Integral Centrifuges for Olive Oil Extraction. The Qualitative Characteristics of Products

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ABSTRACT: Oil extraction experiments with three olive varieties (Coratina, Nebbio, and Grossa di Cassano) were carried out to compare the two-phase centrifugal decanter with conventional three-phase equipment. The results showed that the two-phase centrifugal extractor renders better qualitative characteristics in the oils, which were comparable to pressed or filtered oils. The two-phase decanter product exhibited higher contents of polyphenols, *ortho*-diphenols, hydroxytyrosol, tocopherols, *trans*-2-hexenal and total aromatic volatile substances. Furthermore, the oils received a higher sensorial score and were characterized by higher values of oxidative stability and campesterol/stigmasterol ratio; lower values of turbidity, alcohol index and chromatic indices; lower content of pigments, steroid hydrocarbons, stigmastatriene, waxes and aliphatic and triterpenic alcohols.

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KEY WORDS: Integral centrifuges, olive oil extraction, qualitative characteristics of oil.

Because of problems with the washing away of oil in continuous extracting plants, the adoption of centrifuging systems in industrial olive processing did not occur until 1992 when the two-phase decanters that allow centrifugation of olive paste without dilution with hot water were developed .

A three-phase decanter can be modified easily into a two-phase centrifuge in about half an hour. Thus, with such integral extractors, the oil-mill can shed its old negative image of the so-called "trappeto" and assume all of the characteristics of a real industry.

In this report, the results of a qualitative comparative study between a three-phase centrifuge and a two-phase unit are given. In a previous note (1), we reported the quantitative data, which showed the following: (i) the extraction outputs of a two-phase unit were comparable and sometimes even superior to the three-phase system; (ii) the consumption of hot water to dilute the pastes, as well as the consumption of electric energy to warm the processing water, was reduced to a minimum; (iii) the vegetable water (olive juice), for the most part, was not separated from the husk, which was consequently richer in water and oil. The usual industrial processing of the husk had been difficult and expensive for this reason; however, this was not a problem for the two-phase centrifugal decanters. The quantity of liquid effluent produced was low, and therefore, filtering costs were minimal; (iv) the quantity of olives processed per hour was enhanced, avoiding the use of added water in the process, so that the oily mass to be centrifuged was reduced.

Furthermore, as will be pointed out in this report, the oils produced by integral centrifugation were of better quality, were characterized by a longer shelf life, and were richer in natural antioxidants. They were comparable to and competitive with pressed or percolated oils.

EXPERIMENTAL PROCEDURES

To execute the experiment, three varieties of olives (Coratina, Nebbio, and Grossa di Cassano) were processed at an industrial level. For each variety, a homogeneous sample of 1.8 MT of olives was processed, 0.9 MT with a two-phase decanter and 0.9 MT with a three-phase decanter. Each half was divided into three equal parts, which were processed and tested.

Oil was extracted from the olives by applying the same industrial cycle and using the same operating conditions adopted in previous reports (2,3). The only variation was that the paste was not diluted with water when the two-phase decanter was used. The steps of the two technological processes were as follows: (i) removal of leaves from olive lots; (ii) milling of drupes by a mobile hammer crusher whose grating holes had a diameter of 6 mm; (iii) kneading of paste for 60 min at 28°C; (iv) fluidification of oily paste with water (400 L/h) heated to 28°C when a three-phase decanter was used. With the use of a 400-S type Rapanelli horizontal centrifuge, it was possible to process the olives according to a two- or three-phase cycle by using specific head plates. The plates used in the integral decanter version led to produce oil and husk and only a small quantity of vegetable water. This effluent represents the excess water contained in the processed drupes. The quantity of paste centrifuged per hour was 0.6 MT; and (v) separation of the obtained oily must into oil and water by means of an automated discharge centrifuge.

