

Research Article

Influence of olive crushing methods on the yields and oil characteristics

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In the last years, metallic crushers substituted granite stone mill with some variations in the organoleptic oil characteristics. To control the influence of the crushing method on the yield and oil quality, the olive pastes were obtained using three different ways: (i) new metallic crusher at mobile knives; (ii) granite stone mill; (iii) double olive crushing by the metallic crusher and the granite stone mill. With the aim to ascertain the useful use of a new metallic crusher (at mobile knives), experimental tests were carried out in an industrial oil mill. This oil mill is equipped by a centrifugal decanter generating two oil flows: first and second extraction (recovery) oils. The results showed that the yields obtained by different methods were satisfactory. No statistically significant differences have been observed in terms of oil yield and quality when different crushing devices were used. All first extracted oils are extra virgin with similar organoleptic characteristics, especially for the fruity intensity and for the bitter and pungent taste, as confirmed by the composition of volatile substances and the content of phenolic oil compounds. The recovery oils (second extraction oils) showed, in contrast to first extraction oils, a more intense green colour and a higher content of total phenols.

Practical applications: Processing of sound olives with the right ripening grade and good quality allows to easily obtain an extra virgin olive oil, with commercial qualitative parameters according to the European Union requirements. However, different olive crushing systems affect the concentrations of some compounds responsible of aroma and taste (phenolic compounds). The use of the more violent metallic crushers facilitates obtaining oils with total phenol content higher than when using a stone mill. Here we used a particular metallic crusher (at knives) that, however, is suitable to replace the granite stone mill when a less pungent and bitter oil is required.

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1 Introduction

Continuous centrifugation systems are widely used as mechanical extraction method in virgin olive oil production, involving, in many cases, the substitution of granite stone mill, used in the past for the preparation of the olive paste,

with different metallic crushers which are able to operate at high speed.

The traditional olive oil mill stone, even with the biggest size version, has 1500–1600 kg of hourly production capability. This amount results not enough to feed the new continuous centrifugal decanter, having a large capacity and increasingly spread in Italy.

This kind of transformation, driven by the need to increase the oil mill production capacity and reduce the man working cost, has however controversial consequences: another look of the traditional olive oil mill, always characterised and identified with granite millstones slowly rotating,

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but also a change in oil properties such as organoleptic characteristics, which can be differently accepted by the producers and consumers [1]. In particular, in the olive oil paste preparation procedure, the crushing operation, more than malaxation operation, heavily affects the production yield and the olive oil quality, as previously described Ranalli *et al.* [2, 3], Di Giovacchino *et al.* [4, 5], Angerosa and Di Giacinto [6], Alloggio *et al.* [7], Caponio *et al.* [8], even if with contrasting results. These studies, almost un-animously, allow to demonstrate that the excessive strength of the olive crushing influences the content of the phenolic substances and the organoleptic characteristics of olive oil.

Some authors [6, 7], using the 3-phases centrifugal decanter method, compared the qualitative properties of oils extracted from olive pastes obtained by the stone mill and by the metallic crushers (both discs and fixed-hammers). They found that the use of the fast and stronger metallic crushers, compared to slow and soft action of the stones, allows to obtain an higher content of phenols and, therefore, bitter and pungent oils.

In particular, the industrial oil mills operating with cultivars which give naturally very bitter and pungent oils work by a preliminary olive crushing using stone mill (for a time not more than 3–4 min) followed by a second crushing with the metallic crusher, known as ‘finisher’, that optimises the paste to obtain satisfactory oil yield by the centrifugal decanter. This particular method of olive crushing reduces the oil phenolic content that depends on the less violent crushing process of the mill stone.

With the aim to assess the efficiency of metallic crushers, in particular a recently introduced one with mobile-knives equipped with a hole grid, experimental tests were carried out in an industrial mill equipped with a centrifugal decanter for the oil extraction from olive paste prepared with the above mentioned metallic crusher or with stone mill or with the combination of the two olive crushing methods.

The target was to detect any difference in yield or oil quality as effect of crushing device being used.

2 Materials and methods

2.1 Samples

The tests were carried out using two olive batches, the first one constituted by a mixture of Ogliarola di Bitonto (30%) and Leccino (70%) cultivars and the second one constituted by a mixture of Peranzana (80%) and Leccino (20%) cultivars.

2.2 Olive oil mill description

The experimental tests were carried out in an industrial oil mill, near Chieti (Abruzzo, Italy), working by 3-phases centrifugal decanter at oil recovery and water saving of Rapanelli firm (Andreassi’s mill). The olive mill had a metallic crusher, at mobile knives and equipped with a grid with

holes (8 mm of diameter), and a large size granite millstones (with two stones) and also with suitable instruments to make all technological operations required for the follow oil extraction steps:

- (i) olives weight and subsequent operation of leaf removal and washing;
- (ii) olive crushing by the stone mill or by the metallic crusher;
- (iii) malaxation of olive paste at 27°C;
- (iv) oil separation by centrifugal decanter, fed with 900 kg/h of olive paste and with the addition of 200 l/h of water. The decanter allows to obtain a vegetable water flow and two oil flows, different for quantity and quality, converging in the same collection tank of liquid phases, at the below of the decanter, split in two areas (one for the water and another one for the oils). These two types of oils showed qualitative and quantitative differences. In particular, the first oil (1° oil) represents a 94–96% of the total extracted oil, while the second one (2° oil), called ‘recovered oil’ also, represents a 4–6% of the total oil. The first oil (1° oil) is qualitatively better than the second oil (2° oil). The two oils were sampled directly at the exit from the decanter, since, in practice, they were mixed and together sent to the vertical centrifuge separator;
- (v) sending of oil and vegetable water to the vertical centrifuge separator;
- (vi) weight of the total extracted oil.

In Figure 1 is reported the diagram of process where it is possible to see the streams of the products (two oils), and by-products: the decanter separates the paste into solid phase ‘pomace’ and above that allows to obtain a vegetable water flow (OMW of OVW) and two oil flows 1° and 2°.

