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Research Article

Centrifugation, Storage, and Filtration of Olive Oil in an Oil Mill: Effect on the Quality and Content of Minority Compounds

Alfonso M. Vidal, Sonia Alcalá, Antonia de Torres, Manuel Moya, and Francisco Espínola

Centre for Advanced Studies in Energy and Environment (CEAEMA), Agrifood Campus of International Excellence (ceiA3), Dept. Chemical, Environmental and Materials Engineering, University of Jaén, Paraje Las Lagunillas, Edif. B-3, 23071 Jaén, Spain

Correspondence should be addressed to Alfonso M. Vidal; amvidal@ujaen.es

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Centrifugation, storage, and filtration of olive oil were evaluated in an oil mill to determine their effect on the final quality of virgin olive oil. The main functions of these processes are to clarify the olive oil by removing water, solids, and other possible suspended particles. Although some changes were detected in the oil quality parameters after these processes, all the samples were extra virgin olive oil. The phenolic and volatile compound content of the olive oil was influenced by vertical centrifugation processing. Significantly, vertical centrifugation led to a 53% reduction in ethanol content. Oil storage before filtration resulted in a significant increase of around 30% in the peroxide index, while the antioxidant capacity decreased by 78%. Comparison of the results for filtered and unfiltered oil samples revealed that the most significant change was the reduction in the photosynthetic pigment content, with a decrease of around 50% in chlorophyll. Due of all this, the conditions applied in vertical centrifugation and the time of storage of the olive oils should be further controlled, enabling cleaning and decantation but avoiding the reduction of the antioxidant capacity and the content of phenolics compounds.

1. Introduction

Virgin olive oil (VOO) is a fat known worldwide for its beneficial properties for human health. The consumption of olive oil in the Mediterranean diet is associated with low mortality from cardiovascular disease [1]. Several health benefits have been associated with certain antioxidant compounds such as phenols [2]. The health claims on "olive oil polyphenols" by the EEC [3] refer to the impact of bioactive phenolic compounds on the protection of blood lipids against oxidative stress [4]. High nutritional quality arises from large amounts of unsaturated fatty acids in the composition of oil, such as oleic acid and linolenic acid. The production of VOO is solely carried out by physical and mechanical extraction processes. Oil washing is a step of the process, which is performed in a vertical centrifuge (VC). After obtaining the oil, it is filtered to eliminate any solids in the suspension.

Washing represents an important source of oxidative reactions arising from the contact between water and oil [5]. The distribution of phenolic compounds in the water and oil phases depends on their solubility in the phases [6]; phenolic compounds may thus be found in the wastewater and pomace. Vertical centrifugation has a great effectiveness in clarifying the oil, although this process reduces the concentration of minor compounds in the extra virgin olive oil (EVOO) [7]. The maximum oxygenation levels have been detected after VC treatment. The oxidation of olive oil during its shelf-life is negatively affected by the concentration of dissolved oxygen [8].

Inert gases have been used for oil oxygenation prevention and found to significantly extend the oil shelf-life [9]. Other researchers have focused on the effect of the water employed in the VC and on the content of alkyl esters in olive oils [10], where the content of ethyl and methyl esters were found to decrease with the use of water in the VC.

According to Gila et al. [11], minimal water addition to the VC is the optimum option to improve the oil quality.

The content of certain compounds such as hydroxytyrosol, tyrosol, and the dialdehydic form of elenolic acid linked to hydroxytyrosol, underwent the most significant changes [12]. Other authors such as Masella et al. [13] have described slight variations in the concentrations of phenolic compounds while comparing the composition of olive oil before and after the centrifugation process. Generally, a decrease in the content of these compounds is observed [14], that is, by diffusion from the oil phase to the aqueous phase. Moreover, the temperature of the added water was also found to influence the extraction process [15]. Comparative trials have also been performed on oil samples filtered using a conventional filtration method instead of a VC [16].

The turbidity of oil is caused by particles from plant tissue in suspension and water droplets. Such solids, particles, and water can deteriorate the quality by promoting the oxidation and hydrolysis of olive oil [17]. The aim of filtration is eliminate these to increase oil shelf-life. Several changes in the oil composition can occur during filtration, such as changes in the phenol and volatile compound content or the color of the oil [18, 19]. Natural sedimentation is more favorable than filtration in delaying the oxidative deterioration of oil; nevertheless, filtration provides a more stable sensory profile than do sedimentation and decantation [20].

