ELSEVIER

Food Chemistry 74 (2001) 267–274

Food Chemistry

www.elsevier.com/locate/foodchem

Cornicabra virgin olive oil: a study of five crop seasons. Composition, quality and oxidative stability

M.D. Salvador *, F. Aranda, S. Gómez-Alonso, G. Fregapane

Universidad de Castilla — La Mancha, Facultad de Químicas, Departamento de Química Analítica y Tecnología de Alimentos, Avda. Camilo José Cela, 10, 13071 Ciudad Rea, Spain

Received 17 October 2000; received in revised form 11 January 2001; accepted 11 January 2001

Abstract

The chemical composition of Cornicabra virgin olive oils ($n=181$) from five successive crop seasons (from 1994/95 to 1998/99) and its relationship with quality and oxidative stability is examined. The main characteristics of Cornicabra olive oils were: a high oleic acid (80.4±1.0%, as mean and standard deviation) and low linoleic acid content (4.5±0.6%); high campesterol level (4.2±0.2%), exceeding the EU Regulation upper limit of 4%; large total phenol content (ranging from 19 to 380 mg/kg as caffeic acid for the commercial oils, and from 180 to 614 mg/kg for the Abencor oils), and oxidative stability ranging from 9 to 143 h, by Rancimat, for the commercial oils and from 18 to 193 h for the Abencor oils. Sensory evaluation showed that the total score of 35% of commercial Cornicabra olive oil was lower than the limit of 6.5 established for the 'extra-virgin' category, whereas the minimum score observed for Abencor oils was 6.8. This means that, although the majority of the chemical parameters fell within the limits established for the maximum olive oil category, with a few exceptions, significantly more of these olive oils fail the sensory minimum requirements for the highest category.

Keywords: Virgin olive oil; Cornicabra variety; Chemical composition; Quality; Oxidative stability

1. Introduction

Virgin olive oil is one of the edible fats most highly prized by Mediterranean peoples because it can be consumed without any refining process and thus retains its natural flavor and aroma. It also has highly-appreciated nutritional characteristics thanks to its balanced fatty-acid composition (Boskow, 1996a; Grande Covián, 1996).

The Cornicabra olive variety is the most common in Castilla — La Mancha. It covers an area of 300,000 Ha mainly in the provinces of Ciudad Real and Toledo, and accounts for more than 14% of the olive oil produced in Spain. The fruit is medium to large with a characteristically elongated and asymmetric shape. The fat yield is 22–24% of fresh weight and the oil is valued for its high stability and good sensory characteristics, such as a dense sensation and a balanced aroma, sour and pungent (Barranco, Rallo, Uceda, & Hermoso, 1994; Salvador, Aranda & Fregapane, 1998).

The study of the chemical composition of virgin olive oils of a pure variety or from a specific production area is of great scientific interest. It is also of interest (i) to the local industrial sector, which is composed of many small oil mills lacking adequate laboratory facilities and qualified staff and (ii) to the international olive oil business, since the exportation and commercialization of Spanish oil affects many countries, and (iii) to the final consumer, who demands more information on the characteristics and properties of high quality traditional local products and is learning to appreciate less-processed food. An example of this widespread interest and necessity in Castilla — La Mancha is the recent creation of the 'Montes de Toledo' Foundation (DOCM, 1998), whose task is to certify the origin, authenticity and high quality of the Cornicabra virgin olive oil produced in a delimited geographic area and to improve local and international knowledge of this oil variety, its added value and its marketing.

To this end, a major effort has been made in recent years by the main olive oil-producing countries to study the chemical composition of major and minor components and their relation with oil quality, and to establish

* Corresponding author. Tel.: +34-926-295300; fax: +34-926-295318.

E-mail address: asalvador@qata-cr.uclm.es (M.D. Salvador).

0308-8146/01/$ - see front matter
PII: S0308-8146(01)00148-0

analytical determinations that can effectively identify olive oil varieties or oil produced in a specific area (Conte, Carboni, & Lercker, 1993; Lanza, Russo, & Tomaselli, 1998; Poiana et al., 1997; Poiana, Giuffré, Modafferi, Giuffré, Calogero, & Mincione, 1996; Ranelli, De Mattia, Ferrante, & Giansante, 1997; Russo, & Fichera, 1993; Salvo, Alfa, Lo Curto, & Dugo, 1998). In Spain some of the most important olive varieties such as Picual, Hojiblanca and Arbequina from Andalucia and Cataluña have been exhaustively studied (Aparicio, Ferreiro, Cert, & Lazón, 1990; Cert et al., 1999; Graell et al., 1993; Hidalgo et al., 1993; Motilva, Jaria, Bellart, & Romero, 1998). However, despite the economic importance of the Cornicabra olive oils there are no complete and reliable data on its chemical composition and properties in the scientific literature from a large enough number of successive crop seasons to be statistically relevant.

This paper examines the chemical composition of Cornicabra virgin olive oils ($n=181$) from five successive crop seasons (from 1994/95 to 1998/99) and the relationship of that composition with quality and oxidative stability. Several analytical determinations were chosen: quality indices, as defined by EU Regulations (free fatty acid content, peroxide value, spectrophotometric characteristics in the UV region and sensory evaluation); parameters of oxidation processes (oxidative stability, total phenols, tocopherols and chlorophyll and carotenoids pigments), fatty acid and sterol composition.