2.3 Experimental procedure

In order to verify the influence of crushing method on the yield and on oil quality, the technological tests were performed according to the following thesis:

- (i) olive crushing by the metallic crusher at mobile knives and subsequent crushing of the obtained olive paste by a granite stone mill for 20 min; olive paste malaxation for 45 min at 27°C;
- (ii) olive crushing by the metallic crusher at mobile knives and subsequent olive paste malaxation for 60 min at 27°C;
- (iii) olive crushing using only the granite stone mill for 30 min; olive paste malaxation for 40 min at 27°C.

The different malaxation time used in the experimental procedures in this research depends on technological considerations. When the olive crushing is carried out by granite millstones, the malaxation of olive paste is not very important

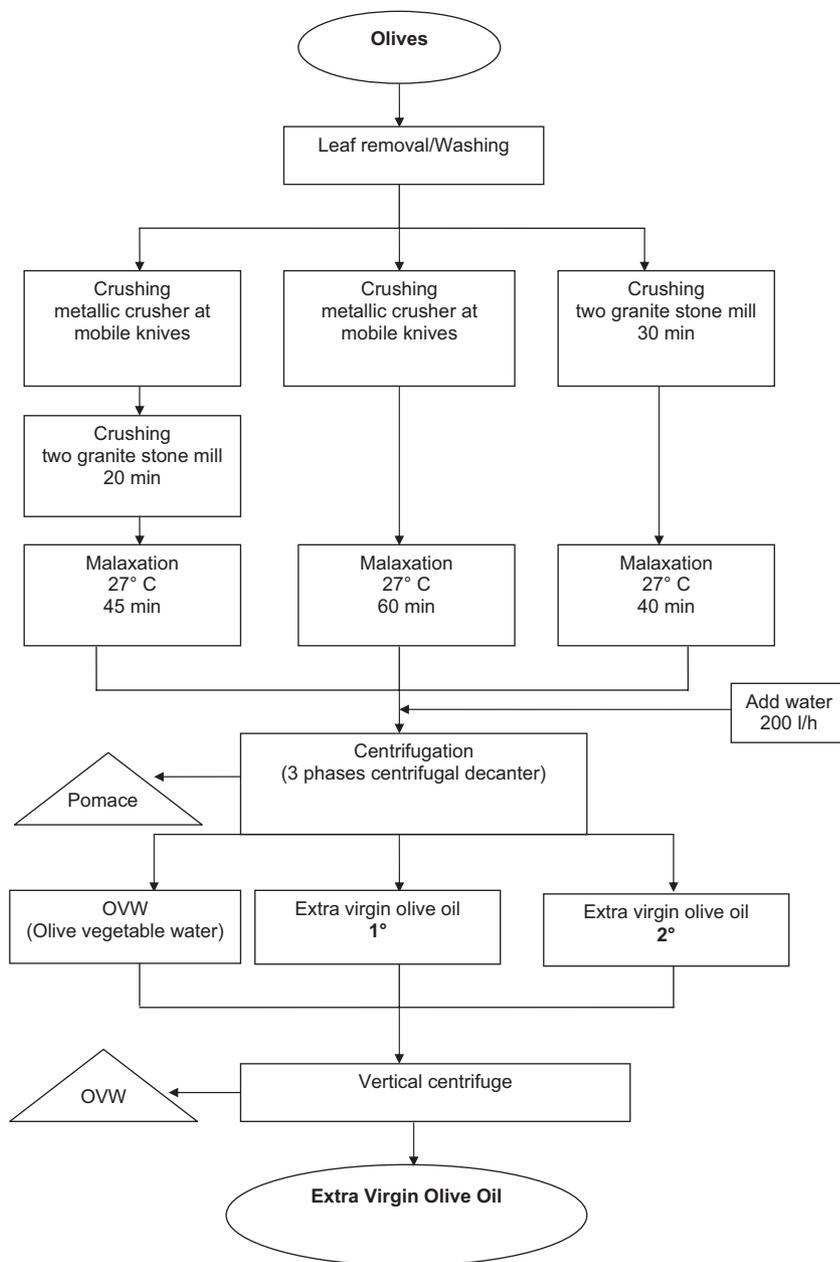


Figure 1. Diagram of olive process by different crushing methods.

because the slow movement of olive paste during the crushing process represents a partial malaxation. For this reason, the malaxation time for the first and third thesis is 40–45 min instead of 60 min used when the olive crushing is carried out by metallic crusher at mobile knives.

2.4 Sensory analyses

The organoleptic characteristics of the oils were evaluated by the CRA-OLI Pescara Panel with a more than 20 years experience, made up of eight selected and trained tasters (6 women and 2 men, medium age 45 years) to recognise

virgin olive oil sensory attributes according to the E.C. regulation 796/02 on change of E.C. regulation 2568/91 [9].

2.5 Chemical analyses

On the olive samples, after the crushing by a micro-crusher, and on the pomace samples were determined:

- (i) water content, in the oven at 105°C;
- (ii) oil content, extracting the dried sample by the Soxhlet's apparatus, for 6 h, using petroleum ether 40–60°C.

On the vegetable water samples were determined:

- (i) the dry residue, in the oven at 105°C;
- (ii) the oil content, according to the method already published by Di Giovacchino [10].

On the oil samples (1° oil and 2° oil) were determined:

- (i) the values of the commercial qualitative characteristics according to the European Official Method of Analysis [9];
- (ii) the chlorophyll pigments content, Wolff [11];
- (iii) the composition of sterols and triterpene dialcohols according to the European Official Method of Analysis [9];
- (iv) the composition of aliphatic and triterpene according to the European Official Method of Analysis [9];
- (v) the composition of waxes according to the European Official Method of Analysis [12];
- (vi) olive oil volatile composition.

Volatile compounds were analysed by dynamic headspace GC using the followed procedure: 50 g oil samples were added of 10 mg of nonan-1-ol as internal standard (I.S.) and stripped with N₂ (1.2 dm³/min, 37°C for 2 h); the volatile compounds were adsorbed on 50 mg of activated charcoal and eluted with 1 mL of diethyl ether.