Regarding filtration, a laboratory-scale study has shown that similar amounts of phenolic compounds are present in filtered and unfiltered EVOO [21]. However, another study, this time at pilot plant scale using filtration systems with inert gas flow (argon and nitrogen) and polypropylene filter bags, showed that the content of most phenolic compounds seemed to increase after filtration [22]. Quantitative and qualitative changes, especially on minor components were detected, which affected the EVOO quality [17]. The volatile compound and sensory characteristics of EVOO can be influenced by oil filtration [23, 24].

The objective of this work was to determine the influence of oil centrifugation, storage, and subsequent filtration on the regulated quality parameters and the phenolic and volatile compound contents of olive oil produced in a mill.

2. Materials and Methods

2.1. Raw Material. Olive fruits (*Olea europaea* L.) cv. Picual were harvested from irrigated land during the 2016–2017 crop season in Mancha Real (Jaén, Spain) and processed after the harvest at a local olive oil mill. A lot of approximately 5000 kg of olives was used for the experimental trials. The maturity index (MI), or ripening degree, was obtained following the method described by Espínola et al. [25]. The Soxhlet method is used to analyse the oil content.

2.2. Olive Oil Mill. The oil mill where the centrifugation, storage and filtration trials were carried out is located in the "Cortijo Virgen de loss Milagros," Mancha Real (Spain), and has a plant for the extraction of EVOO. The experiments were performed with the mill working continuously. The VC (Pieralisi, Jesi, Italy) was operated at 6400 rpm. The optimum water addition content was determined by the millworkers to be 5%. Samples of oil, pomace, and paste were collected in triplicate at different times, at approximately 20 min intervals throughout the experiment. The extracted oil was stored in a stainless-steel tank for 25 days. Then, the oil was filtered through a layer of hydrophilic cellulose acetate. The filtration was carried out continuously with an industrial filter and three oil samples were collected, at both the filter inlet and outlet. All oil samples were stored in amber glass bottles, filled with nitrogen, and kept at −18°C until further analysis. The samples for the sensory analysis were sent to an external laboratory.

2.3. Analysis of Olive Oil Quality Parameters. The free acidity, peroxide index, and extinction coefficients K_{232} and K_{270} were determined according to the European Union standard method [26].

2.4. Analysis of Photosynthetic Pigments. The photosynthetic pigments composition was determined according to the method of Mínguez-Mosquera et al. [27]. The spectrophotometer used was a Shimadzu (model UV-1800). The carotenoids and the chlorophylls were measured at a wavelength of 470 nm and 670 nm, respectively. The pigment concentration of the olive oils was expressed as mg of pigment per kg of oil.

2.5. Analysis of Volatile Compounds. The volatile compounds were quantified by following the method previously described by Vidal et al. [28]. They were analyzed by headspace solid-phase microextraction (HS-SPME) and gas chromatography-flame ionization detection (GC-FID). The SPME fiber is formed of Carboxen/DVB/polydimethylsiloxane and had 2 cm length and 50/30 μm of film thickness. It was acquired from Supelco (Bellefonte, PA, USA). The fiber had been previously conditioned following the instructions of the manufacturer.

GC-FID analysis was carried out on a gas chromatograph, model 7890B (*Agilent Technologies*, CA, USA). The capillary column used to the separation was a DB-WAXetr (*Agilent Technologies*, USA), (30 m of length, 0.25 of mm internal diameter, and 0.25 of μm coating) formed by polyethylene glycol. The chromatographic peaks were quantified by the "Internal Standard" method. This method uses internal and external standards. A calibration curve was made with the relationship between the external and internal standard (4-methyl-2-pentanol). The purpose was to improve the quantification. The results are expressed as mg of compound per kg of olive oil.