2. Materials and methods

2.1. Oil samples

Samples of Cornicabra variety virgin olive oil ($n=152$) were collected from industrial oil mills located in the area of Toledo and Ciudad Real (Castilla — La Mancha) during the crop seasons from 1994/95 to 1998/99. Sixty-eight oils were extracted using the dual-phase decanter, 63 with the triple-phase decanter and 12 with a combination of both systems, while nine were processed by the older pressure technique, which has now practically disappeared.

Olive fruits (*Olea europaea*) of the Cornicabra variety were harvested in olive groves of the same area and during the same crop seasons. Olives were randomly picked at industrial optimum ripening stage, according to their skin color. Harvesting was done by hand, using rakes. The olives were put into 20 kg boxes and taken to the pilot plant. Only healthy fruits, without any kind of infection or physical damage, were processed. Twenty-nine virgin olive oils were extracted separately using an Abencor system (Comercial Abengoa, S.A., Sevilla, Spain), as described by Martinez, Muñoz, Alba, and Lazón (1975).

All samples were filtered with anhydrous Na_2SO_4 and stored at 4°C in darkness using amber glass bottles without head space until analysis. A total of 181 Cornicabra virgin olive oils from five successive crop seasons were used for this study.

2.2. Analytical methods

Determination of free acidity, peroxide value, UV absorption characteristics, and fatty acid composition were carried out following the analytical methods described in Regulations EEC/2568/91 and EEC/1429/92 of the Commission of the European Union (EUC, 1991).

Free acidity, given as % of oleic acid, was determined by titration of a solution of oil dissolved in ethanol/ether (1:1) with ethanolic potash.

Peroxide value, expressed in milliequivalents of active oxygen per kilogram of oil (meq/kg), was determined as follows: a mixture of oil and chloroform/acetic acid was left to react with a solution of potassium iodide in darkness; the free iodine was then titrated with a sodium thiosulfate solution.

K_{232} and K_{270} extinction coefficients were calculated from absorption at 232 and 270 nm, respectively, with an UV spectrophotometer (Hewlett-Packard, HP 8452A), using a 1% solution of oil in cyclohexane and a path length of 1 cm.

Oxidative stability, was evaluated by the Rancimat method (Gutiérrez, 1989). Stability was expressed as the oxidation induction time (h), measured with the Rancimat 679 apparatus (Metrohm), using an oil sample of 3.5 g, warmed to 100°C, and an air flow of 10 l/h.

Total phenols and ortho-diphenol compounds were isolated by extraction of a solution of oil in hexane with a water/methanol mixture (60:40), three times. To a suitable aliquot of the combined extracts, Folin-Ciocalteau reagent and sodium molybdate, 5% in ethanol 50% (Merck), were added and the absorptions of the solution at 725 (total phenolic) and 370 nm (*o*-diphenolic components), respectively, were measured. Values are given as mg of caffeic acid per kg of oil (Gutfinger, 1981; Vazquez, Janer, & Janer, 1973).

Tocopherols were evaluated following the AOCS Method Ce 8-89 (AOCS, 1989). A solution of oil in hexane was analyzed by HPLC (HP 1100) on a silica gel Lichrosorb Si-60 column (particle size 5 μm, 250×4.6 mm i.d.; Sugerlabor, Madrid, Spain) which was eluted with hexane/2-propanol (98.5:1.5) at a flow rate of 1 ml/min. A fluorescence detector (Waters 470) with excitation and emission wavelength set a 290 and 330 nm was used.

Chlorophyll and carotenoid compounds (mg/kg) were determined at 472 and 670 nm in cyclohexane, using the specific extinction values, given by the method of Minguez-Mosquera, Rejano, Gandul, Sanchez, and Garrido (1991).

Table 1
Quality indices of commercial Cornicabra virgin olive oils from 1994/95 to 1998/99 crop seasons ($n = 152$ samples)

Quality indices	Mean ±S.D.	Range	Percentiles				
			10	25	50	75	90
Free fatty acid (%)	0.58±0.42	0.15–1.83	0.21	0.27	0.46	0.78	1.09
Peroxide value (meq/kg)	9.6±5.1	3.6–29.6	5.16	6.3	7.9	11.7	16.8
K_{232}	1.610±0.202	1.295–2.765	1.420	1.478	1.558	1.674	1.894
K_{270}	0.134±0.030	0.083–0.241	0.099	0.109	0.130	0.150	0.175
Oxidative stability (h)	61.0±24.6	8.8–143.4	29.3	43.8	60.8	77.7	92.5
Total phenols (mg/kg)	147±59	19–380	75	104	147	177	214
ortho-Diphenols (mg/kg)	7.8±5.5	0.0–27.2	2.0	4.0	6.7	10.1	15.9
α-Tocopherol (mg/kg)	168±36	55–315	127	149	170	183	213
Chlorophylls (mg/kg)	10.0±4.9	1.7–27.1	4.8	6.7	8.9	12.6	17.0
Carotenoids (mg/kg)	7.0±2.2	2.3–14.0	4.6	5.5	6.7	8.3	10.3

For the determination of fatty acid composition, the methyl-esters were prepared by vigorous shaking of a solution of oil in hexane (0.2 g in 3 ml) with 0.4 ml of 2 N methanolic potash, and analyzed by GC with a Hewlett-Packard (HP 6890) chromatograph equipped with a FID detector. A fused silica column (50 m length ×0.25 mm i.d.), coated with SGL-1000 phase (0.25 μm thickness; Sugerlabor), was used. Helium was employed as carrier gas with a flow through the column of 1 ml/min. The temperatures of the injector and detector were set at 250°C whereas the oven temperature was 210°C. An injection volume of 1 μl was used [Regulation EEC 2568/91, corresponding to AOCS method Ch 2-91].