Quantitative analysis was carried out by GC technique using a Carlo Erba Mega Series 5160 gas chromatograph equipped with a Zebron ZB-WAX silica capillary column (60 m length; 0.32 mm id; 0.5 µm film thickness), an on-column injection system, a CO₂ cryogenic accessory to hold the oven at 28°C and an FID set at 230°C. The oven temperature program was run at 28°C for 6 min, varied at 1.0°C/min to 33°C (no hold), then at 1.8°C/min to 110°C (no hold), and at 2.5°C/min to 215°C at which it was held for 10 min. The carrier gas was H₂ at 40 kPa. The amounts of the different compounds are the mean values calculated from three independent experiments; the confidence intervals within the true response found with 95% of probability were always below the gas chromatographic error (10%). Concentrations were expressed as mg/kg of nonan-1-ol.

- Total and single phenols content.

The phenolic compounds were extracted according to Pirisi *et al.* [1]. The total phenol content in the olive oils was determined using the Folin-Ciocalteu reagent and measuring the absorbance at 750 nm. The total phenols were expressed as mg gallic acid/kg oil. The spectrophotometric analyses were repeated three times.

The determination of single phenols content, the standards used, the repeatability of the method, the procedures for identifying the peaks on each chromatogram, and the quantitative assessment of each compound of interest were carried out according to the method previously described by, Cortesi [13], Gómez-Alonso *et al.* [14], Mateos *et al.* [15] with some small changes.

A solution of I.S. (250 µL of a 15 mg/kg of syringic acid in methanol standard solution) was added to a sample of virgin olive oil (2.5 g) and the solvent was evaporated with a rotary evaporator at 35°C under vacuum. The oil was then dissolved in 6 mL of hexane and a diol-bonded phase cartridge (Supelco) was used to extract the phenolic fraction. The cartridge was conditioned with methanol (6 mL) and hexane (6 mL), the oil solution was then added, and the SPE column was washed with hexane (2 × 3 mL) and with hexane/ethyl acetate (80:20 v/v; 4 mL).

Finally, the phenols were eluted with methanol (10 mL) and the solvent was removed with a rotary evaporator at 35°C under vacuum to dryness. The phenolic residue was dissolved in methanol/water (1:1 v/v; 500 µL).

HPLC analysis was performed using a Perkin-Elmer LC 200 series system equipped with a column oven and a Perkin-Elmer Series 200 UV/VIS detector.

A LiChroCART Superspher C18 column (250 × 4.6 id mm, 4 µm particle size, Merck) was used, maintained at 25°C, with an injection volume of 20 µL and a flow rate of 1.0 mL/min. Mobile phase was a mixture of water/phosphoric acid (98:2 v/v) (solvent A), methanol (B) and acetonitrile (C): from 96% (A)–2% (B)–2% (C) to 50% (A)–25% (B)–25% (C) in 40 min, from 50% (A)–25% (B)–25% (C) to 40% (A)–30% (B)–30% (C) in 5 min, 40% (A)–30% (B)–30% (C) to 0% (A)–50% (B)–50% (C) in 15 min.

Quantification of phenols was carried out at 280 nm, and the results are expressed as mg/kg of tyrosol.

Phenolic compounds were quantified at 280 nm using syringic acid as I.S. and the response factors determined.

- Oil extraction yield.

In order to verify the oil extraction yield, the weights of each olive worked batch, of pomace and oil were determined. The vegetable water volume was calculated by the relations that describe the mass balance with regard to the oil quantity and the dry residue found in the olives, in the pomace and in the vegetable water.

2.6 Statistical analysis

During the tests, two samples of olives, pomace, vegetable water and oil were taken for each thesis. The analytical determinations were carried out in triplicate. The quantitative results were statistically evaluated by ANOVA procedure and the F-test.

3 Results and discussion

In the Table 1, the quantitative results, obtained in the experimental tests in the industrial oil mill, and the characteristics of olives, of pomace and vegetable water, obtained in the different adopted conditions, are reported. The data

suggest that the different methods of the olive paste preparation allowed to obtain satisfactory oil yields, comprised between 80.1 and 82.2%, in the case of the olives batch constituted from Ogliarola di Bitonto and Leccino cultivars, and between 83.6 and 85.2%, in the case of the olives lot composed by Peranzana and Leccino cultivars. The differences in oil yield, obtained from the two different olive batches, depend on the different rheological characteristics of the olive pastes that resulted more 'difficult to work' when Leccino cv was present at high level.

In fact, the yields of different olive batches were statistically significant ($p < 0.01$, F -test).

The different crushing methods adopted for olive paste preparation determined significant differences in oil yield ($p < 0.05$, F -test). The double crushing operation, in fact, allowed to obtain the highest oil yields because the preparation of the olive paste, by using in sequence both the metallic crusher at mobile knives and the stone mill, helped to break more deeply the vegetable cells of olive flesh containing oil. The use of the only one method of olive crushing, the metallic crusher at mobile knives or the stone mill, instead, allowed to obtain a lower oil yield because of the partial breakage of olive flesh.

In any case, the olive paste preparation, as generally it happens in the oil mills, was completed with the subsequent operation of malaxation that, during the technological tests, was carried out in rational conditions and for sufficient times, but not excessively, to assure, in the different adopted conditions, the formation of a satisfactory 'free' oil quantity and, therefore, a satisfactory extraction yield.

The same Table 1 shows that the oil content of the pomace was higher (4.4 and 4.0%) when the olive paste was prepared using only the stone mill that produces the formation of an olive paste not very homogenous, composed by rough fragments of fibrous part and by stones with sizes more variable than those obtained using the metallic crushers, equipped with a hole grid or with other regulation systems.

The double operation of crushing, by metallic crusher at knives and stone mill, that determined the highest extraction

yields, allowed to obtain a pomace with the a lower oil content, resulted, respectively, 3.7 and 3.6%. In consideration of the pomace quantity (kg/100 kg of olives), obtained by the different methods of olive paste preparation, the remaining oil in the pomace was variable between 2.4 and 2.8 kg/100 kg of olives, in the case of olive lot with a 70% of Leccino cultivar, and between 2.2 and 2.4 kg/100 kg of olives, in the other case.