2.6. Analysis of Phenolic Compounds. The phenolic compounds present in the VOO were determined according to the method of International Olive Council [29]. A liquid chromatograph (*Shimadzu Corp*., Kyoto, Japan) was used. The column C18 BDS Hypersil (*Thermo Scientific*, USA) was

employed in the chromatographic separation and its characteristics were 25 cm length, 5 μm of particle size, and 4.6 mm of internal diameter. The quantification was carried out through the addition of syringic acid and tyrosol, as internal and external standard, respectively. The analytical standards were used to identify the phenol compounds. The results are showed as mg of tyrosol per kg of oil.

2.7. Determination of the Antioxidant Potential. The antioxidant potential was determined according to the method described by Vidal et al. [28]. The free radical 2,2-diphenyl-1-picrylhydrazyl (DPPH) was used to determine the antioxidant potential. The absorbance was measured at 515 nm of the sample and the DPPH solution. Methanol was used as solvent and as the control. The absorbance obtained was converted into the DPPH concentration by interpolation of the calibration curve of absorbance versus DPPH concentration. The percentage of inhibition of DPPH radical was calculated according to equation (1):

$$\mathrm{DPPH}_{\mathrm{inhibition}}\ (\%) = \left([\mathrm{DPPH}]_0 - \frac{[\mathrm{DPPH}]_{\mathrm{sample}}}{[\mathrm{DPPH}]_0} \right) \times 100, \quad (1)$$

where $[\mathrm{DPPH}]_0$ and $[\mathrm{DPPH}]_{\mathrm{sample}}$ are the concentration of the control and sample, respectively. The percentage of inhibition DPPH was converted into the Trolox concentration using a calibration curve of the percentage of inhibition versus the Trolox concentration. The antioxidant capacity is expressed as μmol Trolox per kg of olive.

2.8. Sensory Analysis. A panel formed by highly experienced people carried out the quantitative descriptive sensory analysis of the EVOO. The method proposed by the International Olive Council described in the EEC, Annex XII, [26] was used. The determination was carried out in the Agri-food Laboratory of Granada (Granada, Spain). The positive attributes: fruity, bitter, and pungent, and the possible presence of defects were determined.

2.9. Statistical Analysis. The results were processed with the StatGraphics Centurion software, version 17.2.00, (*Statpoint Technologies*, Inc., Warrenton, VA, USA). The mean values of the repeats and the Fisher significant least differences (Fisher's LSD) for each response analyzed was obtained.

3. Results and Discussion

Olives were characterized by a MI of 1.74 and a content of oil of 20.69%. This means that the skin of the olives had a green color with above less than 50% of purple. The percentage of oil content is acceptable to extract considerable oil content, for the early maturation stage at which the sample is found. The effects of vertical centrifugation, storage and filtration were evaluated at industrial scale to obtain realistic results and thus be able to select the best parameters to produce EVOO. Washing the oil in a VC resulted in some changes in the quality parameters and composition. Likewise, some differences were observed between unfiltered and filtered samples.

3.1. Effect of Centrifugation on the EVOO Characteristics. The effect of oil centrifugation or washing was evaluated. For this purpose, the oil exiting the decanter and VC was analyzed. The results are provided in several tables: quality parameters and sensory characteristics (Table 1), volatile compounds (Table 2), and phenolic compounds (Table 3). An analysis of the quality parameters was also conducted. The acidity was reduced by 20.2% after washing and the peroxide index increased by 9.9%. The K_{232} value experienced a slight drop of 4.2% after washing, while K_{270} was reduced by 7.9%. The photosynthetic pigment (chlorophylls and carotenoids) content showed only a slight decrease after vertical centrifugation of the olive oil. These results are similar to those found in the literature [7]. According to the quality parameters, the olive oil category of every sample remained EVOO, as per the limits of the EEC [30]. The olive oil category did not change after the washing process, even though the quality parameters suffered some changes. Few variations in the sensory characteristics were observed, with just a slight decrease in the bitterness and pungency after oil centrifugation.

The results from the volatile compound analysis are presented in Table 2, and are represented in Figure S1(A). After washing, the total content of volatile compounds from the lipoxygenase (LOX) pathway experienced a reduction of 9.0%. These results are consistent with those by Masella et al. [13]. All volatile compounds from the LOX pathway as well as from other analyzed compounds exhibited a reduction in their content after washing. This reduction is due to partition phenomena between the oil and water phases [13]. Of note is the significant decrease of 53.3% in the ethanol content, which is produced by fermentation. According to Alcalá et al. [10], the use of water in the VC reduces the ethyl and methyl ester content, probably because some of the alcohol in the olive oil is extracted into water.

