a simple and fast technique to implement. This method, developed by Arthur and Pawliszyn in 1990 (Arthur & Pawliszyn, 1990; Zhang & Pawliszyn, 1993) for water analyses and then applied to food analysis, has been used recently, in food flavour analysis.

Several studies have been published on the analysis of olive oil volatile compounds using SPME, and many components have been identified (Bentivenga, D'auria, De Luca, De Bona, & Mauriello, 2001; Flamini, Cioni, & Morelli, 2003; Vichi et al., 2003).

Components identified in the present study are listed in Table 2. Forty eight compounds were isolated and characterized by GC-MS, representing 84.8–98.8% of the total amount. For the Tunisian oils involved in this study, (E)-2-hexenal, the main one extracted by SPME, was the major volatile in approximately 50% of the oils tested (Table 2). Other compounds present in a relatively high concentration were (E)-2-hexen-l-ol, (E)-3-hexen-l-ol, heptanal, α-pinene, mesitylene, limonene, 1,8-cineol, β-selinene, α-copaene and (E, E)-α-farnesene. As shown in Table 2, the chemical composition of the volatile fraction of Chétoui olive oils varies widely, depending on the region of cultivation.

A typical mass chromatogram of the volatile components of one of the analysed samples is shown in Fig. 1. The results obtained for almost all analysed samples showed that the chromatograms obtained with FID and mass spectral detection were similar but an important distinction was seen in the relative intensity of peaks, reflecting significant differences in the proportions of volatile constituents in oils of different geographical origins (Table 2). Qualitative differences were detected in some samples; so, their profiles were quite different, mainly at the end of the chromatogram.

In the analysed oils, several terpenic hydrocarbons (mono- and sesquiterpenes) were often detected and the sum of their areas accounted for 8.34–29.3% of the detected volatiles (Table 2). (E, E)-α-farnesene, a tetra-unsaturated acyclic sesquiterpene, was present in the largest amount with respect to the other sesquiterpenes in approximately 70% of the oils analysed, amounting to 13.2% of the total in some samples (Amayem). Besides α-farnesene, the most common sesquiterpenes were β-selinene, α-copaene, γ-muurolene and kessane. α-Copaene is a mono-unsaturated sesquiterpene that has already been detected in Spanish oils, mainly in those obtained from olives of the Hojiblanca variety (Guinda, Lanzon, & Albi, 1996).

The SPME analysis of Gâafour oil allowed us to identify, among the main volatiles, (E)-3-hexen-1-ol (21.64%), (E)-2-hexenal (16.05%), 1,8-cineol (11.0%), mesitylene (8.44%), decanal (7.33%), tricyclene (3.29%), 2,6-dimethyI-l-heptene (2.58%), linalool (2.55%), camphor (2.45%), (E, E)-2,4-octadienal (1.85%), 2,3-dehydro-l,8-cineole (1.82%), nonanal (1.24%), α-copaene (1.28%) and (E, E)-α-farnesene (1.15%) (Table 2).

The major constituents of the volatile fraction of Borj El Amri oil were (E)-2-hexenal (45.8%), 1,8-cineol (12.96%), α-pinene (11.4%), (E)-3-hexen-1-ol (6.96%), heptanal (5.45%), mesitylene (3.19%), limonene (2.98%), *p*-cymene (2.13%), sabinene (1.54%) and 2,3-dehydro-1,8-cineol (1.15%) (Table 2). The composition of the volatile fraction of Chuigui oil was very similar, particularly from the qualitative point of view. The components were almost the same except for heptanal, camphene and *o*-methyl anisole; they were found only in Borj El Amri oil. Others were present only in Chuigui oil : (E)-ocimene, tricyclene, nonanal, camphor, α-terpineol and γ-terpinene (Table 2).

Also, for oils produced from Lakhouet and Amayem regions, the compositions of the volatile fractions were very similar from a qualitative point of view. The main components in both samples were (E)-2-hexenal, (E)-2-hexen-l-ol, heptanal, mesitylene, 1,8-cineol, decanal, α-copaene, β-selinene and (E, E)-α-farnesene (Table 2).