During testing, samples of the olives, along with by-products and oils, were taken. On oils produced, the following analytical-instrumental determinations were effected:

(i) High-resolution gas chromatographic (HRGC) analysis of head-space aromatic volatile fraction at 37°C, by means of a 25-m length carbowax 20 M capillary column, after its ex-

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traction by stripping, carried out by means of a nitrogen stream, and subsequent entrapping with activated carbon from which the components in question were then eluted with diethyl ether. Internal standard was 1-nonanol (4,5); (ii) high-pressure liquid chromatographic (HPLC) analysis of tocopherols by means of a direct-phase column with a hexane–isopropyl alcohol eluent and an ultraviolet (UV) detector at 295 nm wavelength (6); (iii) HRGC analysis of phenolic fraction with a 15-m long fused-silica capillary column, after its extraction with absolute methyl alcohol, and subsequent purification of prepared methanolic extracts by means of a chromatographic column filled with celite and polyclar; elution first with a hexane–acetone mixture (97:3, vol/vol) and then with absolute methanol, followed by the preparation of silyl derivatives with trimethylsilylfluoroacetamide. Internal standard was resorcinol (9); (iv) HRGC analysis of steroid hydrocarbons with a 25-m long, 0.25-mm internal diameter capillary column, having a methylsilicone-type stationary phase, after their separation with a chromatographic column filled with activated silica 60 and tamponed at the bottom with glass wool, and subsequent recovery with a hexane eluent. Internal standard was cholestadiene (8); (v) HRGC analysis of waxes by means of a 10- to 15-m long 0.25- to 0.32-mm internal diameter glass or fused silica capillary column, internally covered with SE-52 or SE-54 liquid, after their separation with a 70- to 230-mesh hydrated silica gel column and (99:1, vol/vol) *n*-hexane/diethyl (99:1, vol/vol) ether eluent. The first eluted fraction (~140 mL), with a polarity lower than the triglycerides, was evaporated to dryness, then recovered with *n*-heptane and finally analyzed. Internal standard was lauryl arachidate (10); (vi) HRGC analysis of fatty acids (methyl ester fatty acids) by means of a 25-m long 0.2 to 0.8-mm internal diameter, having a polyglycol type stationary phase (9); (vii) HRGC analysis of sterol fraction and triterpene dialcohol, by means of a 20- to 30-m long 0.25- to 0.30-mm internal diameter glass or fused-silica capillary column, internally covered with SE-52 or SE-54 liquid (0.10–0.30 μm uniform thickness). The oil is first saponified by an ethanolic potassium hydroxide solution; then, its unsaponifiable matter is extracted by diethyl ether, and the components in question are separated with a basic silica-gel plate chromatography technique with benzene–acetone (95:5, vol/vol) eluent. Subsequently, the recovered sterols and dialcohols are transformed into trimethyl silylethers (TMSE), which are finally analyzed. Under UV light, a mild 2,7-dichlorofluoroscein basic alcoholic solution is used to reveal on the plates the component bands to be analyzed. A pyridine-hexamethyl-disilazane–trimethyl–chlorosilane (9:3:1, vol/vol/vol) mixture is used as a silanizing reactant. Internal standard was α-cholestanol, 0.2 wt% in chloroform (10); (vii) HRGC analysis of aliphatic and triterpene alcohol fraction. The analytical method applied is similar to that relating to sterols except that alcohol bands instead of sterols with the thin-layer chromatography step are recovered. Internal standard was arachidilic alcohol (10).

Other analytical observations to which the oils were submitted (11,12) included free acidity, peroxide index, B.M. Watts and R. Major's carbonyl index, total polyphenols, *ortho*-diphenols (by colorimetry), turbidity (NTU), UV spectrophotometric indices, ΔK, J.P. Wolff's ratio (R), *a* and *b* chlorophylls, *a* and *b* phaeophytins, chlorophyllic color index, and carotenoid color index.

To determine the color of oils objectively, the IEC method with tri-chromatic coordinates was used to assess, through transmittance measurements, brightness or value (h%), purity or chroma (σ%), and hue (λd) in addition to the above chromatic indices. Naudet's integral color index and color ratio (A446/ A668) of oils were also evaluated.

The product was then submitted to sensorial testing (tasting) by the panel method, and the relative scores were statistically compared (difference test). The panelists also considered the nuances as well as the typical characteristics and any off-flavors (if present), in addition to sensorial attributes such as sweet, bitter, or pungent. Furthermore, data were acquired on the shelf life of oils, (their oxidative stability) i.e., the induction time of peroxidation reactions (by use of Rancimat instrument, which automatically applies the accelerated Swift's test).

