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TITLE

Influence of the feed pipe position of an industrial scale two-phase decanter on extraction efficiency and chemical-sensory characteristics of virgin olive oil

RUNNING TITLE

Decanter feeding impact on extraction efficiency and olive oil quality

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25

ABSTRACT

26 BACKGROUND: Nowadays, olive oil extraction is basically made by means of two-phase
27 decanters, which allow to reduce water consumption and leaching of phenolic compounds.
28 Despite this, most of the working settings derive from studies carried out on three-phase
29 decanters. Hence, aim of the present study has been assessing the influence of two-phase
30 decanter feed pipe position (FP) on the extraction efficiency and chemical-sensory
31 characteristics of virgin olive oil. Three different positions have been considered, at 825 mm
32 (FP1), 610 mm (FP2), and 520 mm (FP3) from the outlet of the oily phase.

33 RESULTS: Position FP3 allowed the highest oil recovery (up to 10%), the lowest percentage of
34 oil in the olive pomace and, in general, a regular trend in terms of oil extraction efficiency.
35 However, the oily must that came out of the decanter was not completely clean in terms of
36 residual content of solid sediment and water. The feeding position partially affected the profile
37 of antioxidant compounds.

38 CONCLUSION: In two-phase decanters, loading the olive paste close to the outlet of the oily
39 phase is recommended in order to increase the extraction efficiency without jeopardising the
40 chemical-sensory characteristics of virgin olive oil.

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KEYWORDS

43 Decanter; Olive oil extraction; Virgin olive oil; Extraction efficiency; Quality.

INTRODUCTION

44

45 Virgin olive oil production has been deeply studied from the field stage, to the promotion of
46 main product, and including the disposal of the by-products.¹⁻⁵ Over the years, due to the
47 discontinuous processing and high management costs, the olive oil extraction system has
48 undergone profound changes: the traditional olive processing by pressure has been replaced by
49 more efficient centrifugation systems based on two-phase and three-phase decanters.^{6,7}
50 However, the latter are known to have disadvantages such as: i) high energy and water
51 consumption for the dilution of olive paste; ii) excessive volume of vegetation water, which
52 causes high disposal costs; iii) loss of minor water-soluble constituents of the oils, in particular
53 phenolic substances; iv) variation of organoleptic characteristics of the oils.^{8,9}

54 In addition, Cauteruccio et al. highlighted other critical aspects, due to the thickness of the cloak
55 and to the length and thickness of the auger hollow shaft.¹⁰ The same authors have still found
56 difficulties in modelling a machine such as a three-phase decanter, due to its geometry and the
57 composition of the fluid inside it; in fact, the olive paste, depending on the position along the
58 axis, varies considerably its composition in terms of specific weight and density. On the other
59 hand, already in a previous experimental-theoretical study, some parameters such as solid flow
60 coefficient, drag number, and dynamic productivity coefficient have been considered to design
61 the three-phase decanters, further used by Boncinelli et al.^{11,12} This has led to the development
62 of the three-phase water-saving decanter, equipped with auger/drum differential speed control
63 systems, and assisted by automatic control mechanisms.¹³ Moreover, the same authors found
64 that in order to produce an additional liquid phase separation effect from the solid matrix, the
65 introduction of barriers near the decanter drainage mouth, the use of variable speed screw
66 conveyor, and the modification of the contour cone profile allowed a wide range of processing
67 conditions, such as: water and oil flow rates, differential speed of the cochlea with respect to the
68 drum, drainage levels of the water and oil.¹³ By the proper adjustments of the above parameters,
69 the extraction efficiency was constant in a very wide range of feed rates (1800-2800 kg h⁻¹),
70 higher than the previous decanters, especially at low dilution of the olive paste.¹³ Similar results
71 have been reported recently by Squeo et al.¹⁴ In a study of optimization of a three-phase water-

72 saving decanter it was noted that the extraction efficacy was directly related to the time of paste
73 permanence and inversely to the differential speed (ΔN).¹⁵ Tamborrino et al. investigated,
74 instead, on rheological properties, energy consumption, oil yield, and quality when the calcium
75 carbonate was used during the extraction process.¹⁶

76 The two-phase decanter, on the contrary, has been studied mainly by a qualitative point of
77 view.^{17,18} In particular, Klen et al. evaluated the content of phenols by comparing different
78 extraction systems; Caponio et al. evaluated the chemical composition of extra virgin olive oil
79 in function of the decanter set-up (two- and three-phase) and the differential speeds between
80 drum and cochlea ($\Delta N16$ and $\Delta N11$).^{19,20} Moreover, Giovacchino and co-authors found that by
81 using a decanter capable of working at two- and three-phase, comparable yields were obtained
82 when in the two-phase process the feed rate was reduced to 60-70% of the theoretical value
83 advised; Ayr et al., instead, highlighted the importance of paste preparation in two-phase
84 separation by studying a fluid-dynamic simulation model adaptable to two-phase extraction.²¹⁻²³

85 Finally, a study carried out by Tamborrino et al. using an innovative decanter, able to switch
86 from three- to two-phase processing without interrupting the extraction, allowed a correlation
87 between the flow rate, the oil outlet level, the drum/cochlea ΔN , and the extraction efficiency.⁷

88 In this framework, the need of a further investigation on the working parameters of the two-
89 phase decanter is highlighted, since many of the technical solutions adopted are currently based
90 on those used in three-phase machines. The purpose of this work was therefore to investigate
91 the influence of the position of the olive paste feed pipe (FP) in a two-phase industrial-scale
92 decanter capable of working at high feed rates, by evaluating the results in terms of extraction
93 efficiency and chemical-sensory characteristics of the virgin olive oil obtained. Then, three
94 different positions have been considered, at 825 mm (FP1), 610 mm (FP2), and 520 mm (FP3)
95 from the outlet of the oily phase.