Table 2
Composition[a] of the volatile fraction obtained from Chétoui virgin olive oils

Compounds	LRI[b]	Amdoun	Testour	Bouarada	Goubellat	Lakhouet	Gáafour	Amayem	Slouguia	Elles	Sers	Chuigui	Borj El Amri	Jendouba	Zaghouan
(E)-3-hexen-l-ol	852			2.56			21.6					3.39	6.96	5.83	
(E)-2-hexenal	856	42.8	35.7	69.9	tr	10.5	16.1	27.9	18.8	16.8	41.5	24.1			38.1
(E)-2-hexen-l-ol	862		22.8			44.5		29.94	31.3	18.45			45.8	10.37	21.8
2,6-Dimethyl- 1 heptene	866				39.0		2.58								
Heptanal	901		1.17			5.83		0.68	2.42	2.14	10.8		5.45	12.9	8.11
Tricyclene	928	6.63					3.29		4.05	4.37		0.21			
α-Pinene	941	7.26	0.97	1.84		tr		tr	2.46	13.3	6.99	22.2	11.4	12.7	7.25
Camphene	955		1.81										0.48	0.50	
Sabinene	978											4.59	1.54	0.75	
β-pinene	982											3.04	1.09	0.47	0.13
Myrcene	992											0.68	0.30		
2,3-Dehydro-l, 8-cineole	993		0.33				1.82		0.60	1.80	1.39	1.02	1.15	2.46	0.85
Mesitylene	996	21.10	1.23		7.51	4.73	8.44	2.22	2.52	9.81	6.62	1.56	3.19	9.32	3.64
(E)-3-Hexen-1-ol-acetate	1004									2.08		0.99	0.36	10.3	0.33
3-Methyl-4-penten-1-ol-acetate	1005	0.98		0.75							1.99				
n-Hexyl acetate	1010										1.37			4.03	
3-Carene	1011											0.98			
o-Methyl anisole	1013		0.22										0.86		
(E, E)-2,4-Heptadienal	1017	0.13													
p-Cymene	1028						1.13		0.38	2.38	1.38	3.30	2.13	1.53	1.17
Limonene	1033	1.25				0.87	1.56	tr	1.40	2.78	1.56	3.87	2.98	2.58	1.53
1,8-Cineol	1035		0.35	1.38	12.2	5.51	11.0	tr	9.67	19.4	8.9	18.3	13.0	11.7	7.96
(E)-OCimene	1052		0.26	1.42	5.16	1.65	2.56	tr	0.93		0.89	0.37			0.69
γ-Terpinene	1064			0.55			tr					0.71		2.11	
cis-Linalool oxide	1075													1.02	
Linalool	1101		0.80	0.74		0.87	2.55		1.85		1.68				0.34
Nonanal	1104		0.55		6.58	1.39	1.24	tr				0.43			0.28
(E, E)-2,4-Octadienal	1115			0.47	2.13		1.85								
cis-p-Menthα-2,8-dien-l-ol	1142		0.37			0.65	2.45				2.42				
Camphor	1145					0.65	2.45		3.38	0.53	2.42	0.34			
Menthone	1156								0.74						
Umbellulone	1173								1.00						
Menthol	1175					1.13	0.99		1.11		1.55				
4-Terpineol	1179						1.10		0.59		1.11				
α-Terpineol	1191		0.62						0.72			0.68			
Decanal	1205	5.73	3.12			7.70	7.33	6.53			2.44				
Methyl carvacrol	1244								0.63		0.79				

Isohornyl acetate	1286												
α-Cubebene	1351	0.43											
α-Copaene	1377	3.18	3.72	0.49	4.07	2.42	1.28	1.72	1.68		0.91		1.41
(E)-β-Damascenone	1381		0.39						0.85		0.78		
β-Caryophyllene	1420		0.97										0.99
β-Cedrene	1423		0.24										
γ-Muurolene	1433		0.28						1.32				
β-Chamigzene	1465		0.28										
β-Selinene	1487	6.70	4.64	2.89	5.37	2.91	0.96	3.10	2.21			0.76	
(E, E)-α-farnesene	1506	3.86	3.41	1.55	4.83	3.52	1.15	13.2	3.06	3.55	0.79	1.09	
Kessane[d]	1530		5.60	1.46	1.10			1.77	0.43				

[a] Percentages obtained by FID peak-area normalisation (Hp-5column).
[b] Linear retention indices (DB-5 column).
[c] tr ⩽ 0.1%.
[d] Tentative identification (no reference compound available).