Sterols (%) were determined by a Hewlett-Packard (HP 6890) gas chromatograph with a capillary column (25 m length ×0.25 mm i.d.) coated with SGL-5 (0.25 μm thickness; Sugerlabor). Working conditions were as follows: carrier gas, helium; flow through the column, 1.2 ml/min; injector temperature, 280°C; detector temperature, 290°C; oven temperature, 260°C; injection volume 1 μl [Regulation EEC 2568/91, corresponding to AOCS method Ch 6-91].

Analytical determinations were carried out at least in duplicate.

2.3. Sensory analysis

Sensory analysis of the samples was carried out by 12 selected and trained panellists from the panel of *D.O. Montes de Toledo* (Toledo; in collaboration with the University of Castilla — La Mancha), according to the method described in Annex XII of the European Union Commission (EEC 2568/91). Oils were scored on a nine-point scale, 1 being the value for very poor quality and 9 for optimum quality. For positive attributes, such as fruity and bitterness, a five-point scale was used.

2.4. Statistical analysis

Statistical analysis was performed using the SPSS 9.0 statistical software (SPSS Inc., 1999). Differences were considered statistically significant when probability was greater than 95% ($P < 0.05$).

3. Results and discussion

Results of quality indices, with the exception of sensory analysis, for commercial ($n = 152$) and Abencor ($n = 29$) Cornicabra virgin olive oils from five crop seasons (1994/95 to 1998/99) are shown in Tables 1 and 2, respectively. These tables report the mean value and its standard deviation, range (minimum and maximum) and quartile values (percentiles 10, 25, 50, 75 and 90) to give a better description of the distribution of the values observed for each determination.

Quality and authenticity criteria for various olive oil types are described in detail in the EU Regulations EEC/2568/91 and later modification EEC/656/95. For the majority of the samples analyzed, the values of the analytical parameters fell within the ranges established for the highest quality category 'extra virgin' olive oil.

3.1. Free acidity

Free fatty acid content, as percentage of oleic acid, ranged from 0.15 up to 1.83%, with a median value of 0.46. Only 10% of the oils analyzed showed more than 1% free acidity, the upper threshold limit for the 'extra-virgin' category (EEC Regulations), and none exceeded the limit of 2% for the 'virgin' category. Abencor oils showed a statistically significant lower free fatty acid content, with a maximum value of 0.55%, due to greater care used in the handling and processing of these oils. It is known that the increase of free acidity is mainly due to enzyme activity caused by olive tissue damage (Boskow, 1996b)

3.2. Peroxide value and UV characteristics

Peroxide value, expressed as meq O_2 per kg, presented a median value of 7.9 and a range between 3.6 and 29.6.

Table 2
Quality indices of Abencor Cornicabra virgin olive oils from 1994/95 to 1998/99 Crop Seasons ($n=29$)

Quality indices	Mean ± S.D.[a]	Range	Percentiles				
			10	25	50	75	90
Free fatty acid (%)	0.23±0.11**	0.08–0.55	0.12	0.14	0.20	0.30	0.40
Peroxide value (meq/kg)	6.1±4.5**	1.9–19.1	2.3	3.4	4.6	7.5	15.0
K_{232}	1.665±0.13*	1.438–1.880	1.457	1.531	1.694	1.779	1.855
K_{270}	0.149±0.04*	0.099–0.229	0.109	0.124	0.148	0.165	0.204
Oxidative stability (h)	129.4±46.7**	18.4–193.1	61.2	101.6	127.9	171.8	187.7
Total phenols (mg/kg)	367±121**	180–614	213	286	318	490	557
ortho-diphenols (mg /kg)	29.2±14.0**	10.1–56.6	10.9	16.5	30.1	39.9	49.8
α-Tocopherol (mg/kg)	227±40**	156–311	181	197	216	259	290
Chlorophylls (mg/kg)	17.9±20.0**	1.3–82.6	2.0	5.3	10.3	22.1	51.9
Carotenoids (mg/kg)	11.0±7.4**	2.1–31.5	2.9	5.2	9.9	14.9	22.8

[a] Significant difference with respect to commercial oils: P: * 0.1–0.05; ** <0.001.

Less than 5% of the oils had a peroxide value higher than the upper limit of 20 established for the 'extra-virgin' olive oil; all of them were from the last crop season due to the low temperature registered in December 1998, when the olive fruit was frozen on the trees. The Abencor oils showed a lower peroxide value, in all cases lower than 20 and, in 75% of the cases, lower than 7.5. With respect to the UV characteristics, less than 2% of the oils had K_{232} or K_{270} values higher than the limits established for 'extra-virgin' olive oils.