Finally, the total quality of oils was evaluated by calculating: (i) the global quality index (GQI1) from the linear equation proposed by the IOOC (13). This equation is based on four canonical variables, one of which is the sensorial score to which the highest ponderal coefficient is attributed, resulting in this variable having the largest relative weight on overall oil quality; (ii) the global quality index (GQI2) from the algorithm developed by M. Solinas (14), in which a fifth variable is included, i.e., the total polyphenols. However, this may be considered redundant because it is correlated with the sensorial score.

RESULTS AND DISCUSSION

The results obtained, relating to the analytical characteristics of the oils, are given in Tables 1 through 4.

Acidity, peroxide index, UV spectrophotometric indices, Watts-Majors's index, turbidity. In general, results indicated that these qualitative parameters and indices were not influenced or were only slightly influenced by the type of decanter adopted for the centrifugation of olive pastes. The only exception appeared to be the turbidity values, which were frequently lower in the oils produced without hot water to dilute the pastes.

Total polyphenols, ortho-diphenols, tyrosol, hydroxytyrosol, tocopherols. The oils obtained after extraction by the two-phase centrifugal system always exhibited (for all processed olive varieties) higher contents of polyphenols and *ortho*-diphenols. The phenolic concentration of the oils sometimes rose to 100% and, with some varieties, to 200%. When water is added to the pastes, which are rich in polyphenols (which are soluble in both water and oil), a significant amount of such constituents is carried away from the oily phase because of partitioning between two nonmixable liquids. Even the concentrations of single phenols (hydroxytyrosol), α-tocopherol and of total tocopherols were frequently higher when the pastes were processed by the integral cen-

TABLE 1
Analytical Characteristics of Oils Extracted by a Two- or Three-Phase Decanter

	Coratina		Nebbio		Grossa di Cassano	
	2 phases	3 phases	2 phases	3 phases	2 phases	3 phases
Acidity (% oleic acid)	0.25	0.27	0.33	0.34	0.22	0.23
Peroxide index (meq O_2kg)	12.0	14.6	9.1	9.8	8.0	8.8
Carbonyl index - WMI(E/Y)	4.10	4.13	4.98	4.83	4.97	4.34
Total polyphenols (caffeic acid), mg/L	328	270	272	129	183	60
O-diphenols (caffeic acid), mg/L	226	178	188	92	121	43
Rancimat stability (h)	16.9	15.1	15.5	10.0	13.4	8.3
Turbidity, NTU	980	690	880	1180	340	1880
K232	1.699	1.734	1.510	1.430	1.347	1.260
K270	0.144	0.148	0.091	0.083	0.089	0.091
$\Delta K \times 10^3$	–7	–8	-6	–4	–6	–4
R	11.8	11.7	16.6	17.1	15.1	13.9
Panel test (score)	8.1	7.8	7.7	7.2	7.5	6.8
Global quality index (GQI1)	7.9	7.4	8.0	7.6	8.0	7.3
Global quality index (GQI2)	36.9	34.7	38.1	35.6	37.3	34.5
Chlorophylls and phacophytins, mg/kg	19.0	23.2	7.6	7.3	7.4	8.7
Chlorophyllic color index	105.1	131.1	29.3	32.5	31.8	36.5
Carotenoid color index	241.6	600.0	106.0	117.0	138.6	145.9
Brightness, %	62.6	61.5	72.9	68.7	67.4	65.0
Chroma, %	98.5	98.7	85.6	81.3	89.6	91.2
Hue, nm	578	578	577	577	578	578
Color ratio (A446/A668)	2.5	2.5	3.9	3.8	4.6	4.2
Integral color index	20.0	20.8	11.8	13.6	15.4	17.1
Tyrosol, mg/kg	11.4	21.0	8.7	5.0	1.1	1.2
Hydroxytyrosol, mg/kg	7.3	12.3	14.4	6.1	2.0	1.0
Tocopherols, mg/kg	173.1	188.7	159.0	148.3	164.8	147.5
α-Tocopherol, mg/kg	172.4	187.8	158.8	148.2	164.7	147.1
γ-Tocopherol, mg/kg	0.7	0.9	0.2	0.1	0.1	0.4
Steroid hydrocarbons, mg/kg	0.38	0.77	0.62	0.30	0.37	0.06
Campestadiene, mg/kg	0.03	trace	trace	trace	trace	trace
Stigmastatriene, mg/kg	0.28	0.32	0.26	0.23	trace	0.04
Stigmastadiene, mg/kg	0.07	0.45	0.36	0.07	0.37	0.02
Total waxes, mg/kg	39	100	118	173	104	79
Waxes C_{40}, mg/kg	8	15	56	55	22	21
Waxes C_{42}, mg/kg	13	25	30	40	34	27
Waxes C_{44}, mg/kg	5	19	13	25	15	11
Waxes C_{46}, mg/kg	13	41	19	53	33	20

