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Influence of a new crushing technique on the composition of the volatile compounds and related sensory quality of virgin olive oil

The influence of a new crusher *i.e.* blade crusher on the quality of virgin olive oil from two different italian cultivars (*Coratina* and *Oliarola*) was determined. In addition the quality of this oil was compared with that of olive oil extracted with the traditional hammer crusher.

Tests were performed in an industrial oil mill using the two different crushing instruments.

Results obtained showed that quality parameters *i.e.* free fatty acids, peroxide value, UV absorption and total phenols content as well as the phenolic composition of oils were not significantly affected by the two different crushers used. On the contrary, the use of the blade crusher influenced the composition of the volatile compounds. In particular, the oils obtained using the blade crusher showed significant increases of some aldehydes such as 1-hexanal and *trans*-2-hexenal, esters such as hexyl acetate and 3-hexenyl acetate and a reduction of alcohols such as 1-hexanol. Moreover, the identified pigments of the oils produced using the blade crusher were found at concentrations slightly lower than those in oils obtained after using the hammer crusher. Finally, results of the sensory analysis showed an improved organoleptic quality for the oils obtained using the blade crusher due to an increase of the cut-grass and floral sensory notes.

Keywords: Olives crusher, olive oil quality, phenols, volatile compounds, pigments.

1 Introduction

Virgin olive oil is extracted from olive fruit using only mechanical processes including olives crushing, malaxation of resulting pastes and separation of the oily phase essentially by pressure or centrifugation. All these operations affect the quality of virgin olive oil [1]. The influence of malaxation as well as the effect of the separation process on the quality of the resulting oils have been widely studied [2–5]. However, little knowledge exists about the influence of crushing on the quality of virgin olive oil [6], in particular on phenolic and volatile compounds which are the most important markers of the sensory and healthy qualities of virgin olive oil [7–8].

Olives crushing is currently performed in industrial oil mills with the traditional discontinuous stone-mill or with the continuous hammer crusher. The latter is mainly used in the olive oil industry where the oil extraction is usually performed by centrifugation [9–11].

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Recently, a new olives crusher [12] with blades instead of hammers has been introduced in order to improve oil quality.

In this paper we are the first to refer to the influence of the blade crusher on the olive oil quality, which was compared with the quality of the olive oil obtained using the traditional hammer crusher.

2 Materials and methods

2.1 Materials

2.1.1 Olives

Drupes from *Coratina* and *Oliarola* cultivars were used for this investigation. Olives of both cultivars were picked the same day of December 2000 in the *Apulia* region (Italy) at a known ripening degree and in perfect sanitary conditions. Olives from each cultivar were divided into two homogeneous 800-kg lots and then used the same day for oil extraction, whereas one lot was crushed using a hammer crusher while the other lot was crushed with a blade crusher. All steps were performed in triplicate. In all experimental trials the oil extraction process including the malaxation step was carried out at a temperature below 28 °C.

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2.1.2 Oils

Olive oil extraction was carried out in a cooperative olive oil mill (Molfetta, Italy) equipped with both crushers, *i.e.* hammer crusher and blade crusher (Tab. 1) with different crushing members (Fig. 1). For each cultivar, the processing procedures (Fig. 2) were identical, except for the crushing step that was carried out using the hammer or the blade crusher.

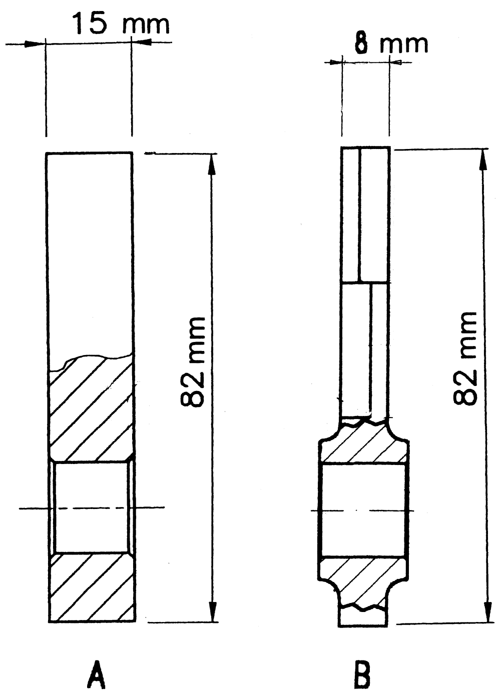

Fig. 1. Schematic drawing of the hammer (A) and blade (B) crushing members.