The phenolic compound content results are presented in Table 3, and are represented in Figure S1(B). The total content of phenolic compounds, mostly belonging to the group of secoiridoids, decreased by 22.9% after washing the oil. Furthermore, the antioxidant capacity decreased by 27.0% during the oil washing process. From an individual analysis of phenolic compounds, hydroxytyrosol, cinnamic acid and lignans did not undergo significant variations during centrifugation. In contrast, tyrosol, ferulic acid, *p*-coumaric acid, vanillin, secoiridoids and flavones had a decrease significant in their content during centrifugation. This reduction may be due to the transfer of the hydrophilic phenols of the oil to the water, and also to the increase of oxygen dissolved in the olive oil during centrifugation, which can cause oxidation reactions on the phenolic compounds [6, 13]. Therefore, the observed decrease in the total content of this type of compounds in EVOO is due to the individual reduction in the amount of each compound.

Table 1: Quality parameters and sensory characteristics for the oil samples before and after vertical centrifugation, storage, and filtration*.

	Decanter exit	Centrifuge exit/beginning of storage	Unfiltered/end of storage	Filtered
Acidity (%)	0.186 ± 0.001^{a}	0.148 ± 0.001^{b}	0.122 ± 0.004^{c}	0.111 ± 0.002^{d}
Peroxide I. (mEq·O_2/kg)	3.07 ± 0.07^{d}	3.38 ± 0.13^{c}	4.45 ± 0.03^{b}	5.00 ± 0.04^{a}
K_{232}	1.33 ± 0.08^{a}	1.27 ± 0.19^{a}	1.46 ± 0.03^{a}	1.35 ± 0.04^{a}
K_{270}	0.13 ± 0.01^{a}	$0.12 \pm 0.02^{a,b}$	$0.106 \pm 0.002^{b,c}$	0.096 ± 0.006^{c}
Chlorophylls (mg/kg)	35.18 ± 1.47^{a}	32.51 ± 0.90^{a}	26.41 ± 0.73^{b}	13.78 ± 0.18^{c}
Carotenoids (mg/kg)	14.17 ± 0.62^{a}	$13.08 \pm 1.06^{a,b}$	12.24 ± 0.32^{b}	7.73 ± 0.05^{c}
Total HPLC phenols (mg/kg)	438.37 ± 3.23^{a}	338.14 ± 3.99^{b}	224.75 ± 5.47^{c}	221.40 ± 3.49^{c}
DPPH (μmol/kg)	1359.83 ± 19.54^{a}	992.07 ± 8.52^{b}	221.09 ± 21.56^{c}	213.71 ± 11.84^{c}
Total LOX volatiles (mg/kg)	9.82 ± 0.26^{b}	8.93 ± 0.43^{b}	11.73 ± 0.88^{a}	11.02 ± 0.37^{a}
Fruitiness	6.4 ± 0.6^{a}	$6.0 \pm 0.3^{a,b}$	5.8 ± 0.1^{b}	5.6 ± 0.9^{b}
Bitterness	$3.5 \pm 0.3^{a,b}$	3.6 ± 0.3^{a}	$3.2 \pm 0.3^{b,c}$	2.9 ± 0.2^{c}
Pungency	4.1 ± 0.1^{a}	4.3 ± 0.2^{a}	$3.9 \pm 0.1^{a,b}$	3.6 ± 0.4^{b}

*Values are expressed as mean ± SD; (a, b, c, d) indicate Fisher's least significant differences (LSD), with statistically significant differences at 95% confidence level.