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97

EXPERIMENTAL

98 **Industrial olive oil extraction plant**

99 The experimental tests were carried out in an industrial olive oil mill (Cooperativa Produttori
100 Olivicoli) located in Bitonto (Apulia, Italy) using a two-phase decanter (REX250 model,
101 Amenduni Nicola S.p.a., Modugno, Italy). In this decanter, olive paste is fed from the opposite
102 side to the conical section (i.e. the “beach” zone), with the possibility to move the feed pipe
103 through the drum section, depending on the ripeness degree of olives and the need to set the
104 two- (oil and pomace) or three-phase (oil, water, and pomace) mode, which is made possible by
105 the simple adjustment of water and oil levels. The internal screw conveyor rotates faster than the
106 decanter drum, and the residence time of the pomace can be adjusted by varying the differential
107 speed (ΔN).

108 Olive fruits (*Olea europaea* L.) of Coratina cultivar, mechanically harvested in the fifteen last
109 days of November 2016, after leaf-removal, were milled unwashed within 24 h. Two lots of
110 olives were processed in different days, characterized by a maturity index, determined as
111 reported in Squeo et al. equal to 1.07 and 1.39, respectively.²⁴

112 A lot of about 12,000 kg of olives was considered for each trial. Figure 1 shows the flux
113 diagram of olive oil processing. In particular, each olive lot was milled with a hammer-crusher
114 (A60 model, Amenduni Nicola S.p.a., Modugno, Italy) operating at 1,500 rpm, with a
115 processing capacity of 7,000 kg h⁻¹ and grid with holes of 6 mm of diameter, then the olive paste
116 was transferred in the malaxer (6V1000 model, Amenduni Nicola S.p.a., Modugno, Italy). After
117 malaxation (90 min at 27±1 °C), the paste was pumped into a two-phase decanter (REX250
118 model, Amenduni Nicola S.p.A., Modugno, Italy) – operating at 2,800 rpm (bowl) and 2,825
119 rpm (screw), with a processing capacity of 6,000 kg h⁻¹ without dilution with water – in three
120 different feed pipe (FP) positions: at 825 mm (FP1), 610 mm (FP2), and 520 mm (FP3) from the
121 outlet of the oily phase, as shown in Figure 2. Finally, the oily must was cleaned with a vertical
122 centrifuge (A3500 model, Amenduni Nicola S.p.a., Modugno, Italy), operating at 6,400 rpm and
123 2,000 L h⁻¹. During olive processing, three different samples (every 5 minutes) of oily must
124 (OM), olive pomace (OP), and cleaned oil (O) were collected for each feed pipe position.
125 Moreover, three samples of olive pastes, for each trial, were collected at the end of malaxation
126 step for the viscosity measurements.

127

128 **Analytical determinations**

129 The viscosity measurements were carried out by a Viscotester VT 550 HAAKE (Thermo Fisher
130 Scientific Inc., Waltham, MA, USA) using 600 mL of olive paste, put into 1,000 mL glass
131 containers and conditioned at same malaxation temperature in a thermostatic bath (27 °C). To
132 interpret the experimental results in terms of viscosity, the acquired torque-speed data and scale
133 readings were converted into shear stress-shear rate relationships using numerical conversion
134 values using the instrument calibration map. The power-law model was used to calculate the
135 apparent viscosity and flow behaviour index from the shear rate using the following Eq (1):

$$136 \quad \eta_{app} = k \gamma^{n-1} \quad (1)$$

137 where η_{app} is the apparent viscosity, γ is the shear rate (s^{-1}), n is the flow behaviour index
138 (dimensionless), k is the consistency index ($Pa \ s^n$). This model is widely used in fluid dynamics
139 analysis when studying biological fluids as the olive paste that is a mixture of three components:
140 olive oil, vegetation water, solid particles. While olive oil and pure water (not vegetation water)
141 are typical Newtonian fluid characterized by a constant viscosity coefficient, olive paste has
142 more complex rheological behaviour. Its viscosity cannot be considered as constant and depends
143 on several parameters.²⁵ The simplest model that can be used for this fluid is just the power-law
144 model.

145 The extraction efficiency (EE) was calculated as the ratio between the weight of virgin olive oil
146 (W_{oil}) obtained at the end of the process, and the weight of oil contained in the corresponding
147 olives determined by Soxhlet extraction (W_{olive}). Results of Eq. (2) were expressed as
148 percentage (%).

$$149 \quad EE = \frac{W_{oil}}{W_{olive}} \times 100 \quad (2)$$

150 The moisture ($g \ 100 \ g^{-1}$) and total oil ($g \ 100 \ g^{-1}$) content of olives and olive pomaces, as well as
151 the free fatty acids (FFA), peroxide value (PV), and spectrophotometric constants of the
152 extracted oil were determined as described in a previous paper.²⁶ The moisture content of oils
153 was determined at $103 \pm 2 \ ^\circ C$, according to the ISO method 662/2016, while the solid impurity

154 content of the oils was determined after centrifugation at $4,625 \times g$ (SL 16R Centrifuge, Thermo
155 Fisher Scientific Inc., Waltham, MA, USA), and the results were expressed as a percentage
156 ratio.²⁷ The determination of total phenolic compounds by UV spectrophotometry, chlorophylls,
157 carotenoids, and tocopherols was carried out as reported in a previous paper.¹⁶ The
158 determination of the phenolic compounds by HPLC was carried out as described in Caponio et
159 al.²⁸ For the determination of the volatile compounds, the oil samples (1 ± 0.005 g) were
160 weighed into 20 mL vials, sealed with a screw top aluminium cap and pierceable butyl rubber
161 septa, and submitted to the (SPME/GC-MS) in the conditions reported by Caponio et al.²⁰ The
162 sensory analysis was performed by a trained panel made of eight judges, experienced in olive oil
163 sensory evaluation in accordance with the Commission Regulation (ECC) No 2568/91.²⁹

164

165 **Statistical analysis**

166 Results were expressed as mean \pm standard deviation (SD) of three measurements for the
167 analytical determination. Analysis of variance (ANOVA), followed by Tukey HSD test for
168 multiple comparisons was carried out on the experimental data by means of XLStat software
169 (Addinsoft SARL, New York, NY, USA). Differences were considered significant at $p \leq 0.05$.