trifugal system. Among the olive varieties considered, Coratina produced an oil with the highest phenol and tocopherol concentrations. Nebbio was richer in polyphenols but poorer in tocopherols compared with Grossa di Cassano.

Panel test, Rancimat stability, global quality indices (GQI1 and GQI2). All oils produced by the two-phase centrifugal decanter received a higher sensorial score. Even oxidation resistance was greater, so that a positive influence of the two-phase continuous system on the shelf life of the product could be hypothesized. These parameters are known to correlate with the phenolic concentration (noted above). Similarly, the global quality index values appeared higher when the nondiluted oily pastes were centrifuged. Oil derived from the Coratina variety was the most resistant to oxidation and received the highest sensorial score. However, Nebbio (followed by Grossa di Cassano) yielded oils with the highest global quality index values.

Aromatic volatile fraction. A total of 21 volatile substances were identified in the oils. Although the oils were all extra-virgin and without defects, they all contained minimal quantities of volatile substances, a normal occurrence but one which has a negative influence on their aroma. However, *trans*-2-hexenal (the most important component), with its pleasantly herbaceous aroma, along with the phenolic substances, had the greatest influence on the flavor of the oils. The concentrations of this aldehyde in oils obtained from the Coratina variety were significantly higher when the two-phase centrifugal decanter was adopted. However, oils produced from the other two varieties had a slightly lower *trans*-2-hexenal content when the innovative decanter was used. Nevertheless, it was fundamentally comparable to that of the reference oil. Instead, the total content of aromatic volatile substances of the oils was frequently higher when the olives were processed by the nonconventional decanter. Because no diluting water was used, consequently, water-soluble volatile components (alcohols and other substances) were not carried away (15).

Chromatic characteristics, lipochrome contents of oils, spectrophotometric curves in the visible light zone between

TABLE 2
Aromatic Volatile Substances (mg/kg) Identified in Oils Extracted by a Two- or Three-Phase Decanter

	Coratina		Nebbio		Grossa di Cassano	
	2 phases	3 phases	2 phases	3 phases	2 phases	3 phases
n-Octane	1.2	3.0	6.0	8.0	1.8	2.9
Etyl-acetate	1.6	1.9	3.4	5.2	3.3	2.4
2-Methyl-butyraldeyde	4.3	1.3	trace	6.4	9.0	7.0
3-Methyl-butyraldeyde	4.0	0.9	2.2	7.7	7.8	6.0
Ethanol	45.4	40.0	12.5	59.3	36.4	36.3
3-Pentanone	62.3	33.3	13.9	16.8	16.7	27.9
1-Penten.3one	33.4	28.9	25.6	19.3	25.1	30.2
Hexanal	24.4	27.4	28.6	21.2	27.2	31.3
Isobutyl alcohol	3.4	1.6	6.8	6.4	8.9	5.1
trans-2-pentenal	4.8	3.8	2.3	1.9	3.1	2.9
1-Penten-3-ol	67.5	46.2	25.8	16.5	26.5	23.7
Iso amyl alcohol	7.8	3.5	20.5	20.1	27.6	11.8
trans-2-hexenal	804.7	620.8	104.4	121.5	154.6	192.3
n-Amyl alcohol	1.6	1.3	1.4	1.4	trace	trace
2-Penten-1-ol	36.4	22.9	16.4	9.5	16.8	14.1
1-Hexanol	43.4	26.0	12.2	12.8	10.8	10.4
3-Exen-1-ol(cis?)	12.8	19.2	48.0	27.6	25.2	31.8
trans-2-hexenol	72.2	66.6	10.0	10.6	9.6	16.6
Acetic acid	6.1	4.7	9.3	6.2	5.4	5.6
1-Octanol	5.2	4.8	4.7	4.4	4.0	4.0
2-Butanol	16.2	5.2	50.1	3.5	3.0	3.9
Total volatile substances	1341.9	1035.8	462.2	430.9	488.3	534.3