Moreover, the data reported in Table 1 shows that a small volume of vegetable water is generally produced. In fact, the volume of the liquid by-product was variable between 55 and 58 L/100 kg of olives, in the case of the first olive lot, considered 'more difficult to work', and between 40 and 42 L/100 kg of olives, in the second case. These quantities can be considered not high and corresponding to those generally produced by the new generation decanters, working at 3-phases with a low quantity of added water. In the Table 2 are reported the results of the analytical determinations carried out on the oil samples (1° oil) obtained in the above mentioned conditions. The data show that all the oil samples were extra virgin and that the different methods used for the olive paste preparation (by the metallic crusher and/or by the stone mill) did not significantly change the qualitative characteristics of oil.

This result confirms that the technological operations, carried out in the oil mill in a rational way and according to the hygienic and sanitary norms, cannot change the oil quality that only depends (less than fortuitous pollution) on the quality of olives, as described in other papers on the influence of the crushing method [6, 7] or other technological operations on the oil quality [16, 17].

The different composition of two olive batches, with the different cvs present, allowed to obtain a significant difference in the total phenols content of virgin olive oils ($p < 0.05$, F -test). At the contrary, the different crushing methods used did not determine a significant difference in the total phenols content of virgin olive oils.

However, the total phenol content of virgin olive oils was always significantly lower in oil samples obtained from olive pastes prepared using only the stone mill, as, besides, it was

Table 1. Oil yield and pomace and vegetable water characteristics obtained in the oil extraction, using centrifugal decanters at water saving, obtained from olive pastes prepared by different crushing methods

Olives					Pomace			Vegetable water			
	Cultivar	Water (%)	Oil (%)	Crushig method	Yield (%)	Quantity (kg)	Water (%)	Oil (%)	Quantity (L)	D.m. (%)	Oil (g/L)
Ogliarola di Bitonto + Leccino		52.6	17.5	Crusher + stones	82.2 ^a	64.1	61.1	3.7	55.0	12.8	13.3
				Crusher	80.4 ^b	62.1	57.9	4.1	58.0	11.6	14.6
				Stone mill	80.1 ^b	62.8	57.0	4.4	56.0	10.4	13.0
Peranzana + Leccino		50.5	20.9	Crusher + stones	85.2 ^c	62.5	60.2	3.6	42.0	15.2	16.7
				Crusher	84.2 ^{c,d}	59.3	58.8	3.8	40.0	18.0	18.3
				Stone mill	83.6 ^d	59.9	57.7	4.0	41.0	14.9	17.0

The pomace quantity (kg) and vegetable water (L) are referred to 100 kg of olives. Different letters indicated significant differences at $p < 0.05$.

Table 2. Qualitative characteristics of 1° oils obtained by the centrifugal decanter at oil and water saving from olive pastes prepared by different crushing and/or malaxation methods^{a)}

Determinations	Ogliarola di Bitonto + Leccino			Peranzana + Leccino		
	Crusher + stones	Crusher	Stone mill	Crusher + Stones	Crusher	Stone mill
Free fatty acids (%)	0.14 ± 0.01	0.17 ± 0.01	0.17 ± 0.01	0.17 ± 0.01	0.16 ± 0.01	0.16 ± 0.01
Peroxide value (meq/kg)	5.9 ± 0.3	6.0 ± 0.4	6.2 ± 0.4	5.3 ± 0.3	4.9 ± 0.4	5.7 ± 0.4
K ₂₃₂	1.56 ± 0.01	1.61 ± 0.01	1.59 ± 0.01	1.47 ± 0.01	1.48 ± 0.01	1.48 ± 0.01
K ₂₇₀	0.11 ± 0.01	0.11 ± 0.01	0.12 ± 0.01	0.12 ± 0.01	0.12 ± 0.01	0.12 ± 0.01
Organoleptic assessment (score) ^{b)}	6.8	6.8	6.8	7.0	7.1	6.9
Total phenols (mg/L) ^{a)}	92 ± 8.9 ^a	86 ± 9.4 ^a	72 ± 7.2 ^b	224 ± 12.5 ^c	212 ± 14.1 ^c	160 ± 9.4 ^d
Chlorophyll pigments (mg/kg)	10.6 ± 0.4	9.2 ± 1.1	8.3 ± 0.3	6.4 ± 0.2	6.7 ± 0.2	6.8 ± 0.3

^{a)} Median value ± SD of three replicates.

^{b)} Median value obtained as described in the Annex XII of the regulation (EEC) 2568/91.

Different letters indicated significant differences at $p < 0.05$.

reported in other papers [6, 7]. It depends on the more or less deep olive crushing methods and, as consequence, on the different sizes of the flesh and stone fragments. By the stone mill, as crusher, allows to obtain a rougher and not homogeneous olive paste, with larger size fragments, with 2 mm mean size diameter (low specific surface) depended on the rough surface of bowl and rotating wheels. Differently, the solid fragments of olive paste prepared using the violent metallic crusher are more homogeneous, 1.5 mm mean size diameter [18], also in relation to the size of the holes of the grid that in this study ranged from 6 to 7 mm.

The smaller sizes of the solid fragments allow the release of the various substances present on the cellular and intracellular tissues of the olive flesh and cause the increase of the specific surface also, favouring the substance exchange process between the oily phase and the aqueous phase of the olive paste. The

substance exchange results more effective when the surface of the solid parts, where the contact between the two not mixed liquids occurs, increases and also favours, in this way, a very quick attainment of the equilibrium of the concentrations of the phenolic substances in the aqueous and in the oily phase.

This is confirmed by the relative data of the single phenols found in the olive oil samples, as reported in the Table 3. The data show that the content of oleuropein and ligstroside aglycons, in their different structural forms, is slightly higher in the oils obtained from olive pastes prepared by the double crushing, using both the metallic crusher at knives and the stone mill. These differences, however, did not influence significantly the bitter and pungent intensity of the same oils that are resulted, in the organoleptic assessment, very similar just for the reason that the different applied methods, for the olive crushing, work with a similar and not excessive violence.