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171

RESULTS AND DISCUSSION

172 **Decanter performance in relation to feed pipe position**

173 Figure 3 reports the rheological profiles of the shear stress versus shear rate for each olive paste
174 after 90 min of malaxation. As shown, the apparent viscosity of the samples decreased with the
175 share rate, in accordance to Tamborrino et al., due to the behaviour as pseudoplastic material of
176 the olive paste.¹⁶ The slightly lower values of apparent viscosity of the samples from the second
177 trial could be imputable to higher maturity index of the olives, which causes a higher amount of
178 oil in the olives (data not shown) and an easier breaking of tissues and cells.³⁰ This allows an
179 increase of the availability of the liquid fraction and, consequently, the separation between the
180 solid and liquid phases within the olive paste and the coalescence of the oil drops.

181 Table 1 reports for each trial the moisture and oil content of the olive pomace after
182 centrifugation with two-phase decanter, as well as the extraction efficiency. The moisture
183 content of the pomace did not vary significantly in relation to the different feeding position of
184 the olive paste in the decanter, while the residual oil content was significantly lower in FP3
185 compared to the other two positions. Extraction efficiency was also significantly higher in FP3,
186 reaching the values of 83.24% in the first trial and 84.32% in the second, confirming the better
187 performances of the FP3 position. Considering the results in both the trials, it is highlighted an
188 average increase of extraction efficiency over FP1 and FP2 above 3%. Such a percentage is
189 relevant in the industrial production of virgin olive oil.

190 Figure 4 reports the moisture and solid content of the oily must withdrawn at the decanter outlet
191 in the second trial. The oily must obtained with the FP3 position had significantly higher
192 moisture content and impurities than the other two samples (FP1 and FP2), which did not show
193 significant differences between them. In particular, more than 5% of moisture and more than
194 17% solids were observed in FP3 samples, whereas less than 2% moisture and approximately
195 8% solids were ascertained in FP1 and FP2 samples.

196 The explanation of such trend could be found in the different resident time experienced by the
197 oily phase in the decanter. In fact, when olive paste is fed in FP3, i.e. the closest position to the
198 oily must outlet, the oily phase has a resident time no long enough to clarify, differently from
199 what happens when the olive paste is fed in a deeper section through the drum, which forces the
200 oily phase to a longer pathway before leaving the decanter (FP1 and FP2). Nonetheless, it is
201 worth to note that no significant difference in content of moisture and impurities was further
202 detected after the oil finishing by means of the vertical centrifuge (data not shown), that is an
203 usual step in the industrial production of virgin olive oil by means of decanters.

204 In the light of the aforementioned results it is possible to conclude that in two-phase decanter is
205 recommended to load the olive paste close to the outlet of the oily phase. Moreover, it should be
206 considered that in the two-phase decanter, in which the feeding is carried out on the opposite
207 side from the conical end, the feed pipe does not reach such lengths able to reach the best
208 position for the introduction into the decanter (Fig. 2); as a consequence, these machines do not

209 require any vibrational check-ups when working at maximum rotation speed. From a
210 constructive point of view, the vibration of the auger shaft is essentially due to the mass of the
211 reducer to which it is attached and to the auger mass, so it can vibrate depending on the length
212 and thickness. On the other hand, the auger shaft is subjected to several design constraints: the
213 outer diameter can not be increased because it is constrained into the inner diameter of the
214 bearings and, in most cases, increasing the bearing diameter would cause a significant increase
215 of the costs.¹⁰ In the two-phase decanters, in which the feeding occur at the conical side, the
216 possibility of varying the relative rotation speed between the auger and the drum (ΔN) is
217 particularly important, both for flexural equilibrium and for the success of the separation
218 process. Indeed, ΔN is one of the parameters which, acting on the olive paste resident time, can
219 effectively improve the separation efficiency without needing excessive rotational speeds, if
220 appropriately adjusted. In this perspective, for the two-phase process, the position where to feed
221 the olive paste should be preferably on the opposite side of the cone, allowing to move the
222 injection tube into more internal drum sections depending on the physical characteristics of
223 olive paste and according to the possible need to set the machine in the three-phase mode.

224

225 **Virgin olive oil quality**

226 Table 2 shows the results of the chemical and sensory analyses performed on the oils. All
227 samples exhibited acidity, peroxide value and spectrophotometric constant values within the
228 limits set by the rules for the classification of oil as extra virgin, indicating the good quality of
229 the olives used.³¹ In fact, the acidity and peroxide values did not exceed the value of 0.4% and 8
230 mEq O₂ kg⁻¹, respectively. Slightly higher K₂₇₀ values were observed for the oils of the second
231 trial. These latter samples, on the other hand, showed higher average values of lipophilic
232 (carotenoids, tocopherols) and hydrophilic (phenolic compounds) antioxidants, as well as more
233 marked pungent sensory notes, compared to those of first trial.

234 The feeding position did not cause substantial changes in the chemical profile, as well as in the
235 sensorial characteristics of the oils. Significantly influenced, in fact, were only the lipophilic
236 antioxidant compounds and, in one of the trials, the pungent note and the content of

237 chlorophylls. Differently, the phenolic content of the oils was not significantly influenced by
238 machine setting. In any case, although without statistical significance, in both the trials FP3
239 samples approximately showed a 10% lower phenolic content than FP1 and FP2. This result
240 could be explained by considering the composition of the oily must. In fact, as shown in Figure
241 4, the oily must obtained from FP3 position had a significantly higher content of water and
242 solids. Given the great affinity of the phenolic compounds for the aqueous phase it can be
243 thought that they are preferably distributed in the latter and therefore lost with it in the
244 subsequent processing step of oil finishing by vertical centrifuges.