The analysis of volatile fraction obtained from Testour oil led to the identification of 24 constituents. The major volatile compounds identified were (E)-2-hexenal (35.68%), (E)-2-hexen-l-ol (22.8%), Kessane (5.60%), (β-selinene (4.64%), α-copaene (3.72%), (E, E)-α-farnesene (3.41%) and decanal (3.12%)). The minor compounds identified were camphene (1.81%), mesitylene (1.23%), heptanal (1.17%), β-caryophyllene (0.97%), α-pinene (0.97%), linalool (0.80%), nonanal (0.55%) and (E)-β-damascenone (0.39%) (Table 2).

The volatile fraction of Zaghouan oil was characterized by the dominance of two compounds: (E)-2-hexenal (38.1%) and (E)-2-hexen-l-ol (21.8%) (Table 2). The other compounds identified were mainly heptanal (8.11%), 1,8-cineol (7.96%), α-pinene (7.25%), mesitylene (3.64%), limonene (1.53%) and *p*-cymene (1.17%) (Table 2).

The main constituents of the volatile fraction of Jendouba oil were heptanal (12.9%), α-pinene (12.7%), 1,8-cineol (11.7%), (E)-2-hexen-l-ol (10.4%), mesitylene (9.32%), (E)-3-hexen-l-ol (5.83%), and *n*-hexyl acetate (4.03%). The minor compounds identified were limonene (2.58%), 2,3-dehydro-l,8-cineole (2.46%), *p*-cymene (1.53%), γ-terpinene (2.11%), α-copaene (1.41%), *cis*-linalool oxide (1.02%), β-caryophyllene (0.99%), sabinene (0.75%), camphene (0.50%) and β-pinene (0.47%) (Table 2).

The major constituent of the volatile fraction obtained from Bouarada oil was identified as (E)-2-hexenal (69.9%). The other volatile compounds were mainly β-selinene (2.89%), (E)-3-hexen-l-ol (2.56%), α-pinene (1.84%), (E, E)-α-farnesene (1.55%), kessane (1.46%), (E)-ocimene (1.42%) and 1,8-cineol (1.38%) (Table 2).

Also, the aldehyde (E)-2-hexenal was the major volatile identified in Amdoun oil (42.8%). The other compounds detected were, α-pinene (7.26%),tricyclene (6.63%), mesitylene (21.1%), (β-selinene (6.70%), decanal (5.73%), (E, E)-α-farnesene (3.86%) and α-copaene (3.18%)) (Table 2).

The chemical composition of the volatile fraction of Elles oil was characterized by the pre-eminence of three compounds: 1,8-cineol (19.4%), (E)-2-hexen-l-ol (18.5%) and (E)-2-hexenal (16.8%). The other compounds detected were mainly, α-pinene (13.3%), mesitylene (9.81%), tricyclene (4.37%), limonene (2.78%), (E, E)-α-farnesene (3.55%), *p*-cymene (2.38%) and heptanal (2.14%) (Table 2).

The analysis of the volatile fraction of Slouguia oil shows the same composition as the Elles oil (Table 2). There are weak qualitative and quantitative variations. Indeed, the major constituents that characterize the volatile fraction were always : (E)-2-hexen-l-ol, (E)-2-hexenal, 1,8-cineol, mesitylene, α-pinene, heptanal, tricyclene, limonene, and (E, E)-α-farnesene (Table 2).

The major constituent detected in the volatile fraction of Sers oil was the aldehyde (E)-2-hexenal (41.5)%. The

