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Chemical characterization and oxidative status of olive oils extracted by expeller pressing and supercritical CO_2 extraction: Impact on quality standards and their regulatory recognition

Assamae Chabni [a,b,*], Celia Bañares [c], Luis Vázquez [a,b], Carlos F. Torres [a,b]

[a] Departmental Section of Food Sciences, Faculty of Sciences, Autonomous University of Madrid, Madrid 28049, Spain
[b] Department of Production and Characterization of Novel Foods, Institute of Food Science Research (CIAL, CSIC-UAM), C/Nicolas Cabrera 9, Cantoblanco Campus, Madrid 28049, Spain
[c] Departmental Section of Nutrition and Food Science, Faculty of Veterinary Medicine, Universidad Complutense de Madrid, Av. Puerta de Hierro s/n, Madrid 28040, Spain

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ABSTRACT

Innovative extraction methods, such as the sequential combination of expeller pressing and supercritical CO_2 (SC-CO_2), were applied to obtain high quality olive oil (OO) while improving sustainability and reducing environmental impact. In this study, the physicochemical properties of the oils obtained at the Spanish Association for Standardization and Certification were evaluated following the quality standards established by the European Union and the International Olive Council. The results revealed that the combined yield of the sequential process was 87 %, significantly higher than centrifugation-based extraction methods, which typically yield around 75–80 %. The oils showed high phenolic concentrations (1013–1855 mg/kg), and superior oxidative stability, underlining their potential for functional and premium market applications. Acidity and peroxide values are lower than those established for virgin olive oil. However, some parameters (e.g., K_{232} and K_{270}) exceeded these limits, probably due to the unique extraction processes employed. These results highlight the need to update regulations to include new extraction methods and validate these oils as high-value sustainable products.

1. Introduction

Olive oil (OO) is a product of great relevance both for its health benefits and for its sensory and nutritional properties, which makes it a food with high added value (Jimenez-Lopez et al., 2020; Uceda et al., 2006). Its quality is influenced by several factors, including the olive variety and its ripening, time of harvest, extraction process, and oxidative stability of the oil, which has been linked to the presence of phenolic compounds (PC) and other natural antioxidants (Salvador et al., 2001; Talhaoui et al., 2016).

The conventional method for obtaining OO includes processes such as pressing or two- or three-phase centrifugation, which separate the oil from the vegetation water and solids. However, these methods leave a large amount of PC in by-products such as oil mill wastewater (OMW) and pomace, which creates environmental problems (Clodoveo, 2019; Marx et al., 2022; Novoselić et al., 2021). Alternative methods such as controlled olive dehydration, expeller pressing and supercritical CO_2 extraction (SC-CO_2) have proven to be more sustainable and efficient for obtaining PC-rich oils (Chabni et al., 2025b, 2023b). These technologies eliminate the use of water, maximize the extraction of a high-quality oil, free of refining and generate a defatted flour usable as a food ingredient. Furthermore, the oils obtained from dehydrated olives using the alternative method of expeller pressing and SC-CO_2, shows high quality and their quality indexes below those established for EVOO. In addition, they present fatty acid profiles similar to those obtained by conventional methods (Chabni et al., 2023a, 2023b; Guillaume et al., 2018). Moreover, their high PC content is strongly correlated with their oxidative stability, which underlines their potential as sustainable and high-quality alternatives (Chabni et al., 2024). Nevertheless, according to EU Regulation 2022/2104 (EEC, 2022), OO must be obtained only by mechanical means from intact olives, which excludes methods such as fruit dehydration or supercritical fluid extraction. However, the

Abbreviations: **cOO**, control olive oil; **eOO**, expeller olive oil; **SCOO**, supercritical CO_2 olive oil; **EE**, expeller extraction; **PCs**, phenolic compounds.

* Corresponding author at: Departmental Section of Food Sciences, Faculty of Sciences, Autonomous University of Madrid, Madrid 28049, Spain.
E-mail addresses: Assamae.chabni@uam.es (A. Chabni), Celiaban@ucm.es (C. Bañares), Luis.vazquez@uam.es (L. Vázquez), Carlos.torres@uam.es (C.F. Torres).

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development and validation of these alternative methods - while ensuring product quality and safety - opens the door to their future recognition in frameworks such as the Novel Foods Regulation (Union, 2015).