245 Carotenoids are a group of tetraterpenoids consisting of isoprene units, and are important
246 liposoluble antioxidants present in oils and fats.³² FP1 feeding caused a significant reduction in
247 carotenoids content respect to both FP2 and FP3 in the second trial, and to FP2 in the first trial.
248 This result can be explained considering the low polarity of these compounds and the behaviour
249 of the pomace in the decanter. In fact, FP1 position caused a quick discharge of the pomace
250 from the decanter and therefore made the pomace retain a significantly higher amount of oil
251 compared to other feeding positions (Table 1). Thus, given the carotenoids affinity for the oily
252 phase, it can be supposed that their dissolution in the pomace was higher in the FP1 samples
253 than in the others.

254 A similar trend was found for α -tocopherol and, consequently, for the total tocopherols,
255 representing the former about 90% of total tocopherols in virgin olive oils.³³ In particular, the
256 FP2 oil had significantly higher content of tocopherols than FP1, in both the trials. This may be
257 due to the chemical characteristics of these compounds which, similarly to carotenoids, have
258 hydrophobic character. However, unexpectedly, a decrease in the tocopherols content was also
259 observed in the FP3 oils compared to the FP2 samples.

260 Regarding the sensory characteristics of the oils, there was a significant influence of the
261 decanter feed pipe position on the pungent note in the case of the first trial, with significantly
262 higher values for the FP2 samples, according to the highest total phenolic content observed in
263 the same samples. The chlorophyll content showed a significant difference only in the second
264 trail for FP1 samples, which had a higher content than the others.

265 Table 3 shows the content of the individual phenolic compounds determined by HPLC. The oils
266 from the second trial had a higher average content of phenolic compounds compared to the first
267 trial. Overall, the feed pipe position of the olive paste in the decanter has weakly affected the
268 phenolic profile. Consistently with total phenols (Table 2), FP3 position caused a reduction of
269 the phenolic content, significant in the first trial. This result was a consequence of the
270 significant decrease in some of the derivative forms of secoiridoids, in particular the dialdehydic
271 form of the elenolic acid linked to the hydroxytyrosol (3,4-DHPEA-EDA) and tyrosol (*p*-
272 HPEA-EDA), known to be among the most abundant in virgin olive oils.³⁴ In particular, the
273 dialdehydic form of elenolic acid linked to the tyrosol (*p*-HPEA-EDA), also known as
274 oleocanthal, has long been recognized as the main responsible for the pungency of virgin olive
275 oils and associated with anti-inflammatory activity similar to that of ibuprofen.³⁵ Nevertheless,
276 in our study no straight correlation has been observed between oleocanthal and samples pungent
277 note (Table 2). In the case of the second trail, the only significant differences involved vanillic
278 acid and apigenin. However, a trend similar to that found for the first trial was observed for 3,4-
279 DHPEA-EDA, but not for the oleocanthal.

280 Table 4 shows the volatile compounds found in oils. In both the trails, *trans*-2-hexenal was the
281 most abundant compound, as widely documented in literature.^{28,36,37} The feed pipe position did
282 not lead to significant changes in the volatile profile and only a few significant differences were
283 observed. Also for volatiles, the behaviour was different in the tests suggesting a possible
284 influence of the ripening degree of olives.

285 Considering the first trial, loading the olives paste in FP1 resulted in a significant decrease in
286 *trans,trans*-2,4-hexadienal, 1-penten-3-one and a significant increase in 1-hexanol, the latter
287 only with respect to FP2 test. Further, the FP2 test showed significantly higher values of ethyl
288 acetate, probably linked to the particular batch of olives used, although derived from a
289 homogeneous batch, and 1-penten-3-one. The FP3 test had significantly lower values of 2-
290 methyl-4-pentenal, 1-penten-3-one and limonene respect to the others. In the second trial,
291 significant decreases of ethanol, *trans*-2-hexen-1-ol and ethyl acetate were observed in FP1,
292 while only *trans*-3-hexen-1-ol acetate in FP2. Finally, FP3 oils had significantly higher content

293 of ethanol and *trans*-3-hexen-1-ol acetate. Tentatively, the explanation of what observed might
294 be linked to the polarity of the volatile compounds, similarly to what observed for phenolics.
295 Indeed, more polar groups such as esters and aldehydes could be more likely found in FP3 oils,
296 richer in water at exit from the decanter, than the other samples. However, such behaviour was
297 not observed in both the trials and it was more evident and statistically significant only in the
298 case of trial II.

299

300

CONCLUSIONS

301 Considering the results about the performances of the two-phase decanter, with the olives used
302 in this experimental study, the position of the feed pipe tube closer to the oil discharge section
303 (FP3) allowed to reach the highest oil recovery and a regular trend in terms of extraction
304 efficiency. On the other hand, the extracted oil was found to contain more sediment and water.
305 The farthest positions from the liquid exit are the worst considering the extraction efficiency,
306 but make it possible to have cleaner oil, although the cleaning of the oily must, carried out in the
307 vertical separator, is usually included in any olive processing plant.
308 Interestingly, our findings highlighted as the different decanter feeding position is also able to
309 influence the balance of lipophilic/hydrophilic antioxidants in the oils. In fact, when the olive
310 paste feeding takes place nearest to the solid discharge (FP1), the resulting oil is richer in
311 phenols and contains less tocopherols and carotenoids than the opposite FP3 position.