3.3. Oxidative stability and antioxidants

Results of other analytical determinations, i.e. oxidative stability, total phenols, *o*-diphenols (both expressed as caffeic acid) and α-tocopherol are also reported in Tables 1 and 2.

Oxidative stability of the commercial oils analyzed ranged from a minimum of only 9 h to a maximum of 143 h, with a median value of 61 h. A quarter of the samples exhibited stability in excess of 78 h while, in another quarter, stability was less than 44 h. According to these results and published data on other Spanish varieties (Cert et al., 1999), Cornicabra and Picual are the two Spanish olive varieties whose oils are most stable to oxidation.

A properly prepared Cornicabra olive oil extracted by centrifugation using good quality olives can easily attain stability upward of 100 h, as observed in the oils prepared at the pilot plant of this University: 75% of the Abencor oils showed an oxidative stability higher than 100 h, with a maximum of 193 h (Table 2).

Virgin olive oil contains phenolic substances which affect its stability and flavor. The median content of total polyphenol compounds in the samples analyzed was 147 mg/kg (as caffeic acid), although a wide range of concentrations was observed, from 19 up to 380. Twenty per cent of the oils contained more than 200 ppm of phenols, and 10% contained less than 100 ppm. As was found with respect to oxidative stability, the polyphenol content of the commercial Cornicabra variety (and the Picual) is among the highest of all Spanish olive varieties (Aparicio, Roda, Albi, & Gutiérrez, 1999; Cert, Alba, León-Camacho, Moreda, & Pérez-Camino, 1996). Abencor olive oils showed a much higher concentration of phenols compounds, ranging from 180 up to 614 mg/kg, with a median value of 318 mg/kg. Similar results were observed with *o*-diphenols; the median contents in commercial and Abencor olive oils were 8 and 29 mg/kg, respectively.

The α-tocopherol content in the Cornicabra variety oils studied ranged from 55 up to 315 mg/kg, with a median value of 170 mg/kg for the commercial oils and from 156 up to 311 mg/kg, with a median value of 216 mg/kg, for the Abencor oils. Cornicabra olive oil apparently has a slightly lower tocopherol content than other Spanish varieties (Hidalgo et al., 1993). The tocopherol content is highly variety-dependent, with concentrations ranging from 5 to 300 ppm. Usual values reported for good quality oils vary between 100 and 300 ppm (Angerosa & Di Giovacchino, 1996; Baldioli, Servili, Peretti, & Montedoro, 1996).

A high correlation, consistent through the five seasons, was observed between total phenol content (from 0.873 to 0.964) and oxidative stability by Rancimat (Table 3). The correlation obtained with each of the 5 years studied was higher than with the total number of olive oil samples, with the exception of crop 1996/97. A good direct correlation between oxidative stability and total phenol content, determined by a colorimetric method has been reported by many authors (Gutierrez, Janer del Valle, Janer del Valle, Gutiérrez, Vazquez, & Roncero, 1977; Montedoro, Servili, Badioli, Selvaggini, Miniati, & Macchioni, 1993; Papadopoulus & Boskow, 1991). The correlation between *o*-diphenols and Rancimat stability was slightly lower than with total phenols for commercial virgin olive oils in all cases (i.e. 0.889 and 0.918, respectively, for crop 1997/98 and 0.886 and 0.920, respectively, for crop 1998/99), as observed also by Aparicio et al. (1999), and always slightly higher for

Table 3
Linear regression of oxidative stability versus total phenol content for Cornicabra virgin olive oil samples obtained from five crop seasons ($n = 181$)

Crop	Slope	Intercept	r
1994/95	0.355±0.031	−20.4±5.7	0.919
1995/96	0.288±0.027	11.1±5.8	0.927
1996/97	0.314±0.036	18.9±8.2	0.873
1997/98	0.266±0.010	26.3±3.0	0.964
1998/99	0.346±0.020	4.7±3.9	0.927
Total	0.306±0.011	10.5±2.6	0.902

Abencor oils (i.e. 0.834 and 0.821, respectively, for crop 1997/98 and 0.986 and 0.947, respectively, for crop 1998/99).

The linear regression obtained for oxidative stability versus total phenol content in Cornicabra virgin olive oils is shown in Table 3 and Fig. 1. The five resulting lines, one for each crop season, are almost parallel, with a slope ranging from 0.266 to 0.355.

3.4. Pigments: chlorophylls and carotenoides

The composition and the total natural pigment content of the oils are important quality parameters because they correlate with color, which is a basic attribute for evaluating olive oil quality. Pigments are also involved in autoxidation and photo-oxidation mechanisms (Gutierrez, Garrido, Gallardo, Gandul, & Minguez, 1992; Minguez-Mosquera, Gandul, & Garrido, 1990). Chlorophyll and carotenoids in Cornicabra oils ranged from 2 to 27 ppm and from 2 to 14 ppm, respectively, as expected for virgin olive oils from Spanish varieties (Gandul & Minguez, 1996). The maximum pigment concentrations observed in the Abencor oils were more than double those of the commercial oils. The concentration of pigments correlates well with the stage of maturity of the harvested olive fruits of Picual and Hojiblanca varieties (Gutierrez, Jimenez, Ruiz, & Albi, 1999).