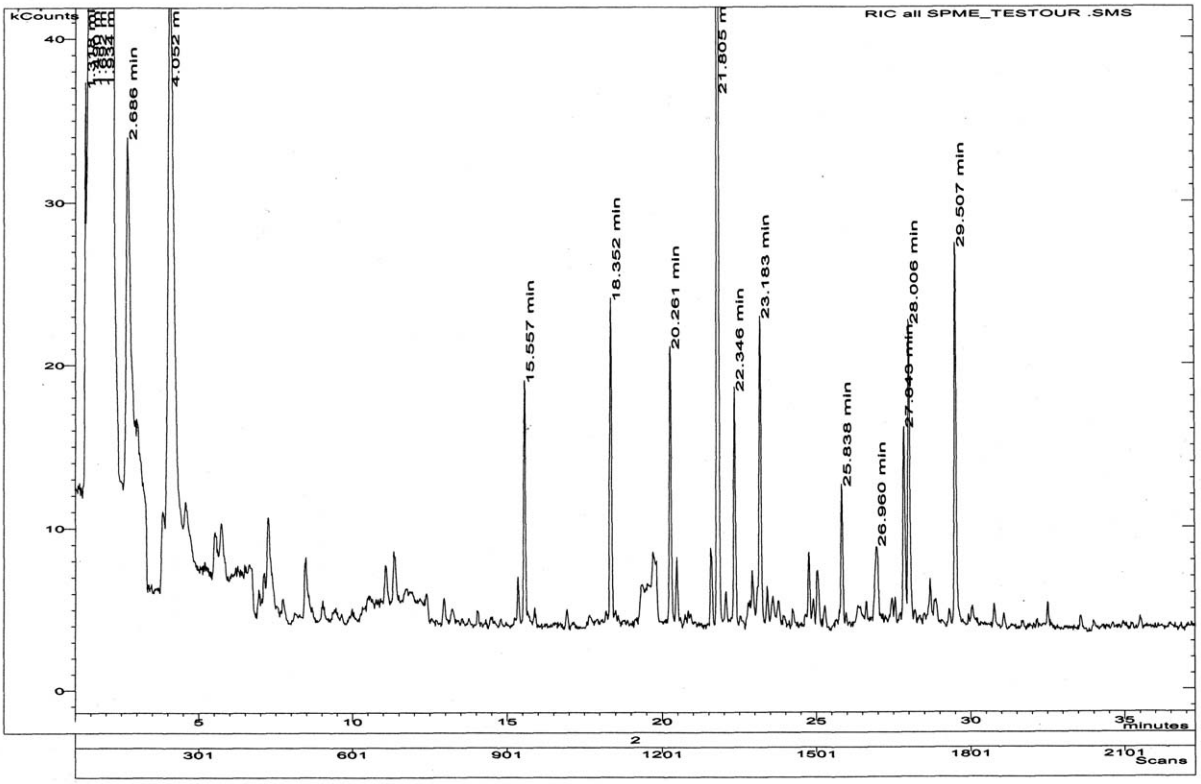

Fig. 1. A typical mass chromatogram of the volatile components of one of the analysed samples.

other compounds identified were mainly, heptanal (10.8%), α-pinene (6.99%), 1,8-cineol (8.90%), mesitylene (6.62%), decanal (2.44%), *cis-p*-mentha-2,8-dien-l-ol (2.42%), camphor (2.42%) and 3-methyl-4-penten-l-ol-acetate (1.99%).

Finally, 2,6-dimethyl-l-heptene (39.0%) and 1,8-cineol (12.2%) were the two compounds that qualitatively characterized the volatile fraction of Goubellat (Table 2). The other compounds identified were mainly mesitylene (7.51%), nonanal (6.58%), β-selinene (5.37%), (E)-ocimene (5.16%), (E, E)-α-farnesene (4.83%) and α-copaene (4.07%) (Table 2). The major constituent, 2,6-dimethyl-l-heptene, has already been detected in Italian oils, mainly in those obtained from olives of the Olivastra Seggianese variety (Flamini et al., 2003).

It should be remembered that the volatile compounds present at higher concentrations are not always the main contributors to oil aroma (Erickson & Covey, 1980). There are not only compounds that contribute to the delicate flavour of Chétoui virgin olive oils. In fact, a large number of volatiles can be formed during autoxidation of unsaturated acyl lipids and many of them can cause off-flavours (Andreas & Arnd, 2000). Table 3 shows the aroma description of the volatile flavour compounds obtained from Chétoui virgin olive oil.

Many studies on volatiles from olive oil can be found in the literature. Some of them describe the relationships between volatile compounds and virgin olive oil odour notes (Angerosa, Mostallino, Basti, & Vito, 2000a; Blekas, Guth, & Grosch, 1994; Grosch, 1994; Guth & Grosch, 1991; Reiners & Grosch, 1998). The influence of operative conditions during storage and processing has been also evaluated (Angerosa, Biasti, Vito, & Lanza, 1999b; Angerosa, D'Alessandro, Basti, & Vito, 1998; Angerosa, Mostallino, Basti, Vito, & Serraiocco, 2000b; Koprivnjak, Procida, & Zelinotti, 2000), as well as their biogenesis (Gardner, 1991; Hatanaka, Kajiawara, & Sekiya, 1987; Olias, Pérez, Rios, & Sanz, 1993). The compounds have been also used to characterise the aroma of the oil obtained from new cultivars (Ranalli, Modesti, Patumi, & Fontanazza, 2000) or the presence of adulterants or contaminants (Overton & Manura, 1995).