312

313

CONFLICT OF INTERESTS

314 The authors declare that there is no conflict of interests regarding the publication of this paper.
315 Lucio Brunetti, Pasquale Catalano, and Biagio Bianchi edited the “Decanter performance in
316 relation to feed pipe position” section; while Francesco Caponio, Giacomo Squeo, Antonella
317 Pasqualone, Carmine Summo, and Vito M. Paradiso edited the “Virgin olive oil quality”
318 section.

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320

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326

327

FIGURE CAPTION

328 **Figure 1.** Experimental plan and sampling points.

329 **Figure 2.** Different positions of the decanter feed pipe, at 825 mm, 610 mm, and 520 mm from
330 the outlet of the oily phase.

331 **Figure 3.** Shear stress versus shear rate of three olive pastes (P1, P2, P3) collected in two
332 different olive processing trials. First trial (I-T) corresponded to olives having maturity index
333 lower than those of second trial (II-T).

334 **Figure 4.** Moisture and insoluble impurity content of the oily must obtained in an olive
335 processing trial with olives having a maturity index of 1.39 (second trial) ($n=3$). Three different
336 decanter feed pipe (FP) positions were considered, positioned at 825 mm (FP1), 610 mm (FP2),
337 and 520 mm (FP3) from the outlet of the oily phase.

- 339 1) Baiano A, Terracone C, Viggiani I and Nobile M, Effects of cultivars and location on
340 quality, phenolic content and antioxidant activity of extra-virgin olive oils. *J Am Oil Chem*
341 *Soc* **90**: 103-111 (2012).
- 342 2) Catalano P, Fucci F, Giametta F, La Fianza G and Bianchi B, 2013. Vibration analysis
343 using contactless acquisition system. In: *Proceedings of SPIE SeTBio Volume 8881*
344 (2013).
- 345 3) Amirante P, Bianchi B, Catalano P and Montel GL, Theoretical-experimental analysis of
346 vegetable water distribution on cropland with a prototype tank-truck spreader. *J Agr Eng* **3**:
347 27-35 (2005).
- 348 4) Bianchi B, Papajova I, Tamborrino R and Ventrella D, Characterization of composting
349 mixtures and compost of rabbit by-products to obtain a quality product and plant proposal
350 for industrial production. *Veterinaria Italiana* **51**: 51-61 (2015).
- 351 5) Catalano P, Bianchi B, Tamborrino A, Leone A and Martinelli C, Full scale composting
352 tests of organic mixtures based on "two phase" pomace using a prototype of turning
353 machine. In: *Proceedings of 36th CIOSTA & CIGR Section V Conference* (2015).
- 354 6) Bianchi B, Tamborrino A and Santoro F, Assessment of the energy and separation
355 efficiency of the decanter centrifuge with regulation capability of oil water ring in the
356 industrial process line using a continuous method. *J Agr Eng* **44**: 278-282 (2013).
- 357 7) Tamborrino A, Leone A, Romaniello R, Catalano P and Bianchi B, Comparative
358 experiments to assess the performance of an innovative horizontal centrifuge working in a
359 continuous olive oil plant. *Biosyst Eng* **129**: 160-168 (2014).
- 360 8) Catalano P, Pipitone F, Calafatello A and Leone A, Productive efficiency of decanters with
361 short and variable dynamic pressure cones. *Biosyst Eng* **86**: 459-464 (2003).
- 362 9) Roig A, Cayuela ML and Sanchez-Monedero MA, An overview on olive mill wastes and
363 their valorization methods. *Waste Manag* **26**: 960-969 (2006).
- 364 10) Cauteruccio G, Papalinia S, Capriottia G and Rocchio G, Studio e ottimizzazione di un
365 decanter per il trattamento di fluidi alimentari. In: *Proceedings of XXXVII AIAS, 37th*
366 *Conference of Italian Association for Stress Analysis* (2008).
- 367 11) Amirante R and Catalano P, Fluid dynamic analysis of the solid-liquid separation process
368 by centrifugation. *J Agric Eng Res* **77**: 193-201 (2000).
- 369 12) Boncinelli P, Daou M, Cini E and Catalano P, A simplified model for designing and
370 regulating centrifugal decanters for olive oil production. *Transactions of the ASABE* **52**:
371 1961-1968 (2009).
- 372 13) Amirante P, Baccioni L, Catalano P and Montel GL, Nuove tecnologie per l'estrazione
373 dell'olio di oliva: il decanter a cono corto a pressione dinamica variabile e controllo della
374 velocità differenziale tamburo/coclea. *Riv Ital Sostanze Grasse* **76**: 129-140 (1999).
- 375 14) Squeo G, Tamborrino A, Pasqualone A, Leone A, Paradiso, VM, Summo C and Caponio F,
376 Assessment of the influence of the decanter set-up during continuous processing of olives
377 at different pigmentation index. *Food Bioprocess Tech* **10**: 592-602 (2017).