Tab. 1. Characteristics of the hammer and blade crushers.

	Hammer crusher	Blade crusher
Grid:		
height	142 mm	142 mm
inner diameter	330 mm	330 mm
holes number	4484	4484
holes diameter	7 mm	7 mm
Crushing member:		
number	10	10
height	82 mm	82 mm
thickness	**15 mm**	**8 mm**
angular velocity	2900 rpm	2900 rpm
working capacity	3000 kg/h	3000 kg/h

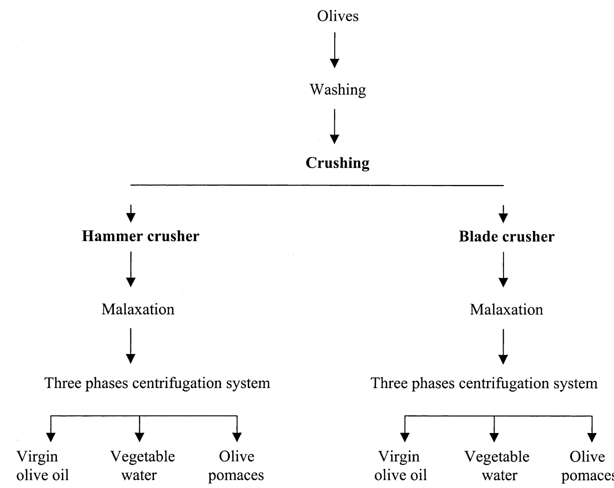

Fig. 2. Flux diagram of the processing procedures using hammer and blade crushers.

2.1.3 Reference compounds

p-Hydroxyphenylethanol (*p*-HPEA) was obtained from *Janssen Chemical* Co. (Beerse, Belgium) and caffeic acid and vanillic acid were obtained from *Fluka* Co. (Buchs, Switzerland). The 3,4-dihydroxyphenylethanol (3,4-DHPEA) was synthesized in the laboratory according to the procedure of *Baraldi* et al. [13]. The dialdehyde form of elenolic acid linked to 3,4-DHPEA (3,4-DHPEA-EDA), the isomer of oleuropein aglycon (3,4-DHPEA-EA) and the dialdehyde form of elenolic acid linked to *p*-HPEA (*p*-HPEA-EDA) were extracted from virgin olive oil using the procedure reported in a previous paper [14]. The purity of these substances was tested by high-performance liquid chromatography (HPLC) while the chemical structures were verified by nuclear magnetic resonance (NMR) spectroscopy.

The volatile compounds were purchased from *Fluka* Co. and *Aldrich* (Steinheim, Germany).

β-carotene, lutein, chlorophyll *a* and chlorophyll *b*, pheophytin *a* and pheophytin *b* were obtained from *Fluka* Co.

2.2 Methods

Oil samples obtained were analyzed to determine free fatty acids, the peroxide value and ultraviolet specific absorptions according to the Italian *Official Methods for Fats and Oils* [15] while total phenols content was determined using the *Folin-Ciocalteau* reagent according to the method previously reported [14].

In addition to these quality parameters the phenolic and volatile compounds, the pigments and the sensory characterization of oils were considered.

2.2.1 Phenolic compounds

The HPLC evaluation of phenolic compounds was carried out according to the method reported in a previous paper [14] but using an *Inertsil ODS-3* column (150 × 4.6 mm i.d.) from *Alltech Italia* S.r.l. (Sedriano, Milan).

2.2.2 Volatile compounds

The volatile compounds were sampled by solid-phase microextraction (SPME) as follows: virgin olive oil (3 g) was put in a 20 ml vial and thermostated at 35 °C for 15 min. Subsequently the fiber (65 μm Carbowax/divinylbenzene purchased from *Supelco,* Inc., Bellefonte, PA, USA) was exposed to the vapour phase for 30 min for sampling volatile compounds. The fiber was inserted in the gas chromatography (GC) injector set at 250 °C in splitless mode using a splitless inlet liner of 0.75 mm ID for 10 min. All the SPME operations were automated using a Varian 8200 CX AutoSampler (*Varian*, Walnut Creek, CA, USA) [16].