Table 2: Individual content of volatile compounds before and after vertical centrifugation, storage, and filtration processes, expressed in mg/kg*

	Decanter exit	Centrifuge exit/beginning of storage	Unfiltered/end of storage	Filtered
LOX pathway				
Hexanal	0.42 ± 0.01^{b}	0.40 ± 0.01^{b}	0.56 ± 0.02^{a}	0.55 ± 0.02^{a}
Hexan-1-ol	0.38 ± 0.02^{b}	0.36 ± 0.02^{b}	0.64 ± 0.06^{a}	0.61 ± 0.02^{a}
(*E*)-2-hexenal	3.25 ± 0.03^{a}	2.90 ± 0.11^{b}	3.27 ± 0.22^{a}	$3.13 \pm 0.05^{a,b}$
(*E*)-2-hexen-1-ol	0.24 ± 0.01^{b}	0.23 ± 0.02^{b}	0.62 ± 0.01^{a}	0.65 ± 0.02^{a}
(*Z*)-3-hexen-1-ol	1.80 ± 0.04^{b}	1.61 ± 0.11^{b}	2.33 ± 0.21^{a}	2.16 ± 0.06^{a}
(*Z*)-3-hexenyl acetate	2.33 ± 0.16^{b}	2.25 ± 0.10^{b}	3.07 ± 0.35^{a}	2.44 ± 0.08^{b}
1-penten-3-ol	$0.28 \pm 0.00^{a,b}$	$0.23 \pm 0.03^{b,c}$	0.21 ± 0.004^{c}	0.32 ± 0.07^{a}
1-penten-3-one	0.65 ± 0.02^{c}	0.56 ± 0.04^{b}	0.62 ± 0.01^{b}	0.71 ± 0.02^{a}
(*Z*)-2-penten-1-ol	0.45 ± 0.01^{a}	0.39 ± 0.04^{b}	$0.41 \pm 001^{a,b}$	0.46 ± 0.02^{a}
Sugar fermentation				
Ethanol	8.31 ± 0.17^{a}	3.88 ± 0.09^{c}	4.70 ± 0.07^{c}	6.27 ± 0.18^{b}
Acetic acid	0.64 ± 0.09^{b}	0.53 ± 0.07^{c}	0.77 ± 0.04^{a}	$0.55 \pm 0.01^{b,c}$
Other compounds				
(*E*)-2-pentenal	$0.28 \pm 0.01^{b,c}$	0.25 ± 0.03^{c}	0.29 ± 0.004^{b}	0.34 ± 0.02^{a}
Pentan-3-one	0.34 ± 0.01^{b}	0.31 ± 0.02^{c}	0.35 ± 0.01^{b}	0.41 ± 0.01^{a}
Nonanal	1.96 ± 0.05^{c}	1.75 ± 0.12^{d}	2.39 ± 0.05^{b}	2.63 ± 0.12^{a}

*Values are expressed as mean ± SD; (a, b, c, d) indicate Fisher's least significant differences (LSD), with statistically significant differences at 95% confidence level.

3.2. Effect of Storage of EVOO before Filtration. Oil storage was performed in a stainless-steel tank for 25 days at room temperature. A comparison was made between the oil samples obtained on the day of oil elaboration and the samples collected on the day of filtration to determine any changes in the composition, which will in turn have an effect on the quality parameters. The results are those compared between the columns called "centrifuge exit/beginning of storage" and "unfiltered/end of storage" from Tables 1–3. The most significant changes were the increase in the peroxide index by 31.66% and an increase of 14.96% for K_{232}, similar to the results reported by Rodrigues et al. [31]. In contrast, the other parameters decreased after those 25 days. The most significant changes were observed for the antioxidant capacity with a drop of 77.71%, and a decrease of 54.93% for the chlorophyll content and of 48.05% for the carotenoid content. These results are similar to those reported in the literature by Gutiérrez and Fernández [32]. The phenolic compound content decreased by 33.53%, similar to the data reported by Gutiérrez and Fernández [32] and Kotsiou and Tasioula-Margari [33]. A decrease in the content of most phenolic compounds was also observed, which could explain at least in part the loss of antioxidant capacity. This may be due to the loss of hydroxytyrosol, and the decrease of secoiridoid compounds, since they are compounds with a high antioxidant capacity. Making an individual analysis of the phenolic compounds during storage, it is worth highlighting the total disappearance of hydroxytyrosol. Furthermore, the secoiridoid compounds experiment a great decrease except *p*-HPEA-EA. On the contrary, tyrosol, flavones and cinnamic acid have a slight increase in their content.

In the sensory analysis, only a slight decrease was observed, similar to Gutiérrez results [32].