Table 3. Content of the principal phenolic substances of 1° oils (mg/kg) obtained by the centrifugal decanter at oil and water saving from olive pastes prepared by different methods^{a)}

Determinations	Ogliarola di Bitonto + Leccino			Peranzana + Leccino		
	Crusher + Stones	Crusher	Stone mill	Crusher + Stones	Crusher	Stone mill
Hydroxytyrosol	1.1 ± 0.2	1.8 ± 0.4	1.1 ± 0.1	2.8 ± 1.3	3.4 ± 0.1	4.4 ± 1.6
Tyrosol	11.6 ± 1.7	12.7 ± 1.6	10.0 ± 1.0	7.9 ± 0.8	6.7 ± 0.5	9.9 ± 0.9
3,4-DHPEA-EDA ^{b)}	18.0 ± 2.0	21.0 ± 2.1	15.8 ± 1.9	85.8 ± 3.6	70.5 ± 3.3	88.5 ± 3.9
3,4-DHPEA-EA ^{c)}	2.1 ± 0.5	3.6 ± 0.7	6.3 ± 0.5	26.7 ± 2.6	18.4 ± 1.6	20.1 ± 1.8
<i>p</i> -HPEA-EDA ^{d)}	108.1 ± 21.4	107.5 ± 20.5	80.2 ± 14.2	140.5 ± 27.4	114.4 ± 21.2	130.5 ± 24.5
Lignans	37.4 ± 3.4	33.3 ± 3.5	34.5 ± 4.1	75.9 ± 14.1	66.7 ± 12.8	85.8 ± 14.9
3,4-DHPEA,-EA,H ^{e)}	17.5 ± 2.0	24.5 ± 3.2	15.7 ± 2.1	39.2 ± 4.5	33.2 ± 4.1	39.5 ± 4.9
<i>p</i> -HPEA,-EA,H ^{f)}	28.5 ± 3.1	24.9 ± 3.4	36.2 ± 3.6	38.4 ± 4.1	32.5 ± 3.9	40.5 ± 4.4

^{a)} Mean value ± SD of three replicates.

^{b)} 3,4-Dihydroxyphenyl-ethanol linked to dialdehydic form of elenolic acid.

^{c)} 3,4-dihydroxyphenyl-ethanol linked to elenolic acid.

^{d)} *p*-Hydroxyphenyl-ethanol linked to dialdehydic form of elenolic acid.

^{e)} 3,4-Dihydroxyphenyl-ethanol linked to aldehydic and hydroxylic form of elenolic acid.

^{f)} *p*-Hydroxyphenyl-ethanol linked to aldehydic and hydroxylic form of elenolic acid.

Respect to total phenols content, significant differences were found by statistical analysis only for different olive batches ($p < 0.05$, F -test).

A further confirmation of the slight violent mechanical action, and with similar intensity, exerted by the different machines used for the olive crushing, is that the intensity of green colour of oils, calculated using the determination of the chlorophyll pigments, did not result significantly different for the oils obtained in the unlike conditions used for the olive paste preparation.

Table 4 shows the data obtained in the analysis of the volatile compounds of the head-space of oils. As can be observed the principal compound, as it happens with good quality oils, is the unsaturated aldehyde *trans*-2-hexenal and its quantity did not change considerably using different crushing techniques but only for the olive cultivars. Also the content of the other volatile compounds has endured a not significant variation in relation to the different crushing methods and this is a confirmation of as much as already said about the similar mechanical actions that the same methods exert on the olives.

However, the different techniques used appear to have an influence on the pentene dimers content and on the C₅ oxygenated compounds derived from linolenic acid. The content of these compounds was always lower when oils were

obtained from olive pastes prepared using the stone mill only, probably for the soft mechanical action applied from the granite stone that, besides, does not produce heat variations into the obtained mixture.

In conclusion, the chemical and organoleptic characteristics of the oils (1° oils) obtained from olive pastes prepared using different described crushing methods, resulted very similar for the values of the commercial qualitative parameters and also for the content and composition of the phenolic and volatile compounds. The lower content of these substances in oils obtained from olive pastes prepared by the stone mill resulted not significantly different and it is due to the soft mechanical action that the mentioned stone mill exerts on the olives.

The characteristics of the 2° oils, called 'recovered oil' also and sampled at the exit of the same decanter, are reported in the Table 5. The data show that these oils are extra virgin, but their commercial quality is lower than that of the 1° oils because of the higher values of free fatty acids, peroxide index and the specific spectrophotometric absorption in the UV region.

Table 5, moreover, shows that the 2° oils present a higher content of chlorophyll pigments and total phenols. It depends on the fact that the 2° oil comes from the partially extracted olive paste in the final conical part of the rotating drum of the decanter, shortly before that the pomace is ejected from the

Table 4. Concentrations of volatile compounds (mg/kg of 1-nonanol) of 1° oils extracted by a centrifugal decanter at oil and water saving from olive pastes obtained with different crushing and/or malaxation methods^{a)}