2.2.3 GC-MS

A GC Varian 3600 equipped with a split/splitless injector coupled with a mass spectrometer Varian Saturn 3 (*Varian,* Walnut Creek, CA, USA) was used. A fused-silica capillary column DB-Wax, 50 m, 0.32 mm ID, 1 μm film thickness (*J & W Scientific,* Folsom, CA, USA) was employed. The column was operated with helium at a pressure of 103 kPa with a flow rate of 2.2 ml/min and a linear velocity of 30.7 cm/sec at 35 °C. The GC oven heating was started at 35 °C, this temperature was maintained for 8 min, then increased to 45 °C at a rate of 1.5 °C/min, increased to 150 °C at a rate of 3 °C/min, increased to 180 °C at a rate of 4 °C/min, increased to 210 °C at a rate of 3.6 °C/min where it was held for 14.51 min; the total time of analysis was 80 min. The injector was always maintained at 250 °C. The temperature of the transfer line was fixed to 220 °C. The mass spectrometer was operated in the mass range of 10 – 350 amu at a scan rate of 1 s/scan and a manifold temperature of 180 °C. The GC-MS was operated through the Saturn GC-MS Version 5.2 software (*Varian,* Walnut Creek, CA, USA). The volatile compounds were identified by comparison to analytical standards and to data in the NIST–92 library.

The quantification of these chemical substances was performed choosing the three masses of each compound showing the highest intensities [16, 17].

The olive oil pigments were determined by HPLC as previously reported [18].

2.3 Sensory analysis

A panel composed by eight assessors trained to virgin olive oil sensory analysis was used. Oil samples (15 g) were presented to each assessor in amber-colored glasses at room temperature (20 °C) to be scored using a graphical scale of 10 cm. The following descriptors were chosen: fruity, cut-grass, artichoke, hay-like, green apple, floral, tomato, almond and fatty to describe aroma and sweet, bitter and pungent to define taste while green and yellow were used as color descriptors [8].

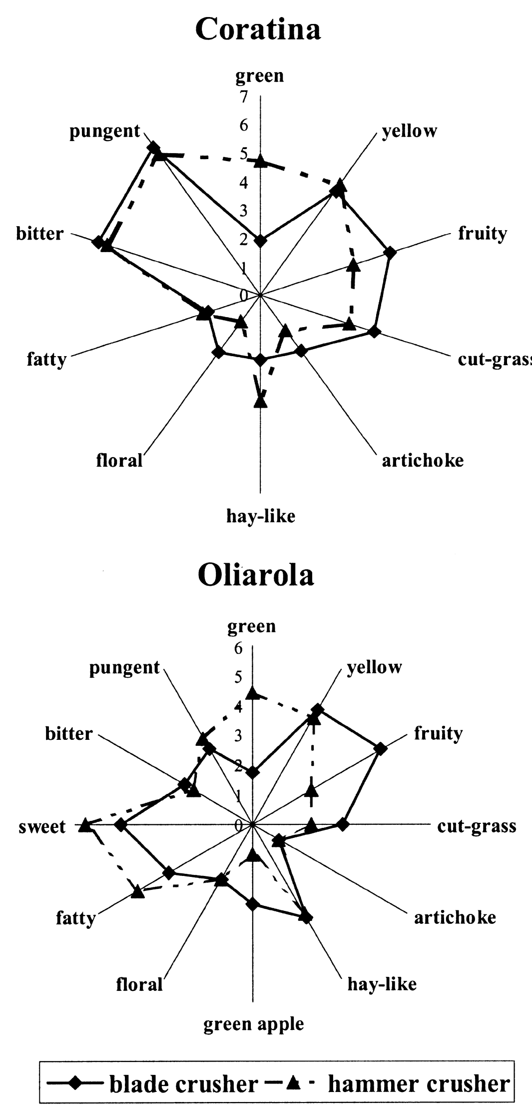

Fig. 3. Sensory profile of virgin olive oils from *Coratina* and *Oliarola* cultivars obtained using blade and hammer crushers.