3.3. Effect of Filtration on the EVOO Characteristics. The effect of filtration on the characteristics of the olive oil samples was evaluated. For this purpose, the characteristics of filtered and unfiltered samples were compared. The quality parameters, sensory data, and phenolic and volatile compound content in the filtered and unfiltered oil samples are

Table 3: Individual content of phenolic compounds before and after vertical centrifugation, storage, and filtration, expressed in mg/kg*

	Decanter exit	Centrifuge exit/beginning of storage	Unfiltered/end of storage	Filtered
Phenolic alcohols				
Hydroxytyrosol	6.24 ± 0.10^{a}	6.15 ± 0.10^{a}	–	–
Tyrosol	3.87 ± 0.07^{a}	2.34 ± 0.15^{c}	2.85 ± 0.02^{b}	2.88 ± 0.03^{b}
Phenolic acids				
p-coumaric acid	3.04 ± 0.15^{a}	1.69 ± 0.09^{c}	2.74 ± 0.01^{b}	1.42 ± 0.05^{d}
Ferulic acid	7.36 ± 0.16^{a}	5.32 ± 0.13^{b}	0.77 ± 0.03^{c}	0.87 ± 0.01^{c}
Cinnamic acid	1.70 ± 0.08^{a}	1.64 ± 0.05^{a}	1.04 ± 0.04^{b}	0.88 ± 0.29^{b}
Secoiridoids				
3,4-DHPEA-EDA (oleacein)	141.57 ± 3.68^{a}	94.63 ± 0.32^{b}	28.93 ± 0.37^{c}	29.95 ± 0.92^{c}
3,4-DHPEA-EA	108.14 ± 2.11^{a}	85.27 ± 2.18^{b}	27.99 ± 0.29^{c}	28.98 ± 0.56^{c}
p-HPEA-EDA (oleocanthal)	75.18 ± 2.98^{a}	62.30 ± 2.90^{b}	32.72 ± 0.32^{c}	32.74 ± 0.24^{c}
p-HPEA-EA	21.98 ± 2.38^{a}	17.80 ± 1.49^{b}	18.45 ± 0.17^{b}	18.61 ± 0.37^{b}
Lignans				
Pinoresinol + acetoxypinoresinol	14.56 ± 1.37^{a}	14.05 ± 1.65^{a}	11.60 ± 0.84^{b}	11.45 ± 0.46^{b}
Flavones				
Luteolin	7.67 ± 0.29^{b}	$7.50 \pm 0.48^{b,c}$	9.75 ± 0.56^{a}	6.76 ± 0.50^{c}
Apigenin	5.24 ± 0.48^{b}	4.49 ± 0.31^{c}	7.37 ± 0.13^{a}	6.99 ± 0.23^{a}
Others				
Vainillin	1.85 ± 0.03^{a}	1.67 ± 0.07^{b}	1.52 ± 0.04^{c}	1.56 ± 0.09^{c}

*Values are expressed as mean ± SD; (a, b, c, d) indicate the Fisher's least significant differences (LSD), with statistically significant differences at 95% confidence level.

presented in Tables 1–3. After oil filtration, slight but significant differences were observed. The acidity and K_{232} value decreased slightly and the peroxide index increased by 12.2%. In contrast, the photosynthetic pigment content was reduced during the filtration process. The chlorophyll concentration decreased by 47.8% in relation to the unfiltered oil, and the carotenoid concentration decreased by 36.8%. This means that the cellulose acetate filter collects a very important fraction of photosynthetic pigments. These results are consistent with those reported by Gordillo et al. [34] and Brkic Bubola et al. [24]. According to the quality parameters determined, the olive oil category was still EVOO for all the samples as per the EEC [30]. Although the quality parameters underwent some changes, the category of the olive oil was not changed by the filtration process. The antioxidant capacity was also similar in both cases.

The results of the volatile compounds are presented in Table 2, and are represented in Figure S2(A). The volatile compounds were analyzed separately to detect any differences between the unfiltered and filtered samples. Overall, no major differences were observed between the two samples, except for some compounds. (*E*)-2-Hexenal, (*Z*)-3-hexenol, and (*Z*)-3-hexenyl acetate were found in greater proportion in the unfiltered sample; in contrast, (*Z*)-2-pentenol, 3-pentanone, and (*E*)-2-pentenal were detected in smaller proportion in the unfiltered sample. The amount of six-carbon-atom volatile compounds decreased after filtration; however, the amount of five-carbon-atom volatile compounds increased after the filtration process. Although the observed differences are minor, they still reveal a slight trend. These results are similar to those previously reported in the literature by Bottino et al. [23] and Brkic Bubola et al. [24].