- 378 15) Altieri G, Di Renzo G and Genovese F, Horizontal centrifuge with screw conveyor
379 (decanter): Optimization of oil/water levels and differential speed during olive oil
380 extraction. *J Food Eng* **119**: 561-572 (2013).
- 381 16) Tamborrino A, Squeo G, Leone A, Paradiso VM, Romaniello R, Summo C, Pasqualone A,
382 Catalano P, Bianchi B and Caponio F, Industrial trials on coadjuvants in olive oil extraction
383 process: Effect on rheological properties, energy consumption, oil yield and olive oil
384 characteristics. *J Food Eng* **205**: 34-46 (2017).
- 385 17) Albuquerque JA, González J, García D and Cegarra J, Agrochemical characterisation of
386 “alperujo” a solid by-product of the two-phase centrifugation method for olive oil
387 extraction. *Bioresource Technol* **91**: 195-200 (2004).
- 388 18) Altieri G, Comparative trials and an empirical model to assess throughput indices in olive
389 oil extraction by decanter centrifuge. *J Food Eng* **97**: 46–56 (2010).
- 390 19) Klen TJ and Vodopivec BM, The fate of olive fruit phenols during commercial olive oil
391 processing: traditional press versus continuous two –and three-phase centrifuge. *LWT -*
392 *Food Sci Technol* **49**: 267-274 (2012).
- 393 20) Caponio F, Summo C, Paradiso VM and Pasqualone A, Influence of decanter working
394 parameters on the extra virgin olive oil quality. *Eur J Lipid Sci Tech* **116**: 1626-1633
395 (2014).
- 396 21) Di Giovacchino L, Costantini N, Serraiocco A, Surricchio G and Basti C, Natural
397 antioxidants and volatile compounds of virgin olive oils obtained by two or three-phases
398 centrifugal decanters. *Eur J Lipid Sci Tech* **103**: 279-285 (2001).
- 399 22) Di Giovacchino L, Costantini N, Ferrante ML and Serraiocco A, Influence of malaxation
400 time of olive paste on oil extraction yields and chemical and organoleptic characteristics of
401 virgin olive oil obtained by a centrifugal decanter at water saving. *Grasas Aceites* **53**: 179-
402 186 (2002).
- 403 23) Ayr U, Tamborrino A, Catalano P, Bianchi B and Leone A, 3D computational fluid
404 dynamics simulation and experimental validation for prediction of heat transfer in a new
405 malaxer machine. *J Food Eng* **154**: 30-38 (2015).
- 406 24) Squeo G, Silletti R, Summo C, Paradiso VM, Pasqualone A and Caponio F, Influence of
407 calcium carbonate on extraction yield and quality of extra virgin oil from olive (*Olea*
408 *europaea* L. cv. Coratina). *Food Chem* **209**: 65-71 (2016).
- 409 25) Boncinelli P, Catalano P, Cini E, Olive paste rheological analysis. *Transactions of the*
410 *ASABE* **56**: 237-243 (2013).
- 411 26) Caponio F, Squeo G, Monteleone J, Paradiso VM, Pasqualone A and Summo C, First and
412 second centrifugation of olive paste: Influence of talc addition on yield, chemical
413 composition and volatile compounds of the oils. *LWT - Food Sci Technol* **64**: 439-445.
414 (2015).
- 415 27) International Standard Organization, ISO/TC 34, Food products, Subcommittee SC 11,
416 Animal and vegetable fats and oils. ISO 662:2016 Animal and vegetable fats and oils -
417 Determination of moisture and volatile matter content. Geneve, Switzerland (2016).
- 418 28) Caponio F, Durante V, Varva G, Silletti R, Previtali M, Viggiani I, Squeo G, Summo C,
419 Pasqualone A, Gomes T and Baiano A, Effect of infusion of spices into the oil vs.

- 420 combined malaxation of olive paste and spices on quality of naturally flavoured virgin
421 olive oils. *Food Chem* **202**: 221-228 (2016).
- 422 29) Official Journal of the European Communities. European Community Regulation No.
423 2568/1991, N. L. 248 of September 5th, Publications Office of the European Union,
424 Bruxelles (1991).
- 425 30) Mafra I, Lanza B, Reis A, Marsilio V, Campestre C, De Angelis M and Coimbra M, Effect
426 of ripening on texture, microstructure and cell wall polysaccharide composition of olive
427 fruit (*Olea europaea*). *Physiol Plant* **111**: 439-447 (2001).
- 428 31) European Communities. European Community Regulation No. 1348/2013, N. L. 338 of
429 December 17th, Publications Office of the European Union, Bruxelles (2013).
- 430 32) Choe E and Min D, Mechanisms of antioxidants in the oxidation of foods. *Compr Rev*
431 *Food Sci Food Saf* **8**: 345-358 (2009).
- 432 33) Špika MJ, Kraljić K, Koprivnjak O, Škevin D, Žanetić M and Katalinić M, Effect of
433 agronomical factors and storage conditions on the tocopherol content of Oblica and
434 Leccino virgin olive oils. *J Am Oil Chem Soc* **92**: 1293-1301 (2015).
- 435 34) Bendini A, Cerretani L, Carrasco-Pancorbo A, Gómez-Caravaca AM, Segura-Carretero A,
436 Fernández-Gutiérrez A and Lercker G, Phenolic molecules in virgin olive oils: a survey of
437 their sensory properties, health effects, antioxidant activity and analytical methods. An
438 overview of the last decade. *Molecules* **12**: 1679-1719 (2007).
- 439 35) Beauchamp G, Keast R, Morel D, Lin J, Pika J, Han Q, Lee C, Smith A and Breslin P,
440 Phytochemistry: Ibuprofen-like activity in extra-virgin olive oil. *Nature* **437**: 45-46 (2005).
- 441 36) Kalua C, Allen M, Bedgood D, Bishop A, Prenzler P and Robards K, Olive oil volatile
442 compounds, flavour development and quality: A critical review. *Food Chem* **100**: 273-286
443 (2007).
- 444 37) Runcio A, Sorgonà L, Mincione A, Santacaterina S and Poiana M, Volatile compounds of
445 virgin olive oil obtained from Italian cultivars grown in Calabria. *Food Chem* **106**: 735-740
446 (2008).

Table 1. Pomace moisture, pomace oil content and oil extraction efficiency ($n=3$) determined in two different olive processing trials. First trial corresponded to olives having maturity index lower than those of second trial. Three different decanter feed pipe (FP) positions were considered, at 825 mm (FP1), 610 mm (FP2), and 520 mm (FP3) from the outlet of the oily phase.