A direct comparison with the literature data is not possible because of the great variability of the volatile composition with reference to the different ripeness stages of olives, extraction techniques, and analytical methods. According to Kiritsakis (1998a, 1998b) and Salas and Sachez (1999), methods used and conditions applied to obtain olive oil from olive fruit affects its volatile composition. In fact, the number of volatile compounds detected in the aroma of an olive oil depends on the quality of the virgin olive oil and on the methodology adopted for their determination. Thus, depending

Table 3
Aroma description[a] of the volatile flavour compounds obtained from "Chétoui" virgin olive oils

Compound	Aroma description
(E)-2-Hexenal	Apple, green, leaf
(E)-2-Hexenol	Green, grassy
(E)-3-Hexenol	Green, leaf, nuts
Heptanal	Fat, citrus, rancid
α-Pinene	Pine
Camphene	Camphor
Sabinene	Pepper, wood
β-Pinene	Pine, resin
Myrcene	Balsamic, must, spice
2,3-Dehydro-l,8-cineole	Mint, lemon
Hexyl acetate	Fruit, herb, green (grassy)
Limonene	Citrus, lemon, orange
1,8-Cineol	Mint, sweet
p-Cymene	Solvent, citrus
(E)-Ocimene	Sweet, herb
Nonanal	Fat, citrus, green
2,4-Octadienal	Green, seaweed, cucumber
Menthone	Fresh, green
Camphor	Camphor
Decanal,	Soap, orange peel, tallow
α-Cubebene	Herb, wax
α-Copaene	Wood, spice
(E)-β-Damascenone	Apple, rose, honey
β-Caryophyllene	Wood, spice
β-Selinene	Herb
(E, E)-α-Farnesene	Wood, sweet

[a] (Aparicio et al., 1996; Blekas & Guth, 1993; Morales et al., 1997; Kiritsakis, 1998a).

on the temperature and time used for obtaining the volatile fraction, different results can be found (Cert, 2000).

However, also in the Chétoui cultivar, the main constituents identified were aldehydes, alcohols, ketones and esters. Furthermore, mono- and sesquiterpene hydrocarbons have been detected among the main volatile of this cultivar. The role of these components in the definition of flavour is not clear. In fact, in the literature, only very few papers (Bentivenga et al., 2001; Bortolomeazzi, Berno, Pizzale, & Conte, 2001; Fedeli, 1977a, 1977b; Flath et al., 1973; Vichi et al., 2003; Zunin et al., 2004) report the presence of these compounds, which could play a very important role in the fragrance of this valuable food.

Kubo, Lude, and Kubo (1995) evaluated the antimicrobial activity of the compounds identified in the distillate obtained from olive and olive oil extract. Among these were acyclic compounds such as hexanal, nonanal, 1-hexanol, 3-hexanal, 2-heptanal and acyclic and cyclic mono- and sesquiterpene hydrocarbons, such as α-farnesene. They obseved that most of these compounds exerted antimicrobial activities against a range of different microorganisms, among them *Staphylococcus aureus*, *Streptococcus mutans*, *Esherichia coli*, *Candida utilis* and *Aspergillus niger* (Kubo et al., 1995).

4. Conclusion

Analysis of the 14 Tunisian virgin olive oils by SPME enabled us to identify compounds, representing 84.1–98.8% of the chemical composition.

Among the C6 compounds, (E)-2-hexenal, the principal compound extracted by SPME, distinguished the oil samples from Bouarada, Amdoun, Sers, Chuigui, Borj El Amri, Zaghouan and Testour, whereas, the corresponding alcohols: (E)-2-hexenal and (E)-3-hexenol, characterized the samples from Lakhouet, Gâafour, Amayem and Slouguia. The other compounds identified were mainly heptanal, α-pinene, (E, E)-α-farnesene, β-selinene, 1,8-cineol and limonene.

The application of SPME to the analysis of virgin olive oil headspace allowed the detection of significant differences in the proportions of volatile constituents from oils of various geographical origins.

As the harvesting period and extraction conditions were similar for all studied samples, the results indicate that, besides the genetic factor, environmental conditions influence the volatile production.

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