3.5. Fatty acid composition

As expected, no statistically significant differences were observed between fatty acids or sterol compositions of commercial and Abencor olive oils, and therefore the data presented refer to all samples analyzed ($n = 181$). The distribution of fatty acid composition of the oil samples studied is shown in Table 4 and covers the normal range expected for olive oil. Cornicabra oil has a high percentage of oleic acid, with a median value of 80.6 and an interquantile range of 1.3 (difference between 75th and 25th percentiles), and a low percentage of linoleic acid, with a median value of 4.5 and an interquantile range of 0.7. Cornicabra and Picual are the Spanish varieties with the lowest linoleic acid levels

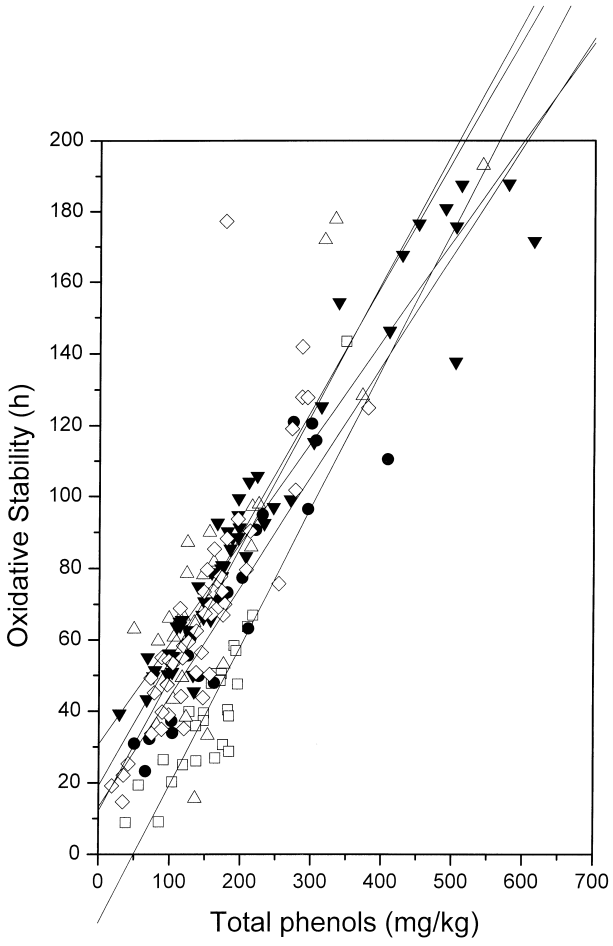

Fig. 1. Linear regression of oxidative stability versus total phenol content for Cornicabra virgin olive oil samples obtained from five crop seasons ($n = 181$). Crop season: □, 94/95; ●, 95/96; △, 96/97; ▼, 97/98; ◇, 98/99.

(Alba et al., 1996). Primary known factors affecting fatty acid composition, especially the oleic acid content, are latitude, climate, variety and stage of maturity of the olives when collected (Ranelli et al., 1997).

3.6. Sterol composition

Sterols are also important constituents of olive oils because they relate to the quality of the oil and are widely used to check authenticity. Sterol composition of the oils is reported in Table 5. All the Cornicabra virgin olive oil samples analyzed showed high campesterol content, with a median value of 4.2 and interquartile range from 4.1 to 4.3, which exceeded the threshold established by EU Regulations (4%). Campesterol content was below this threshold in less than 10% of the oils studied, indicating a peculiarity of this olive oil variety. Another unusual feature of the Cornicabra and Hojiblanca varieties is high Δ5-avenasterol content, with a mean value of 6.2±1.2% (Alba et al., 1996; Aparicio, Sanchez-Navarro, & Ferreiro, 1991). In the

Table 4
Fatty acid composition of Cornicabra virgin olive oils from 1994/95 to 1998/99 crop seasons ($n = 181$)

Fatty acid	Mean ±S.D.	Range	Percentiles				
			10	25	50	75	90
$C_{16:0}$	9.20±0.75	6.99–11.05	8.35	8.62	9.08	9.68	10.30
$C_{16:1}$	0.79±0.11	0.49–1.11	0.66	0.70	0.78	0.86	0.94
$C_{17:0}$	0.06±0.06	0.04–0.07	0.05	0.05	0.06	0.06	0.06
$C_{17:1}$	0.10±0.07	0.08–0.11	0.09	0.09	0.10	0.10	0.11
$C_{18:0}$	3.39±0.31	2.61–4.43	3.06	3.16	3.34	3.62	3.78
$C_{18:1}$	80.4±1.05	76.5–82.5	79.0	79.8	80.6	81.0	81.6
$C_{18:2}$	4.50±0.59	3.07–6.62	3.85	4.09	4.49	4.78	5.26
$C_{18:3}$	0.62±0.08	0.48–0.95	0.53	0.55	0.61	0.67	0.73
$C_{20:0}$	0.51±0.03	0.28–0.62	0.48	0.49	0.51	0.53	0.55
$C_{20:1}$	0.35±0.02	0.29–0.39	0.32	0.33	0.35	0.36	0.37
$C_{22:0}$	0.14±0.02	0.12–0.21	0.13	0.13	0.14	0.15	0.16