Tab. 2. Quality characteristics† of virgin olive oils obtained from *Coratina* and *Oliarola* cultivars using hammer and blade crushers.

	Coratina		*Oliarola*	
	Hammer crusher	Blade crusher	Hammer crusher	Blade crusher
Free fatty acids [% oleic acid]	0.49 ± 0.04[a]	0.47 ± 0.04[a]	0.52 ± 0.05[b]	0.48 ± 0.02[b]
Peroxide value [meq O_2/kg]	8.5 ± 0.28[a]	8.4 ± 0.24[a]	8.8 ± 0.42[b]	8.0 ± 0.14[b]
K_{232}	1.935 ± 0.043[a]	1.953 ± 0.041[a]	2.397 ± 0.04[b]	2.304 ± 0.03[b]
K_{270}	0.129 ± 0.014[a]	0.150 ± 0.012[a]	0.174 ± 0.028[b]	0.185 ± 0.021[b]
ΔK	–0.006	–0.007	–0.006	–0.005
Total phenols‡	328.5 ± 58.7[a]	383.0 ± 38.2[a]	174.7 ± 15.3[b]	186.7 ± 27.2[b]

† Mean values ± SD of three independent determinations. For each cultivar, values in each row bearing the same superscript are not significantly different ($p < 0.05$).

‡ Evaluated colorimetrically and expressed in mg kg^{-1} as 3,4-DHPEA equivalent.

Tab. 3. Phenolic compounds† of virgin olive oils obtained from *Coratina* and *Oliarola* cultivars using hammer and blade crushers.

	Coratina		*Oliarola*	
	Hammer crusher	Blade crusher	Hammer crusher	Blade crusher
3,4-DHPEA	2.3 ± 1.7[a]	1.1 ± 0.4[a]	0.6 ± 0.4[b]	0.5 ± 0.5[b]
p-HPEA	2.9 ± 0.1[a]	3.8 ± 0.9[a]	7.3 ± 5.7[b]	8.1 ± 7.1[b]
Vanillic acid	0.3 ± 0.1[a]	0.5 ± 0.1[a]	0.3 ± 0.2[b]	0.3 ± 0.3[b]
Caffeic acid	0.3 ± 0.1[a]	0.3 ± 0.1[a]	0.4 ± 0.1[b]	0.4 ± 0.1[b]
3,4-DHPEA-EDA	301.6 ± 25.2[a]	366.6 ± 36.4[a]	53.3 ± 9.4[b]	54.6 ± 5.2[b]
p-HPEA-EDA	52.6 ± 9.0[a]	67.0 ± 10.6[a]	43.0 ± 6.5[b]	40.6 ± 5.7[b]
p-HPEA-ester	36.1 ± 8.7[a]	42.4 ± 2.2[a]	32.4 ± 22.0[b]	37.6 ± 10.4[b]
3,4-DHPEA-EA	257.0 ± 4.8[a]	269.4 ± 31.0[a]	97.2 ± 9.6[b]	124.6 ± 19.4[b]

† Mean values ± SD of three independent determinations. For each cultivar, values in each row bearing the same superscript are not significantly different ($p < 0.05$). Evaluated by HPLC and expressed as mg kg^{-1}.

2.4 Statistical analysis

Mean values are reported in the tables. To know the significance of differences between the mean values of the oils from the two different crushers, one way analysis of variance (ANOVA) using the paired *t*-test was employed. Statgraphics Version 6.1 (*Statistical Graphics Corp*., 1992, *Manugistics*, Inc., Rockville, MD, USA) was used to perform all statistical analyses.

3 Results and discussion

For this study, *Coratina* and *Oliarola* cultivars were used. The oils from these cultivars were separately extracted from homogeneous batches of fruits using identical processing procedures, except for the crushing step performed as described in the Materials and methods section.

Quality parameters of virgin olive oils of both cultivars showed no significant differences in free fatty acids, peroxide value, UV absorption and total phenols content owing to the use of a specific crusher (Tab. 2). Neither was the concentration of phenolic compounds, identified and evaluated by the HPLC analysis, significantly affected by the different crusher used (Tab. 3).