Determinations	Ogliarola di Bitonto + Leccino			Peranzana + Leccino		
	Crusher + stones	Crusher	Stone mill	Crusher + stones	Crusher	Stone mill
Ethyl acetate	5.7 ± 0.2	4.0 ± 0.1	5.6 ± 0.1	8.5 ± 0.3	10.2 ± 0.2	9.3 ± 0.2
Ethyl alcohol	14.5 ± 0.5	14.8 ± 0.6	16.1 ± 0.5	13.9 ± 0.4	18.7 ± 0.3	14.7 ± 0.5
2 + 3-Pentan-one	13.8 ± 0.5	9.6 ± 0.1	12.3 ± 0.2	21.9 ± 0.7	21.0 ± 0.6	15.3 ± 0.8
1-Penten-3-one	9.6 ± 0.6	8.7 ± 0.7	6.1 ± 0.4	16.4 ± 0.6	15.8 ± 0.4	7.8 ± 0.3
Pentene dimers	32.1 ± 3.4	26.5 ± 3.1	17.2 ± 2.3	32.7 ± 4.8	34.2 ± 4.6	14.7 ± 0.6
Hexanal	24.6 ± 3.0	17.5 ± 2.4	24.8 ± 3.4	46.8 ± 6.4	41.5 ± 5.4	41.1 ± 4.9
Isobutyl alcohol	3.8 ± 0.2	3.1 ± 0.1	3.8 ± 0.2	9.0 ± 1.0	9.8 ± 1.4	7.7 ± 0.7
<i>trans</i> -2-Pentenal	3.7 ± 0.1	0.6 ± 0.1	2.6 ± 0.2	5.1 ± 0.1	5.1 ± 0.6	3.3 ± 0.1
1-Penten-3-ol	22.9 ± 2.6	18.0 ± 2.2	16.4 ± 2.4	36.8 ± 4.2	36.4 ± 4.1	22.4 ± 3.2
<i>cis</i> -3-Hexenal	5.8 ± 0.4	5.2 ± 0.4	5.7 ± 0.3	7.7 ± 0.6	7.7 ± 0.2	6.6 ± 0.6
Isoamyl alcohol	19.6 ± 2.6	16.8 ± 2.4	20.6 ± 2.8	22.0 ± 2.1	24.7 ± 2.6	22.4 ± 2.8
<i>trans</i> -2-Hexenal	677.0 ± 56.3	608.0 ± 48.6	676.0 ± 58.3	923.0 ± 71.5	924.0 ± 80.2	798.0 ± 61.6
Hexyl acetate	5.0 ± 0.3	4.2 ± 0.4	4.7 ± 0.6	4.7 ± 1.1	4.6 ± 1.0	4.3 ± 1.0
<i>cis</i> -3-Hexenyl acetate	2.0 ± 0.1	1.8 ± 0.2	1.4 ± 0.2	2.5 ± 0.3	2.5 ± 0.3	1.5 ± 0.2
<i>cis</i> -2-Penten-1-ol	11.9 ± 0.4	10.4 ± 0.2	9.4 ± 0.5	15.5 ± 0.7	16.8 ± 0.6	10.8 ± 0.6
1-Hexanol	33.5 ± 2.9	23.9 ± 2.7	31.6 ± 3.4	42.5 ± 4.6	47.1 ± 5.9	40.7 ± 4.9
<i>cis</i> -3-Hexen-1-ol	15.3 ± 0.8	14.0 ± 0.4	15.8 ± 0.6	6.0 ± 0.5	5.9 ± 0.5	7.0 ± 0.8
<i>trans</i> -2-Hexen-1-ol	43.0 ± 5.6	29.1 ± 4.4	45.6 ± 6.1	45.9 ± 7.2	58.2 ± 8.1	56.3 ± 7.7
Total C ₆ -Lna acid	741.0 ± 60.8	656.0 ± 48.7	743.0 ± 32.8	983.0 ± 65.4	996.0 ± 60.9	868.0 ± 53.6
Total C ₅ -Lna acid	80.2 ± 6.2	64.2 ± 7.6	51.7 ± 4.9	106.5 ± 10.1	108.4 ± 9.2	58.9 ± 7.2
Total C ₆ -La acid	58.1 ± 5.3	41.4 ± 4.6	56.4 ± 3.9	89.3 ± 4.8	88.6 ± 6.6	81.8 ± 5.9
Total LOX esters	7.0 ± 0.5	6.0 ± 0.4	6.1 ± 0.5	7.2 ± 0.5	7.1 ± 0.2	5.8 ± 0.2

^{a)} Mean value ± SD of three replicates.

Table 5. Qualitative characteristics of the 2° oils ('recovered oils') extracted by a centrifugal decanter at oil and water saving from olive pastes obtained with different crushing methods^{a)}

Determinations	Ogliarola di Bitonto + Leccino			Peranzana + Leccino		
	Crusher + stones	Crusher	Stone mill	Crusher + stones	Crusher	Stone mill
Free fatty acids (%)	0.27 ± 0.01	0.25 ± 0.01	0.24 ± 0.01	–	0.25 ± 0.01	0.24 ± 0.01
Peroxide value (meq/kg)	13.2 ± 0.3	12.5 ± 0.4	13.0 ± 0.4	–	6.8 ± 0.4	5.6 ± 0.4
K ₂₃₂	1.81 ± 0.01	1.83 ± 0.01	1.82 ± 0.01	–	1.62 ± 0.01	1.70 ± 0.01
K ₂₇₀	0.19 ± 0.01	0.20 ± 0.01	0.18 ± 0.01	–	0.15 ± 0.01	0.17 ± 0.01
Organoleptic assessment (score) ^{b)}	6.5	6.6	6.5	–	6.7	6.7
Total phenols (mg/L)	244 ± 10.9	247 ± 11.4	220 ± 10.6	–	282 ± 14.1	256 ± 12.6
Chlorophyll pigments (mg/kg)	35.9 ± 6.4	39.1 ± 7.1	37.0 ± 6.8	–	32.8 ± 6.2	30.9 ± 5.2

^{a)} Mean value ± SD of three replicates. * The 2° oil of this thesis was not sampled.

^{b)} Median value obtained as described in the Annex XII of the regulation (EEC) 2568/91.

Table 6. Concentration of volatile compounds (mg/kg of 1-nonanol) of the head-space of the 2° oils obtained by a centrifugal decanter at oil and water saving from olive pastes obtained with different crushing methods^{a)}