Table 5
Sterol composition of Cornicabra virgin olive oils from 1994/95 to 1998/99 crop seasons ($n = 181$)

Sterol	Mean ± S.D.	Range	Percentiles				
			10	25	50	75	90
Cholesterol	0.21±0.07	0.06–0.60	0.13	0.17	0.20	0.23	0.30
Brassicasterol	0.15±0.17	0.00–0.57	0.00	0.00	0.08	0.28	0.39
24-Metilencolesterol	0.14±0.08	0.00–0.31	0.05	0.07	0.13	0.18	0.24
Campesterol	4.21±0.15	3.82–4.64	4.00	4.11	4.22	4.30	4.40
Campestanol	0.25±0.10	0.00–0.43	0.10	0.18	0.29	0.32	0.37
Stigmasterol	0.76±0.24	0.38–1.75	0.50	0.60	0.74	0.89	1.08
Δ7-Campesterol	0.19±0.22	0.00–2.27	0.05	0.08	0.16	0.24	0.36
Δ5.23-Stigmastadienol	0.11±0.08	0.00–0.38	0.01	0.03	0.10	0.16	0.22
Clerosterol	0.89±0.10	0.53–1.05	0.81	0.85	0.90	0.95	1.00
β-Sitosterol	85.3±1.36	81.2–88.2	83.4	84.5	85.5	86.3	87.0
Sitostanol	0.54±0.42	0.00–1.27	0.01	0.03	0.71	0.85	1.00
Δ5-Avenasterol	6.21±1.52	3.34–10.97	4.72	5.14	5.91	6.90	8.31
Δ5.24-Stigmastadienol	0.52±0.22	0.26–1.09	0.29	0.32	0.44	0.70	0.81
Δ7-Stigmastenol	0.25±0.15	0.09–1.30	0.14	0.16	0.20	0.27	0.41
Δ7-Avenasterol	0.26±0.05	0.18–0.44	0.20	0.22	0.25	0.29	0.33
Apparent β-Sitosterol (%)	93.6±0.54	91.7–94.9	92.9	93.3	93.6	94.0	94.3
Total sterols (mg/kg)	1518±162	1014–2055	1306	1429	1523	1600	1716

Table 6
Sensory analysis of commercial Cornicabra virgin olive oils from 1997/98 to 1998/99 crop seasons (n = 72)

Quality indices	Mean ± S.D.	Range	Percentiles				
			10	25	50	75	90
Score	6.7±0.5	6.0–7.3	6.1	6.3	6.7	7	7.2
Fruitiness	2.0±0.7	1.5–2.6	1.6	1.7	1.9	2.2	2.4
Pungency	1.9±0.7	0.9–3.6	1.3	1.6	2.0	2.3	2.7
Bitterness	2.1±0.8	0.9–3.8	1	1.4	1.9	2.4	3.0
Intensity of bitterness[a]	1.8±0.8	0.0–4.6	0.9	1.4	1.7	2.1	2.7

[a] $n = 152$ (from 1994/95 to 1998/99)

literature this compound has been associated with antioxidant activity (Williamson, 1988). All of the olive oil samples contained more than 1000 mg/kg of total sterols, the minimum value established by EU Regulations for the category 'extra virgin' olive oil, with a median value of 1523 mg/kg. In the case of apparent β-sitosterol, about 15% of the samples contained less than the threshold value of 93%.

3.7. Sensory analysis

Results of sensory evaluation are reported in Table 6. These indicated that the total score in 35% of commercial Cornicabra olive oil was lower than the limit of 6.5 established for the 'extra-virgin' category, whereas, the minimum score observed for Abencor oils was 6.8. Total scores ranged from 6.0 to 7.3, with a median value

of 6.7, for commercial olive oils and from 6.8 to 8.0, with a median value of 7.5, for the Abencor oils. This means that, although the majority of the chemical parameters fell within the limits established for the maximum olive oil category, with just 10% of the commercial oils classified as 'virgin' for exceeding free acidity limits, significantly more of these olive oils fail the minimum sensory requirements for the highest category.

Fruity score ranged form 1.5 to 2.6 and from 1.8 to 3.2, for commercial and Abencor olive oils respectively. Pungency and bitterness, two positive attributes peculiar to Cornicabra virgin olive oils, ranged similarly from 0.9 to about 3.7 in commercial oils and from 2.9 up to about 4.2 in Abencor ones. The intensity of bitterness, as defined by Gutierrez, Perdiguero, Gutierrez, and Olias (1992), as compared to sensory bitterness, showed a similar distribution. It should be noted that the parameter 'intensity of bitterness' was determined on all commercial oils ($n = 152$), whereas sensory analysis was performed only in the last two crop seasons ($n = 72$). The oils obtained from green olives were excessively bitter according to the panellists comments. This does not imply rejection of the oil, but if the level of bitterness is too high it could cause some problems for consumer acceptance.

4. Conclusions

The study of the quality characteristics of the Cornicabra virgin olive oil is of great interest to the local industrial sector, the international olive oil business, and the final consumer since, despite the economic importance of this olive oil variety, there were no complete and reliable data on its chemical composition and properties from a large enough number of samples form successive crop seasons to be statistically relevant and representative of the oil production.