On the contrary, the concentration of some volatile compounds of virgin olive oils obtained (Tab. 4) was significantly affected by the crusher used. In particular the oils obtained using the blade crusher showed higher concentrations of C6 aldehydes such as 1-hexanal and *trans*-2-hexenal and of some esters such as hexyl acetate, 3-hexenyl acetate and *cis*-4-hexenyl acetate with respect to the oils obtained using the hammer crusher. These aldehydes and esters are correlated to the cut-grass and floral sensory notes of virgin olive oil, respectively [8, 19]. The use of the blade crusher caused lower concentrations of some alcohols such as 1-hexanol and *trans*-2-hexen-1-ol that are related to hay-like sensory note [19].

Tab. 4. The volatile compounds† of virgin olive oils obtained from *Coratina* and *Oliarola* cultivars using hammer and blade crushers.

	Coratina		*Oliarola*	
	Hammer crusher	Blade crusher	Hammer crusher	Blade crusher
3-Pentanone	13.2 ± 3.2[a]	7.0 ± 0.6[a]	25.1 ± 4.3[c]	7.3 ± 0.9[d]
1-Pentanal	9.4 ± 1.9[a]	11.4 ± 2.1[a]	18.7 ± 2.2[c]	4.7 ± 1.0[d]
1-Penten-3-one	170.0 ± 17.6[a]	179.4 ± 9.6[a]	169.3 ± 13.1[c]	159.0 ± 23.8[c]
1-Hexanal	125.2 ± 20.5[a]	284.5 ± 14.1[b]	137.1 ± 16.4[c]	289.3 ± 9.9[d]
1-Butanol	16.4 ± 0.9[a]	5.4 ± 1.7[b]	25.9 ± 0.3[c]	9.0 ± 0.5[d]
2-Pentenal	16.6 ± 4.8[a]	18.6 ± 2.0[a]	19.4 ± 0.8[c]	16.4 ± 3.0[c]
1-Penten-3-ol	162.0 ± 24.3[a]	184.9 ± 19.7[a]	133.1 ± 6.7[c]	138.5 ± 26.2[c]
Cis-2-hexanal	128.1 ± 7.2[a]	72.9 ± 1.6[b]	123.5 ± 9.0[c]	76.0 ± 10.6[d]
2-Methyl-1-butanol	68.9 ± 7.1[a]	26.7 ± 2.0[b]	57.4 ± 6.9[c]	31.4 ± 4.5[d]
Trans-2-hexenal	6606.5 ± 688[a]	8475.8 ± 765.4[b]	5359.5 ± 360.3[c]	7149.7 ± 717.7[d]
1-Pentanol	10.3 ± 1.8[a]	7.3 ± 0.9[a]	14.3 ± 2.8[c]	8.4 ± 1.6[c]
Hexyl acetate	1.5 ± 1.4[a]	3.6 ± 0.2[b]	6.6 ± 0.0[c]	14.1 ± 1.1[d]
3-Hexenyl acetate	3.2 ± 0.9[a]	5.4 ± 0.3[b]	3.6 ± 0.1[c]	4.7 ± 0.5[d]
Cis–4-hexenyl acetate	2.7 ± 1.3[a]	18.2 ± 0.8[b]	3.9 ± 0.5[c]	14.1 ± 0.7[d]
Cis-2-heptenal	3.8 ± 0.3[a]	2.9 ± 0.3[a]	7.9 ± 1.1[c]	4.1 ± 1.1[d]
Trans-2-hexenil-acetate	0.3 ± 0.2[a]	0.2 ± 0.1[a]	0.2 ± 0.1[c]	0.2 ± 0.1[c]
1-Hexanol	205.7 ± 30.9[a]	97.7 ± 9.0[b]	234.7 ± 13.7[c]	115.1 ± 15[d]
Trans-3-hexen-1-ol	10.1 ± 2.5[a]	3.7 ± 1.7[b]	7.5 ± 1.2[c]	5.1 ± 0.7[c]
Cis-3-hexen-1-ol	102.0 ± 23.6[a]	113.2 ± 18.0[a]	95.3 ± 4.5[c]	105.5 ± 12.0[c]
1-Nonanal	158.7 ± 22.5[a]	130.4 ± 26.0[a]	111.6 ± 13.4[c]	114.5 ± 15.2[c]
Trans-2-hexen-1-ol	1061.5 ± 105.8[a]	520.4 ± 18.8[b]	1065.4 ± 76.3[c]	566.4 ± 64.0[d]
Cis-2-hexen-1-ol	155.1 ± 24.2[a]	145.6 ± 17.6[a]	147.9 ± 3.3[c]	138.1 ± 3.2[c]
1-Hexen-3-ol	8.7 ± 1.4[a]	9.4 ± 1.7[a]	10.0 ± 2.3[c]	8.5 ± 1.9[c]
1-Octanol	106.2 ± 5.1[a]	125.7 ± 18.9[a]	111.4 ± 5.6[c]	116.9 ± 15.6[c]