TABLE 3
Fatty Acid and Alcoholic Composition of Oils Extracted by a Two- or Three-Phase Decanter

	Coratina		Nebbio		Grossa di Cassano	
	2 phases	3 phases	2 phases	3 phases	2 phases	3 phases
Myristic acid (C_{14},0), %	trace	trace	trace	trace	0.1	trace
Palmitic acid (C_{16},0), %	9.5	9.5	12.9	12.4	10.3	10.1
Palmitoleic acid (C_{16},1), %	0.4	0.4	1.0	1.0	0.6	0.6
Eptadecanoic acid (C_{17},0), %	trace	trace	trace	trace	0.2	trace
Eptadecenoic acid (C_{17},1), %	0.1	trace	0.1	0.1	0.1	0.1
Stearic acid (C_{18},0), %	2.8	2.9	2.2	2.2	2.9	2.7
Oleic acid (C_{18},1), %	79.2	79.1	74.1	74.4	76.4	77.2
Linoleic acid (C_{18},2), %	6.3	6.5	8.6	8.6	7.6	7.7
Linolenic acid (C_{18},3), %	0.8	0.7	0.5	0.6	0.8	0.9
Arachidonic acid (C_{20},0), %	0.4	0.5	0.3	0.3	0.3	0.3
Eicosenoic acid (C_{20},1), %	0.4	0.3	0.2	0.3	0.3	0.3
Behenic acid (C_{22},0), %	0.1	0.1	0.1	0.1	0.3	0.1
Lignoceric acid (C_{24},0), %	trace	trace	trace	trace	trace	trace
Saturated/unsaturated	0.15	0.15	0.18	0.18	0.16	0.15
Oleic/linoleic	12.6	12.2	8.6	8.6	10.0	10.0
Alcoholic index	0.10	0.04	0.50	0.56	0.11	0.23
Aliphatic alcoh., mg/100 g	2.0	6.0	11.3	16.8	8.6	7.3
1-Docosanol (C_{22}), mg/100 g	0.4	0.7	3.4	4.3	1.3	0.8
1-Tetracosanol (C_{24}), mg/100 g	0.6	1.1	3.8	5.0	2.7	2.1
1-Hexcosanol (C_{26}), mg/100 g	0.6	2.7	3.4	5.3	3.5	3.4
1-Octacosanol (C_{28}), mg/100 g	0.4	1.5	0.7	2.2	1.1	1.0
Triterpenic alc., mg/100 g	41.2	59.3	85.8	90.5	57.0	44.0
β-Amyrin + butyrospermol, mg/100 g	7.9	11.9	7.8	10.7	10.1	9.6
Cycloartenol, mg/100 g	19.4	34.2	22.8	23.4	13.8	10.3
24-Methylenecycloartanol, mg/100 g	13.9	13.2	55.2	56.4	33.1	24.1

400- and 700-nm. The oils produced by the integral centrifugal system were characterized by lower chlorophyllic and phaeophytinic pigment contents, and by lower values of the chlorophyllic color index and the carotenoid color index. The values of σ% (chroma), in agreement with the previous parameters, tended to be lower, whereas the h% (brightness) values were regularly and significantly higher. Finally, the λd (hue) values did not differ from the reference oil, indicating that

TABLE 4
Sterol and Triterpene Dialcohol Content (values %) of Oils Extracted by a Two- or Three-Phase Decanter