The results obtained from the analysis of phenolic compounds are shown in Table 3, and are represented in Figure S2(B). The total amount of phenolic compounds was similar in both filtered and unfiltered samples. Certain similarities exist in both samples, except for some particular compounds. Larger amounts of luteolin and *p*-coumaric acid were detected in the unfiltered sample, results similar to those obtained by Bakhouche et al. [19], that finds a reduction of phenolic alcohols and flavones. On the other hand, oleacin and 3, 4-DHPEA-EA were found in smaller proportion in the unfiltered sample, although they are not significant differences. According to Gómez-Caravaca et al. [35], the content of phenolic compounds slightly increases after the filtration process, which may be due to the removal of water from the oil, thus increasing the concentration of dissolved substances in the oil.

All other phenolic compounds presented no differences in the filtered and unfiltered samples. It should be noted that there are some investigations changing the filtering conditions, such as that of Lozano-Sánchez et al. [22], and find some differences in the oils. Also, the type of filter used in the filtration process can be affected the content of phenolic compounds, according to results obtained by Bakhouche et al. [19] and Gómez-Caravaca et al. [35].

4. Conclusion

The use of centrifugation, storage (in order to decant) and filtration in an industrial olive mill have the function of to clean and to clarify olive oils. The olive oil category was not changed after the centrifugation, storage and filtration processes with slight changes in the fruitiness, bitterness and pungency. However, centrifugation, storage and filtration produced some significant changes found in the quality parameters and minor composition.

A relevant result was how the content of phenolic compounds was affected by centrifugation. A reduction in the concentration of these compounds was observed after the vertical centrifugation process. This is probably the result

of the transfer of hydrophilic phenols from the oil to the water phase. Centrifugation led to a 22.9% reduction in the total content of phenolic compounds. Similarly, the content of volatile compounds from the LOX pathway exhibited a decrease after washing, although the loss was just of 9%. It should be noted that a significant decrease of 53.3% of the ethanol compound content was observed after vertical centrifugation of the olive oil.

The most relevant results from the oil samples stored for 25 days before filtration were a significant increase in the peroxide index (around 30%) and a 78% decrease in the antioxidant capacity. A small number of differences were detected after oil filtration, with no differences in the sensory characteristics. The total amount of phenolic compounds and volatile compounds from the LOX pathway was similar in both filtered and unfiltered samples; furthermore, the antioxidant capacity exhibited a similar trend to the phenolic compound content. On the contrary, the photosynthetic pigment content decreased after the filtration process.

From these results, it is concluded that the water addition in the vertical centrifugation and the time of storage of olive oils should be reduced in order to avoid the decrease of the antioxidant capacity and phenolics compounds.

Abbreviations

3, 4-DHPEA-EA:	aldehyde and hydroxylic forms of oleuropein aglycone
3, 4-DHPEA-EDA (oleacein):	dialdehyde form of decarboxymethyl oleuropein aglycone
DPPH:	2, 2-diphenyl-1-picrylhydrazyl
EVOO:	extra virgin olive oil
LSD:	least significant difference
MI:	maturity index
MUFA:	monounsaturated fatty acid
p-HPEA-EA:	aldehyde and hydroxylic forms of ligstroside aglycone
p-HPEA-EDA (oleocanthal):	dialdehyde form of decarboxymethyl ligstroside aglycone
VC:	vertical centrifuge
VOO:	virgin olive oil.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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Supplementary Materials

Figure S1. Comparison of the volatile (A) and phenolic (B) compound content in oil before and after centrifugation. Data at the decanter exit and centrifuge exit. The error bars show the standard deviation. Figure S2. Comparison of the volatile (A) and phenolic (B) compound content in unfiltered and filtered oil. Data at the unfiltered and filtered oils. The error bars show the standard deviation. (*Supplementary Materials*)

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