Olive oils extracted by supercritical fluid methodology are not yet commercially available; however, oils from dehydrated olives - such as *Alwana* oil sold in North African markets - respond to a clear consumer demand (Guillaume et al., 2018). Although heating processes could potentially degrade olive fruit, previous studies have shown no significant alteration of the oil's physicochemical quality. In *Alwana* oil, dehydration does modify the sensory profile - imparting a smoky aroma and flavor- but these notes are regarded as positive attributes rather than defects (Chabni et al., 2023a). Quality certification under European Commission regulations (EEC 2022/2104) remains essential to guarantee authenticity and competitiveness in the global market (Bajoub et al., 2018). In addition, mass-balance analyses from earlier work indicate that dehydration and heat treatment do not cause a significant loss of phenolic compounds, underscoring the robustness of these sustainable extraction methods (Chabni et al., 2025a, 2025b).

The commercial quality and purity criteria for OO defined by international legislative standards include several parameters, such as acidity, which measures free fatty acids, where low acidity indicates high quality, such as values of < 0.8 % for extra virgin olive oil (EVOO) (COI, 2019; EEC, 2022). The peroxide value (PV), which reveals the initial oxidation of the oil and whose elevation suggests deterioration and loss of essential antioxidants such as vitamin E, is measured in milliequivalents (mEq) of active oxygen per kg of oil. Its limit for consumption is 20 mEq O_2/kg (AOAC, 2002; Morales and Przybylski, 2013). The extinction coefficients k_{232} and k_{270} are based on ultraviolet (UV) spectrophotometric measurement at wavelengths of 232 and 270 nm. The first, like the PV, indicates the primary oxidation of the oil through the absorption of hydroperoxides at 232 nm, and its maximum limit for EVOO is 2.5 and for virgin olive oil (VOO), 2.6. The second coefficient detects a more advanced oxidative state of hydroperoxides since, as the oxidative process progresses, the peroxides are modified to obtain another type of component such as α-dicetones or α-unsaturated ketones that absorb UV light at 270 nm. The limits established for k_{270} are 0.2 for EVOO and 0.25 for virgin olive oil (VOO). Extinction coefficients also serve as a purity criterion to identify the presence of refined oils in a blend, since, during refining processes, conjugated dienes and trienes are generated that also absorb at 266 and 274 nm, for which Delta K (ΔK) is calculated. For virgin oils, the value of k_{270} is approximately the semisum of k_{266} and k_{274}, resulting in a ΔK of 0, or less than 0.01 in EVOO and VOO (Houshia et al., 2014).

However, results obtained through UV spectroscopy should be interpreted with caution, as dienes and trienes are not the only compounds that absorb at these wavelengths. PC also absorb in the 220–320 nm range, overlapping with diene and triene signals. Their various classes (phenolic acids, simple phenols, secoiridoids, lignans, flavonoids) each exhibit characteristic peaks - often multiple maxima between 200 and 290 nm - while elenolic acid and hydroxytyrosol derivatives absorb near 240 nm, other phenolics around 280 nm, and flavonoids/phenolic acids near 320 nm (Chabni et al., 2024; Fuentes et al., 2012; Orfanakis et al., 2023).

Another quality parameter is sensory analysis, the organoleptic characteristics of OO, such as fruitiness, bitterness and pungency, along with its balance and absence of defects, are key to assessing its quality and authenticity. EVOO stand out for offering a complex and well-balanced sensory experience, while the detection of defects ensures the identification of lower quality or poorly handled products, thus highlighting the importance of tasting in their assessment (Bendini et al., 2012; Chabni et al., 2023a).

In order to guarantee the purity of the OO, parameters such as wax content, which comes from the esterification of aliphatic alcohols with free fatty acids, are also determined (Uncu and Ozen, 2022). Their analysis makes it possible to identify mixtures with pomace oils, since virgin oils contain up to 150 mg/kg of waxes while pomace oils can exceed 350 mg/kg (EEC, 2022). The composition of fatty acids and triglycerides, which contributes to the verification of purity, highlighting oleic acid as the main component in OO; stigmastadiene compounds, total sterol content, erythrodiol and uvaol, which act as indicators of refining or adulteration through the addition of seed oils, together with the determination of the composition of triglycerides expressed in their equivalent carbon number (ECN) (COI,2019; Proietti et al., 2017).