The majority of the quality parameters of Cornicabra olive oils fell within the limits established for the 'extra-virgin' olive oil category, with a few exceptions, mainly related to adverse climatic conditions in certain crop seasons. However, Abencor oils showed statistically significant better values for these quality parameters, mainly due to greater care used in the handling and processing of the olives and oils. Moreover, a greater number of the olive oils evaluated fail the sensory minimum requirements for the highest category. These results indicated the importance of advising the industrial sector to further improve the organoleptic quality of the virgin olive oils produced.

The main composition characteristics of Cornicabra olive oils are a high oleic acid and low linoleic acid content, a high campesterol level, exceeding the EU Regulation upper limit, a large total phenol content, and high oxidative stability.

Acknowledgements

The authors wish to express their sincere gratitude to the Spanish Comision Interministerial de Ciencia y Tecnología (CICYT) for supporting this research project (ALI95-0197-CO2-02 and 1FD97-0177).

References

Alba, J., Hidalgo, F., Ruiz, M. A., Martinez, F., Moyano, M. J., Cert, A., Perez, M. C., & Ruiz, M. V. (1996). Características de los aceites de oliva de primera y segunda centrifugación. *Grasas y Aceites, 47*(3), 163–181.

Angerosa, F., & Di Giovacchino, L. (1996). Natural antioxidants of virgin oil obtained by two and tri-phases centrifugal decanters. *Grasas y Aceites, 47*(4), 247–254.

AOCS. (1989). *Determination of tocopherols and tocotrienols in vegetable oils and fats by HPLC. Official Method, Ce 8-89*. AOCS.

Aparicio, R., Ferreiro, L., Cert, A., & Lazón, A. (1990). Caracterización de aceites de oliva vírgenes andaluces. *Grasas y Aceites, 41*(1), 23–39.

Aparicio, R., Roda, L., Albi, M. A., & Gutiérrez, F. (1999). Effect of various compounds on virgin olive oil stability measured by Rancimat. *Journal of Agricultural and Food Chemistry, 47*, 4150–4155.

Aparicio, R., Sanchez-Navarro, M., & Ferreiro, M. S. (1991). Definite influence of the extraction methods on the chemical composition of virgin olive oil. *Grasas y Aceites, 42*(5), 356–362.

Baldioli, M., Servili, M., Perretti, G., & Montedoro, G. F. (1996). Antioxidant activity of tocopherols and phenolic compounds of virgin olive oils. *Journal of the American Oil Chemists' Society, 73*, 1589–1593.

Barranco, D., Rallo, L., Uceda, M., & Hermoso, M. (1994). Fichas varietales del olivo, Cornicabra. *Unaproliva*, *1*(3).

Boskow, D. (1996a). *Olive oil: Chemistry and technology*. Champaign, IL, USA: AOCS Press.

Boskow, D. (1996b). Changes caused by enzymes and oxidation. In D. Boskow, *Olive oil: Chemistry and technology* (pp. 96–100). Champaign, IL, USA: AOCS Press.

Cert, A., Alba, J., Camino, M. C., Ruiz, A., Hidalgo, F., Moreda, W., Moyano, M. J., Martinez, F., Tubaileh, R., & Olias, J. M. (1999). Influencia de los sistemas de extracción sobre las características y los componentes menores del aceite de oliva virgen extra. *Olivae, 79*, 41–50.

Cert, A., Alba, J., León-Camacho, M., Moreda, W., & Pérez-Camino, M. C. (1996). Effects of talc addition and operating mode on the quality and oxidative stability of virgin oils obtained by centrifugation. *Journal of Agricultural and Food Chemistry, 44*, 3930–3934.

Conte, L. S., Carboni, M. F., & Lercker, G. (1993). Gli oli di oliva della Romagna. Nota 1: gli oli della Valle del Lamone. *La Rivista Italiana delle Sostanze Grasse, LXX*, 157–160.

DOCM. (1998, September 18). *Diario oficial Castilla La Mancha*, (pp. 7460–7461).

EUC. (1991). *Characteristics of olive and olive pomance oils and their analytical methods*. Regulation EEC/2568/91 and later modification. *Off. J. Eur. Commun. L248*, 1–82.

Gandul, B., & Minguez, M. I. (1996). Chlorophyll carotenoid composition in virgin olive oils from various Spanish olive varieties. *Journal of Science of Food and Agriculture, 72*, 31–39.

Graell, J., Arbos, J., Duro, V., Clota, J., Torrelles, R., Ciria, R., Puig, J., & Munté, M. (1993, October). Calidad del aceite de oliva virgen D.O. Borjas Blancas. *Alimentación, Equipos y Tecnología,* 97–127.

Grande Covián, F. (1996). Nutrición y valor biológico. In *Enciclopedia mundial del olivo. Consejo Oleícola Internacional* (pp. 345–267). Barcelona: Plaza y Janés Editores SA.

Gutfinger, T. (1981). Polyphenols in olive virgin oils. *Journal of the American Oil Chemists' Society, 58*, 966–968.

Gutiérrez, F. (1989). Determinación de la estabilidad oxidativa de aceites de oliva vírgenes: comparación entre el método A.O.M. y el método Rancimat. *Grasas y Aceites, 40*(1), 1–5.