	Coratina		Nebbio		Grossa di Cassano	
	2 phases	3 phases	2 phases	3 phases	2 phases	3 phases
Cholesterol	0.2	0.2	trace	0.1	0.3	0.2
Brassicasterol	trace	trace	trace	trace	0.1	trace
24-Methylenecholesterol	0.3	0.2	0.3	0.2	trace	0.1
Campesterol	3.6	3.5	2.6	2.5	3.3	3.4
Campestanol	0.6	0.6	0.2	0.3	0.4	0.4
Stigmasterol	0.5	0.6	0.4	0.5	0.8	0.9
Δ^7-Campesterol	0.1	0.3	trace	0.1	0.2	0.3
Δ5-23-Stigmastadienol	trace	trace	trace	trace	trace	trace
Chlerosterol	1.0	1.1	0.9	0.8	1.3	1.1
β-Sitosterol	83.9	82.2	77.0	77.4	86.2	89.3
Sitostanol	1.6	1.6	1.4	1.2	1.4	1.2
Δ5-Avenasterol	7.4	7.9	16.1	15.9	4.4	1.7
Δ5-24-Stigmastadienol	0.2	0.4	0.2	0.1	0.8	0.8
Δ7-Stigmastenol	0.3	0.5	0.7	0.6	0.5	0.4
Δ7-Avenasterol	0.3	0.9	0.2	0.3	0.3	0.2
Triterpene dialcohols	2.9	3.2	1.0	0.9	2.5	0.9
Total β-sitosterol	94.1	93.2	95.6	95.4	94.1	94.1
Total sterol, mg/100 g	86.1	90.4	193.4	204.1	125.7	113.2
Campesterol/stigmasterol	7.2	5.5	6.0	4.7	4.2	4.0

both oil types were characterized by a yellow color, which clearly prevailed over green. This was confirmed by values of the color ratio (between chlorophyll absorbance and carotenoid absorbance). Such values, in general, varied from 2.5 to 4.6 and were not dissimilar in the two oil types compared (two- or three-phase centrifuged oils) when they originated from the same olive variety. The visible-light spectra between 400- and 700-nm) and the values of the integral color index gave information that agreed with that obtained by examining the quoted above chromatic parameters and indices.

Steroid hydrocarbons and waxes. The determination of steroid hydrocarbons has recently been required by international commercial regulation, to reveal adulteration of olive oils with other vegetable oils whose sterol content had been modified. In fact, this procedure appears to be more reliable than the determination of *trans*-fatty acids (enacted by the previous norm).

The analytical data relating to the steroid hydrocarbon fraction obtained by the performed tests showed the following: (i) the oils contained traces of campestadiene. In only one case was it possible to detect this substance, i.e., in oil obtained from the Coratina olive variety that was processed by the two-phase decanter; (ii) in two cases, stigmastatriene exceeded the limit value (0.10 mg/kg); (iii) the amounts of stigmastadiene and stigmastatriene were comparable; (iv) the stigmastatriene content in oils obtained with the two decanter types did not seem to differ significantly. However, the contents of this substance, as well as the total steroid hydrocarbon contents, frequently appeared to be slightly higher when the conventional centrifuge was used; (v) in contrast, the stigmastadiene content was frequently higher in oils produced by the two-phase decanter; (vi) oil produced from the Grossa di Cassano variety was characterized by the lowest total steroid hydrocarbon contents.

The analytical tests for waxes (which are similarly correlated with the genuineness of oil) gave the following information: (i) C_{40}, C_{42}, C_{44}, C_{46} and the total wax were frequently higher in oils produced by traditional centrifugation (diluting the oily paste with hot water). Evidently, the fluidificating liquid provokes the mobility of these substances by means of mechanical actions, permitting their major contact with the oily phase and leading to their solubilization in higher quantity; (ii) the olive variety influenced the wax contents of oils. In fact, Nebbio gave an oil richest in waxes, followed by Grossa di Cassano and Coratina.

Composition of sterol fraction, campesterol/stigmasterol ratio, triterpene dialcohols. The analytical data collected suggested the following: (i) the campesterol/stigmasterol ratio (believed to be an index of quality) in oils obtained by the integral centrifugation of pastes was constantly higher; (ii) the sterol fraction composition was significantly influenced by the processed olive varieties; (iii) the Δ7-stigmastenol percentage in oils originating from the Nebbio variety, slightly exceeded the 0.5% value, which is the maximum limit; (iv) the total sterol concentration in oil produced from the Coratina variety did not reach 1 g/kg, which is the minimum limit set by the EC norm.

Aliphatic and triterpene alcohol fraction. Alcohol index. The following observations were made regarding the composition of the alcohol fraction of oils: (i) in oils produced by the two-phase centrifugation system, the alcohol index (which is also a quality index and with which it is inversely correlated) was frequently lower; (ii) the processed variety of olives also greatly influenced the composition of the alcohol fraction of the oils; (iii) the total aliphatic and triterpenic alcohol contcentrations in oils obtained by the two-phase decanter were frequently higher.

Fatty acid composition. The fatty acid composition of oils was affected by the olive variety but not by the paste centrifuging system.

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