Trial	Pomace moisture (%)	Pomace oil content (% d.m.)	Oil extraction efficiency (%)
<i>I trial</i>			
FP1	62.11±0.89 a	8.89±0.25 a	75.32±0.40 c
FP2	62.78±0.65 a	8.58±0.10 ab	77.71±0.21 b
FP3	62.14±1.55 a	8.17±0.32 b	83.24±1.36 a
<i>II trial</i>			
FP1	63.42±1.16 a	8.42±0.47 a	81.60±1.26 b
FP2	65.02±3.03 a	8.62±0.57 a	82.71±1.48 ab
FP3	63.56±1.35 a	7.60±0.12 b	84.32±0.55 a

Different letters on the same row for the same trial indicate significant differences ($p \leq 0.05$) according to one way ANOVA followed by Tukey's HSD test.

Table 2. Mean values, standard deviations and results of the one way ANOVA followed by Tukey's HSD test of the chemical and sensory analyses performed on the oils ($n=3$) obtained in two different olive processing trials. First trial (I) corresponded to olives having maturity index lower than those of second trial (II). Three different decanter feed pipe (FP) positions were considered, at 825 mm (FP1), 610 mm (FP2), and 520 mm (FP3) from the outlet of the oily phase.

Trial Sample	I			II		
	FP1	FP2	FP3	FP1	FP2	FP3
FFA (g 100g ⁻¹ oleic acid)	0.37±0.00 a	0.37±0.00 a	0.37±0.00 a	0.37±0.00 a	0.37±0.00 a	0.37±0.00 a
PV (mEq O ₂ kg ⁻¹)	8.0±0.8 a	7.7±1.0 a	7.8±0.5 a	6.7±0.9 a	6.4±0.7 a	6.6±1.1 a
K ₂₃₂	1.70±0.02 a	1.71±0.04 a	1.69±0.04 a	1.67±0.03 a	1.66±0.03 a	1.73±0.12 a
K ₂₇₀	0.17±0.03 a	0.17±0.05 a	0.18±0.04 a	0.19±0.02 a	0.20±0.03 a	0.22±0.08 a
TPC (mg kg ⁻¹)	356±28 a	387±41 a	314±26 a	480±23 a	460±27 a	433±7 a
Chlorophylls (mg kg ⁻¹)	15.72±0.81 a	16.04±0.03 a	16.41±0.14 a	15.56±1.45 a	13.30±0.24 ab	13.09±0.76 b
Carotenoids (mg kg ⁻¹)	12.34±1.33 b	14.22±1.68 ab	16.50±0.57 a	12.88±2.32 b	18.54±2.43 a	18.03±0.88 a
β+γ-Tocopherols (mg kg ⁻¹)	3.71±0.40 a	4.41±0.39 a	4.37±0.15 a	5.28±0.03 a	5.75±0.23 a	5.24±0.51 a
α-Tocopherol (mg kg ⁻¹)	159.37±1.37 b	163.93±1.58 a	162.07±1.79 ab	170.16±0.99 ab	172.27±0.07 a	169.25±1.14 b
Tocopherols (mg kg ⁻¹)	163.08±1.75 b	168.35±1.95 a	166.44±1.91 ab	175.44±0.96 b	178.02±0.23 a	174.49±1.15 b
Fruity*	3.5±0.7 a	5.0±0.7 a	4.3±0.5 a	3.6±0.5 a	3.8±0.6 a	3.5±0.7 a
Bitter*	2.2±0.3 a	2.6±0.7 a	1.7±0.5 a	2.5±0.2 a	2.5±0.4 a	2.1±0.2 a
Pungent*	2.3±0.1 a	3.2±0.6 a	2.2±0.6 a	3.3±0.3 a	2.4±0.1 a	2.8±0.4 a

FFA, free fatty acids; PV, peroxide value; K₂₃₂, specific absorption at 232 nm; K₂₇₀, specific absorption at 270 nm; TPC, total phenolic content.

*Median ± robust standard deviation (Regulation ECC No 2568/91). Statistical significance was determined by Kruskal-Wallis test ($p \leq 0.05$).

Different letters on the same row for the same trial indicate significant differences ($p \leq 0.05$).

Table 3. HPLC phenolic profile (mg kg⁻¹ ± sd) of the oils obtained in two different olive processing trials (*n*=3). First trial (I) corresponded to olives having maturity index lower than those of second trial (II). Three different decanter feed pipe (FP) positions were considered, at 825 mm (FP1), 610 mm (FP2), and 520 mm (FP3) from the outlet of the oily phase.

Trial Sample	I			II		
	FP1	FP2	FP3	FP1	FP2	FP3
Hydroxytyrosol	0.28±0.02 a	0.28±0.03 a	0.25±0.05 a	0.54±0.10 a	0.58±0.10 a	0.56±0.04 a
Tyrosol	0.47±0.02 a	0.46±0.04 a	0.47±0.01 a	0.66±0.09 a	0.75±0.05 a	0.76±0.02 a
Vanillic acid	0.20±0.06 a	0.19±0.04 a	0.22±0.05 a	0.22±0.01 ab	0.20±0.02 b	0.25±0.01 a
Syringic acid	0.35±0.04 a	0.37±0.05 a	0.37±0.03 a	0.32±0.07 a	0.32±0.09 a	0.33±0.08 a
3,4-DHPEA-EDA	28.26±0.68 a	28.13±1.33 a	21.86±0.74 b	28.47±4.47 a	24.89±4.71 a	23.27±1.31 a
Oleuropein	1.43±0.27 a	1.34±0.20 a	1.03±0.05 a	2.73±0.29 a	2.52±0.87 a	2.18±0.15 a
<i>p</i> -HPEA-EDA	25.35±0.26 ab	25.70±0.85 a	23.89±0.70 b	25.49±1.15 a	26.51±0.27 a	26.31±0.48 a
(+)-Pinoresinol	10.36±0.23 a	10.90±0.22 a	10.05±0.54 a	12.26±0.54 a	12.44±0.15 a	12.09±0.20 a
1-Acetoxy-pinoresinol	0.86±0.03 a	1.02±0.05 a	1.00±0.22 a	2.38±0.16 a	2.61±0.19 a	2.28±0.11 a
Cinnamic acid	0.43±0.24 b	0.91±0.10 a	1.14±0.21 a	1.94±0.43 a	2.32±0.36 a	2.42±0.13 a
3,4-DHPEA-EA	4.75±0.25 a	4.78±0.10 a	4.43±0.20 a	4.43±0.41 a	4.98±0.16 a	4.60±0.16 a
Luteolin	3.65±0.46 a	3.95±0.43 a	3.24±0.26 a	4.14±0.37 a	4.38±0.60 a	4.50±0.36 a
<i>p</i> -HPEA-EA	3.54±0.17 a	3.70±0.26 a	3.65±0.18 a	3.30±0.06 a	3.55±0.31 a	3.31±0.17 a
Apigenin	6.07±0.06 a	6.41±0.35 a	6.02±0.17 a	4.13±0.05 b	4.36±0.03 a	4.19±0.05 b
Total	86.00±0.18 a	88.15±3.49 a	77.61±2.10 b	91.01±7.89 a	90.39±6.14 a	87.05±2.43 a