Gutierrez, F., Garrido, J., Gallardo, L., Gandul, B., & Minguez, M. I. (1992). Action of Chlorophylls on the Stability of virgin olive oil. *Journal of the American Oil Chemists' Society, 69*(9), 866–871.

Gutierrez, R., Janer del Valle, C., Janer del Valle, M. L., Gutierrez, F., & Vazquez Roncero, A. (1977). Relación entre los polifenoles y la calidad y estabilidad del aceite de oliva virgen. *Grasas y Aceites, 28*(2), 101–106.

Gutierrez, F., Jimenez, B., Ruiz, A., & Albi, M. A. (1999). Effect of Olive ripeness on the oxidative stability of virgin olive oil extracted from the varieties picual and hojiblanca and the different compounds involved. *Journal of Agricultural and Food Chemistry, 47*, 121–127.

Gutierrez, F., Perdiguero, S., Gutierrez, R., & Olias, J. M. (1992). Evaluation of the bitter taste in virgin olive oil. *Journal of the American Oil Chemists' Society, 69*(4), 394–395.

Hidalgo, F., Navas, M. A., Guinda, A., Ruiz, A., León, M., Lazón, A., Maestro, R., Janer, M. L., Pérez, M. C., Cert, A., Alba, J., Gutierrez, F., Dobarganes, M. C., & Graciani, E. (1993). La calidad del aceite de oliva virgen: posibles nuevos criterios para su evaluación. *Grasas y Aceites, 44*(1), 10–17.

Lanza, C. M., Russo, C., & Tomaselli, F. (1998). Relationship between geographical origin and fatty acid composition of extra-virgin olive oils produced in three areas of eastern Sicily. *Italian Journal of Food Science, 4*(10), 359–366.

Martinez, J. M., Muñoz, E., Alba, J., & Lazón, J. (1975). Informe sobre utilización del Analizador de Rendimiento Abencor. *Grasas y Aceites, 26*(6), 379–385.

Mínguez-Mosquera, M. I., Gandul, B., & Garrido, J. (1990). Pigments present in virgin olive oil. *Journal of the American Oil Chemists' Society, 67*(3), 192–196.

Mínguez-Mosquera, M. I., Rejano, L., Gandul, B., Sanchez, A. H., & Garrido, J. (1991). Color-pigment correlation in virgin olive oil. *Journal of the American Oil Chemists' Society, 68*(5), 322–337.

Montedoro, G., Servili, M., Baldioli, M., Selvaggini, R., Miniati, E., & Macchioni, A. (1993). Simple and hydrolyzable compounds in virgin olive oil. 3. Spectroscopic characterizations of the secoiridoid derivatives. *Journal of. Agricultural and Food Chemistry, 41*, 2228–2234.

Motilva, M. J., Jaria, I., Bellart, I., & Romero, M. P. (1998). Estudio de la calidad del aceite de oliva virgen de la Denominación de Origen "Les Garrigues" Lleida durante la campaña 1995/96. *Grasas y Aceites, 49*(5-6), 425–433.

Papadopoulus, G., & Boskow, D. (1991). Antioxidant effect of natural phenols on olive oil. *Journal of the American Oil Chemists' Society, 68*, 669–671.

Poiana, M., Giuffré, A. M., Giuffré, F., Modafferi, V., Neri, A., Mincione, B., & Taccone, P. L. (1997). Ricerche sugli Oli di oliva monovarietali. Nota IV. Contributo alla caratterizzazione dell'olio estratto da olive della cv Nocellara messinese. *La Rivista Italiana delle Sostanze Grasse, LXXIV*, 59–71.

Poiana, M., Giuffré, F., Modafferi, V., Giuffré, A. M., Calogero, V., & Mincione, B. (1996). Ricerche sugli Oli di oliva monovarietali. Nota III. Contributo alla caratterizzazione dell'olio estratto da olive della cv Carolea, coltivata in due diversi areali della Calabria. *La Rivista Italiana delle Sostanze Grasse, LXXIII*, 499–509.

Ranelli, A., De Mattia, G., Ferrante, M. L., & Giansante, L. (1997). Incidence of olive cultivation area on the analytical characteristics of the oil. Note 1. *La Rivista Italiana delle Sostanze Grasse, LXXIV*, 501–508.

Russo, C., & Fichera, G. (1993). Caratterizzazione degli oli di pressione della zona a nord dell'Etna. *Industrie Alimentari, XXXII*, 609–613.

Salvador, M. D., Aranda, F., & Fregapane, G. (1998). Chemical composition of commercial Cornicabra Virgin Olive Oils from 1995/96 to 1996/97 crops. *Journal of the American Oil Chemists' Society, 75*(10), 1305–1311.

Salvo, F., Alfa, M., Lo Curto, S., & Dugo, G. (1998). Caratterizzazione di oli di oliva vergine siciliani. Note I. L'Olio di Minuta. *La Rivista Italiana delle Sostanze Grasse, LXXV*, 325–336.

SPSS Inc. (1999). *SPSS 9.0 for Windows*.

Vázquez, A., Janer, C., & Janer, M. L. (1973). Determination of total polyphenols in olive oils. *Grasas y Aceites, 24*, 350–357.

Williamson, E. (1988). *The antioxidant activity of Δ5-avenasterol*. PhD thesis, University of Reading, UK.