Determinations	Ogliarola di Bitonto + Leccino			Peranzana + Leccino		
	Crusher + stones	Crusher	Stone mill	Crusher + stones	Crusher	Stone mill
Ethyl acetate	10.4 ± 0.4	9.4 ± 0.2	12.6 ± 0.4	–	12.8 ± 0.4	11.8 ± 0.3
Ethyl alcohol	19.0 ± 0.7	23.5 ± 1.0	27.1 ± 1.3	–	35.5 ± 2.2	29.9 ± 2.4
2 + 3-Pentan-one	12.3 ± 0.5	15.0 ± 0.7	20.6 ± 1.1	–	14.6 ± 0.6	13.9 ± 0.8
1-Penten-3-one	18.0 ± 0.6	21.0 ± 0.7	15.8 ± 0.4	–	70.5 ± 3.4	88.5 ± 4.2
Pentene dimers	3.8 ± 0.2	4.6 ± 0.6	3.8 ± 0.4	–	6.3 ± 0.6	5.7 ± 0.6
Hexanal	20.5 ± 3.0	18.2 ± 2.4	17.0 ± 2.4	–	21.0 ± 3.4	20.4 ± 2.9
Isobutyl alcohol	3.1 ± 0.7	2.8 ± 0.9	3.1 ± 0.2	–	6.4 ± 0.5	5.0 ± 0.3
<i>trans</i> -2-Pentenal	1.6 ± 0.1	2.2 ± 0.2	2.2 ± 0.2	–	1.6 ± 0.2	2.1 ± 0.3
1-Penten-3-ol	1.6 ± 0.3	2.2 ± 0.2	2.2 ± 0.2	–	1.6 ± 0.1	2.1 ± 0.2
<i>cis</i> -3-Hexenal	2.5 ± 0.4	3.0 ± 0.2	3.1 ± 0.3	–	2.7 ± 0.2	3.4 ± 0.6
Isoamyl alcohol	10.9 ± 0.6	10.0 ± 0.4	11.4 ± 0.6	–	13.2 ± 1.3	12.7 ± 0.9
<i>trans</i> -2-Hexenal	373.0 ± 18.2	357.0 ± 16.9	372.0 ± 17.9	–	445.0 ± 24.2	474.0 ± 26.3
Hexyl acetate	4.2 ± 0.3	4.4 ± 0.4	4.9 ± 0.6	–	6.5 ± 1.1	6.2 ± 1.0
<i>cis</i> -3-Hexenyl acetate	1.6 ± 0.1	1.6 ± 0.2	1.7 ± 0.1	–	1.7 ± 0.2	1.2 ± 0.1
<i>cis</i> -2-Penten-1-ol	9.4 ± 0.6	8.4 ± 0.4	8.9 ± 0.3	–	13.0 ± 0.7	10.8 ± 0.6
1-Hexanol	35.3 ± 3.1	27.7 ± 2.4	33.3 ± 3.0	–	34.5 ± 4.6	42.8 ± 3.9
<i>cis</i> -3-Hexen-1-ol	13.1 ± 0.9	12.5 ± 0.5	13.6 ± 0.4	–	5.6 ± 0.4	5.7 ± 0.6
<i>trans</i> -2-Hexen-1-ol	70.9 ± 7.6	53.1 ± 4.8	59.9 ± 6.3	–	79.7 ± 7.2	65.5 ± 7.4
Total C ₆ -Lna acid	477.0 ± 50.6	426.0 ± 32.8	449.0 ± 37.9	–	533.0 ± 51.3	549.0 ± 48.9
Total C ₅ -Lna acid	44.7 ± 7.2	44.1 ± 6.4	44.4 ± 5.4	–	61.0 ± 9.4	52.5 ± 5.2
Total C ₆ -La acid	55.5 ± 5.3	45.9 ± 4.5	50.3 ± 3.7	–	56.5 ± 4.6	63.2 ± 6.3
Total LOX esters	5.8 ± 0.6	6.0 ± 0.4	6.6 ± 0.6	–	8.2 ± 0.6	7.4 ± 0.2

^{a)} Mean value ± SD of three replicates.

decanter. In this area, the olive paste is not more in contact with the added water and, consequently, the residual oil, included between cellular and intracellular tissues of the olive flesh and in contact with the only constitutive vegetable water of the same olive, can be released by the centrifugal force and keeps the same total phenols content reached after the malaxation operation and corresponding to that established from

the partition equilibrium conditions between aqueous phase (olive vegetable water) and oily phase of the mixture. Moreover, the temperature of this part of decanter is higher than that of the exit of 1° oil and this fact favours the increase of total phenol content of 2° oil.

With regard to the aromatic substances of oils, instead, the 2° oils showed, in comparison with the 1° oils, a lower

Table 7. Content of some aliphatic alcohols, single and total (mg/kg oil), waxes, erythrodiol + uvaol (%) and some sterols of two types of oil (1° oil and 2° oil) obtained by a centrifugal decanter at oil and water saving from olive pastes prepared with different crushing methods^{a)}

Determinations	Type of oil	Ogliarola di Bitonto + Leccino			Peranzana + Leccino		
		Crusher + stones	Crusher	Stone mill	Crusher + stones	Crusher	Stone mill
C ₂₂	1°	22.8 ± 2.6	20.5 ± 2.2	16.6 ± 1.8	10.1 ± 1.1	9.5 ± 0.8	10.5 ± 1.4
	2°	22.8 ± 2.4	23.5 ± 2.1	24.4 ± 2.5	–	17.2 ± 1.6	15.9 ± 1.8
C ₂₄	1°	49.0 ± 5.6	45.8 ± 4.8	34.2 ± 2.6	22.2 ± 2.4	20.5 ± 2.6	21.1 ± 2.0
	2°	48.6 ± 5.4	51.9 ± 5.3	52.7 ± 4.6	–	36.4 ± 2.8	32.2 ± 2.9
C ₂₆	1°	84.3 ± 7.8	84.9 ± 8.6	56.4 ± 4.9	35.7 ± 3.6	33.5 ± 4.1	35.4 ± 3.6
	2°	83.1 ± 6.6	84.9 ± 7.7	79.4 ± 8.1	–	59.1 ± 5.6	49.7 ± 4.8
C ₂₈	1°	56.1 ± 5.1	60.3 ± 7.6	38.3 ± 2.6	20.8 ± 2.9	21.7 ± 3.4	20.3 ± 2.6
	2°	49.7 ± 4.2	59.8 ± 5.7	51.9 ± 4.8	–	36.5 ± 5.1	34.1 ± 2.2
Total aliphatic alcohols	1°	212.1 ± 20.4	211.5 ± 17.9	145.5 ± 15.7	88.9 ± 7.8	85.2 ± 9.1	87.3 ± 7.9
	2°	204.0 ± 19.7	220.0 ± 19.6	208.0 ± 18.9	–	149.0 ± 15.4	132.0 ± 14.1
Total waxes (mg/kg)	1°	121.0 ± 13.6 ^a	124.0 ± 13.6 ^a	103.0 ± 10.6 ^a	86.0 ± 9.4	77.0 ± 7.6	92.3 ± 8.9
	2°	164.0 ± 16.1 ^b	175.5 ± 18.6 ^b	188.0 ± 20.2 ^b	–	139.4 ± 13.4	143.3 ± 14.7
Erythrodiol + Uvaol (%)	1°	2.2 ± 0.1 ^a	1.6 ± 0.1 ^a	1.7 ± 0.3 ^a	1.6 ± 0.1	2.1 ± 0.1	2.3 ± 0.2
	2°	2.7 ± 0.2 ^b	2.5 ± 0.3 ^b	2.8 ± 0.2 ^b	–	3.6 ± 0.2	3.9 ± 0.2
Campesterol (%)	1°	3.3 ± 0.1	3.1 ± 0.2	3.3 ± 0.1	3.1 ± 0.1	3.5 ± 0.2	3.6 ± 0.1
	2°	3.3 ± 0.1	3.4 ± 0.1	3.2 ± 0.1	–	3.6 ± 0.2	3.5 ± 0.1
Stigmasterol (%)	1°	0.7 ± 0.1	0.6 ± 0.1	0.7 ± 0.1	0.8 ± 0.1	0.8 ± 0.1	0.8 ± 0.1
	2°	0.8 ± 0.1	0.9 ± 0.1	0.8 ± 0.1	–	0.9 ± 0.2	0.8 ± 0.1
Apparent β sitosterol (%)	1°	94.9 ± 0.3	95.1 ± 0.3	94.8 ± 0.3	95.1 ± 0.3	94.5 ± 0.3	94.5 ± 0.3
	2°	94.9 ± 0.4	94.7 ± 0.3	95.2 ± 0.2	–	94.6 ± 0.3	94.9 ± 0.3