3,4-DHPEA-EDA, dialdehydic form of elenolic acid linked to hydroxytyrosol; *p*-HPEA-EDA, dialdehydic form of elenolic acid linked to tyrosol; 3,4-DHPEA-EA, oleuropein aglycon; *p*-HPEA-EA, ligstroside aglycon.

Different letters on the same row for the same trial indicate significant differences ($p \leq 0.05$) according to one way ANOVA followed by Tukey's HSD test.

Table 4. Profile of volatile compounds (mg kg⁻¹ ± sd) of the oils obtained in two different olive processing trials (*n*=3). First trial (I) corresponded to olives having maturity index lower than those of second trial (II). Three different decanter feed pipe (FP) positions were considered, at 825 mm (FP1), 610 mm (FP2), and 520 mm (FP3) from the outlet of the oily phase.

	Trial Sample	I			II		
		FP1	FP2	FP3	FP1	FP2	FP3
Aldehydes	Hexanal	8.34±0.93 a	7.88±0.91 a	7.67±0.64 a	7.57±1.48 a	8.10±0.99 a	10.21±0.48 a
	<i>cis</i> -3-Hexenal	5.30±0.55 a	4.81±0.81 a	5.07±1.24 a	4.78±1.50 a	6.58±0.68 a	5.41±0.16 a
	<i>trans</i> -2-Hexenal	232.90±25.53 a	222.80±32.64 a	225.15±21.93 a	177.08±25.01 a	195.70±17.34 a	217.63±5.37 a
	3-Metilbutanal	0.28±0.06 a	0.35±0.05 a	0.24±0.03 a	0.03±0.03 b	0.12±0.01 a	0.09±0.00 a
	<i>trans</i> -2-Pental	1.33±0.13 a	1.17±0.18 a	1.20±0.29 a	1.20±0.29 a	1.38±0.16 a	1.63±0.20 a
	Nonanal	0.84±0.12 a	0.94±0.15 a	0.62±0.20 a	0.28±0.10 a	0.25±0.04 a	0.28±0.10 a
	<i>trans,trans</i> -2,4-Hexadienal	2.70±0.46 b	4.92±0.93 a	4.34±0.40 a	3.39±1.47 a	4.82±0.45 a	2.94±0.31 a
	2-Metil-4-pental	6.80±0.78 a	6.23±0.81 a	0.65±0.09 b	5.98±1.70 a	6.60±1.05 a	7.07±0.36 a
Alcohols	Ethanol	1.68±0.60 a	1.61±0.24 a	1.01±0.21 a	1.31±0.48 b	1.79±0.47 ab	2.97±0.58 a
	<i>trans</i> -2-Hexen-1-ol	1.66±0.29 a	1.65±0.25 a	2.02±0.32 a	0.84±0.41 b	2.40±0.94 a	3.41±0.16 a
	1-Hexanol	1.85±0.32 a	1.05±0.18 b	1.37±0.16 ab	2.12±0.51 a	1.77±0.71 a	1.65±0.68 a
Esters	<i>trans</i> -3-Hexen-1-ol acetate	4.60±0.50 a	4.98±0.84 a	4.31±1.26 a	4.21±0.37 ab	3.56±0.52 b	5.01±0.68 a
	Ethyl acetate	0.68±0.04 b	1.11±0.16 a	0.67±0.16 b	0.45±0.06 b	0.71±0.03 a	0.72±0.08 a
	<i>cis</i> -3-Hexenil acetate	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00 a	0.13±0.23 a	0.00±0.00 a
	Metil acetate	0.06±0.11 a	0.19±0.17 a	0.20±0.06 a	0.18±0.07 a	0.20±0.08 a	0.24±0.01 a
Ketones	1-Penten-3-one	9.99±0.61 b	12.03±0.82 a	7.24±0.56 c	8.88±2.83 a	9.96±1.80 a	12.57±0.99 a
	3-Pentanone	1.34±0.14 a	1.48±0.26 a	1.14±0.22 a	1.01±0.44 a	1.26±0.23 a	1.50±0.17 a
Acids	Acetic acid	1.04±0.57 a	0.73±0.07 a	1.95±1.94 a	0.00±0.00 a	0.00±0.00 a	0.00±0.00 a
Others	Octane	0.82±0.14 a	1.08±0.19 a	1.14±0.53 a	0.56±0.09 a	0.62±0.15 a	0.59±0.07 a
	Limonene	0.53±0.03 a	0.56±0.05 a	0.40±0.06 b	0.56±0.16 a	0.69±0.11 a	0.80±0.13 a
Total		282.75±28.97 a	275.57±38.81 a	266.38±24.49 a	220.43±25.73 b	246.63±21.12 ab	274.72±7.77 a

Different letters on the same row for the same trial indicate significant differences ($p \leq 0.05$) according to one way ANOVA followed by Tukey's HSD test.