† Mean values ± SD of three independent determinations. For each cultivar, values in each row bearing different superscript are significantly different ($p < 0.05$). Evaluated by GS-MS and expressed as µg kg^{-1}.

The sensory analysis of virgin olive oils (Fig. 3) confirmed the results mentioned above. The oils obtained using the blade crusher were characterized by an increase of cut-grass and floral sensory notes while hay-like sensory character increased in the oil when the hammer crusher was used. These results are related to the different crushing effect on the constitutive parts of the olive fruit. In fact, due to the different form of crushing members (Tab. 1 and Fig. 1), the blade crusher allows a combinated effect of percussion and cut, while the hammer crusher achieves essentially a percussion effect.

Finally, results related to the olive oil pigments (Tab. 5) showed lower concentrations of these compounds in the oils obtained using the blade crusher. Particularly the contents of β-carotene and chlorophyll *a* in the *Coratina* oil and the content of the pheophytin *a* in the *Oliarola* oil were significantly lower in the oils produced with a blade crusher. These differing concentrations confirm the different crushing effect on the constitutive parts of the olive fruit using the two crushers. In fact the use of the blade reduced the release of the pigments (present essentially in the peel tissues) in the oil during the malaxation step due to a lower degradation of the peel tissues.

4 Conclusions

The results obtained showed that the quality parameters and the phenolic composition of oils obtained were not significantly affected by the two different crushers used.

On the contrary, the oils obtained with the blade crusher showed an improved concentration of volatile compounds with significant increases of some compounds such as 1-hexanal and *trans*-2-hexenal and a reduction of alco-

Tab. 5. The pigments† of virgin olive oils obtained from *Coratina* and *Oliarola* cultivars using hammer and blade crushers.

	Coratina		*Oliarola*	
	Hammer crusher	Blade crusher	Hammer crusher	Blade crusher
β-Carotene	2.3 ± 0.1[a]	1.7 ± 0.1[b]	2.5 ± 0.2[c]	2.3 ± 0.3[c]
Pheophytin *a*	1.5 ± 0.3[a]	1.3 ± 0.2[a]	0.7 ± 0.1[c]	0.1 ± 0.1[d]
Pheophytin *b*	0.3 ± 0.2[a]	0.2 ± 0.1[a]	0.8 ± 0.2[c]	0.5 ± 0.1[c]
Chlorophyll *a*	1.3 ± 0.3[a]	0.5 ± 0.1[b]	1.7 ± 0.2[c]	1.4 ± 0.3[c]
Chlorophyll *b*	0.1 ± 0.1[a]	0.0 ± 0.0[a]	0.1 ± 0.1[c]	0.0 ± 0.0[c]
Lutein	0.7 ± 0.2[a]	0.6 ± 0.2[a]	0.9 ± 0.1[c]	0.7 ± 0.1[c]

† Mean values ± SD of three independent determinations. For each cultivar, values in each row bearing the same superscript are not significantly different ($p < 0.05$). Expressed as mg kg^{-1}.

hols such as 1-hexanol respective to oils from hammer crusher.

Moreover, the use of blades instead of hammers allowed to obtain oils with lower concentrations of pigments such as β-carotene and chlorophylls and consequently with a reduced intensity of green colour.

Finally, the sensory analysis showed that oils obtained using the blade crusher were characterized by an increase of cut-grass and floral sensory notes in comparison to oils produced with the hammer crusher.

In conclusion, oils produced with a blade crusher showed an improved organoleptic quality compared to oils obtained using a hammer crusher.

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