^{a)} Mean value ± SD of three replicates.

Different letters indicated significant differences at $p < 0.05$.

Table 8. Average content of some triterpene alcohols (mg/kg of oil) of 1° and 2° oils obtained by a centrifugal decanter at oil and water saving from olive pastes prepared with different crushing methods^{a)}

Determinations	Type of oil	Ogliarola di Bitonto + Leccino			Peranzana + Leccino		
		Crusher + stones	Crusher	Stone mill	Crusher + stones	Crusher	Stone mill
α-Amirin	1°	1.7 ± 0.1	1.7 ± 0.1	6.5 ± 0.1	1.2 ± 0.1	4.3 ± 0.1	16.8 ± 0.1
	2°	22.3 ± 0.3	33.0 ± 0.2	15.7 ± 0.1	–	19.6 ± 0.2	17.9 ± 0.1
β-Amirin	1°	48.8 ± 4.8	53.0 ± 5.1	44.6 ± 3.8	45.1 ± 4.2	57.9 ± 6.1	51.2 ± 5.8
	2°	80.3 ± 5.1	93.1 ± 6.8	81.5 ± 5.4	–	88.9 ± 6.4	79.8 ± 6.1
Butyrospermol	1°	85.2 ± 6.2	100.8 ± 7.2	71.9 ± 4.3	57.1 ± 4.8	54.3 ± 3.6	52.4 ± 3.3
	2°	135.1 ± 9.8	157.3 ± 8.8	128.0 ± 7.2	–	130.2 ± 7.3	112.7 ± 11.1
Cicloarthenol	1°	283.0 ± 20.3	359.0 ± 17.6	273.0 ± 16.1	473.0 ± 26.4	433.0 ± 21.8	486.0 ± 24.6
	2°	370.0 ± 19.7	427.0 ± 23.6	361.0 ± 17.9	–	567.0 ± 32.6	511.0 ± 30.4
24-Methylene-cicloarthenol	1°	297.0 ± 18.8	374.0 ± 21.4	301.0 ± 20.1	283.0 ± 17.9	259.0 ± 18.9	312.0 ± 22.5
	2°	327.0 ± 21.4	366.0 ± 18.7	310.0 ± 22.3	–	273.0 ± 21.4	243.0 ± 23.6
Total triterpene alcohols	1°	716.0 ± 38.7	888 ± 44.8	697.0 ± 34.9	859.0 ± 44.7	808.0 ± 48.6	918 ± 43.5
	2°	935 ± 44.6	1076 ± 52.9	896.0 ± 46.5	–	1079 ± 53.2	964 ± 46.1

^{a)} Mean value ± SD of three replicates.

content of volatile compounds, as underlined in Table 6. In particular, *trans*-2-hexenal content resulted about 40% lower. It depends on the loss of volatile substances that happened because of the longer stay of the 2° oils inside the decanter

and, probably, for the slight increase of the temperature of oil in this part of the decanter.

The two oils types, however, showed identical composition of the sterol fraction, as underlined in Table 7. The data

confirmed that the 2° oils, compared with the 1° oils, had a higher content of the aliphatic alcohols, of waxes and of the erythrodiol and uvaol percentage also. Significant differences were found by statistical analysis for waxes, uvaol and erythrodiol ($p < 0.05$).

The ANOVA test, for the total triterpene alcohols content (Table 8), showed that the content of these substances is significantly different ($p < 0.01$) for 1° and 2° oils and for their extraction methods.

4 Conclusions

The technological tests carried out in industrial olive oil mill, working by a three-phases centrifugal decanter at oil and water saving, to verify the influence of the different olives crushing methods, put in evidence that:

- (i) the olive paste preparation, by the use of a metallic crusher at knives and/or a granite stone mill, did not determine significant differences on oil yield that, in the carried out tests, was satisfactory and depending mainly on the olive characteristics;
- (ii) the qualitative characteristics of 1° oils resulted very similar and not influenced by the different methods used for the olive paste preparation. It depends on the fact that both the mobile-knives metallic crusher and granite stone mill exert a not violent action on the olives the whose breaking is not very deep and produces similar conditions that, therefore, cannot determine significant differences on the commercial qualitative parameters of oil that depend on the characteristics of the olive basically;
- (iii) as for the organoleptic characteristics, all samples resulted with few differences in the intensity of the bitter and pungent sensations. This fact confirms that the metallic crusher used (at knives) is suitable to replace, in all or part, the granite stone mill when a less pungent and bitter oil is required;
- (iv) the oils obtained from olive paste prepared only by the mill stone showed to have a content of phenolic substances, either total or single, slightly lower respect to oils obtained from olive paste prepared using other methods, according to the results obtained in other papers published on this subject;
- (v) the volatile substances content of the oils was not influenced by the different crushing methods used but only by the different olive cultivars. Just the content of pentene dimers and C₅ oxygenated compounds come from linolenic acid resulted lower in the oils obtained from olive paste prepared using only the mill stone;
- (vi) the 2° oils showed different qualitative characteristics, with respect to those of the 1° oils.

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