This study aims to chemically characterize the oils obtained by expeller pressing extraction and SC-CO_2 from dehydrated olives, following the standards established by the Spanish Association for Standardization and Certification (AENOR) and the European Commission regulations (EEC, 2022/2104). This work contributes to that effort by proposing a clean and sustainable extraction process outside the current legal definition of virgin olive oil. This approach allows evaluating the quality, purity and oxidative stability of the oils, as well as their potential for market introduction as sustainable products with high added value. In addition, the main quality indices were evaluated by external and internal analysis to ensure the consistency and reliability of the results.

2. Materials and methods

2.1. Materials

The olives were *Verdeja* variety (also known as *Castellana*), produced at the beginning of the 2022/2023 crop season, provided by private farmer in Toledo, Spain. For the extraction by supercritical fluids, carbon dioxide (CO_2) with a purity of 99.9 % was acquired from Carburos Metálicos (Barcelona, Spain). Solvents used were cyclohexane (CHEX) and methanol (MeOH). Regarding reagents used for some determinations, these were phenolphthalein indicator, Folin-Ciocalteu reagent and sodium carbonate (Panreac, Barcelona, Spain), hydroxytyrosol standard $\geq$ 98 % supplied by Seprox Biotech (Madrid, Spain), Tyrosol $\geq$ 98 %, α-tocopherol $\geq$ 98 %, and elenolic acid $\geq$ 95 % were supplied by Sigma-Aldrich (St. Louis, USA). Commercial extra virgin olive oil (EVOO, Fidelco, Guadalajara, Spain) obtained exclusively by mechanical processes from *Verdeja* variety, was used as a known reference for the different determinations carried out.

2.2. Methods

2.2.1. Two-step sequential extraction of olive oil

Step 1: Expeller olive oil extraction

Clean olives were dehydrated in a ventilated oven (Memmert 600, Memmert GmbH + Co. KG, Schwabach, Germany) at 105 °C and 4.5 h. After removing the bone with an olive pitter (Westmark, Germany), 250 g of dehydrated and pitted olives were used to extract OO in an oil expeller for domestic use Rommelsbacher, brand model OP 700 (Dinkelsbühl, Germany). After the pressing process, expeller OO (eOO) and press cake were obtained. The oil was filtered to remove any olive solid material, then weighed, and stored in amber glass vials under an inert atmosphere (N_2) at room temperature (18 ºC) in a dark room. The press cake was vacuum packed and stored under refrigeration at 4 °C.

To evaluate the efficiency of the press expeller extraction system of OO, the extraction yield (EY) (1), oil recovery (OR) (2), and material balance (MB) (3) were calculated as follows:

$$\text{Extraction yield (\%)} = (\text{Oil weight obtained}) / (\text{Initial sample weight}) \times 100 \quad (1)$$

$$\text{Oil recovery (\%)} = (\text{Oil weight obtained}) / (\text{Total fat content in source material*}) \times 100 \quad (2)$$

Material balance (%) = (Oil weight + defatted flour) / (Initial sample weight) × 100 (3)

* The total fat content of the dehydrated and pitted olives was determined using the Folch method (Folch et al., 1957), briefly, 10 g of sample was mixed with 200 mL of chloroform:methanol (2:1 v/v), homogenized with an Ultraturrax T18 (IKA, Germany) and stirred for 60 min. The organic phase was combined with water, centrifuged and the lower phases were evaporated to constant weight using a rotary evaporator at 40°C under 10^{-3} MPa to constant weight.

Step 2: Supercritical CO_2 oil extraction from the press cake

Olive press cake obtained after the expeller extraction was used in a SC-CO_2 pilot plant to extract the remaining oil, following the procedure described by Vázquez et al. (2024). The equipment works with SC-CO_2 up to maximum pressure of 413 bar and temperature of 150 °C. Briefly, 150 g of press cake with a particle size between 250 and 1000 μm were used to perform a SC-CO_2 extraction in a home-made pilot plant device consisting of a 350 cm^3 cylindrical extraction vessel (EV), and two different separators (S1 and S2), each with a capacity of 270 cm^3, with independent control of temperature and pressure. In general, it is recommended that the ratio between the height and diameter of the cylindrical EV be 5–7. The equipment design involves a batch procedure to incorporate the press cake into the EV. The CO_2 is first heated to the desired temperature and then pumped (P1) from the bottom of the EV, up to the desired extraction pressure. For proper pumping and to avoid pump cavitation, it is necessary to pre-cool the CO_2. At the outlet of the extractor, CO_2 with the extracted solute flows through a depressurization valve (V) to the separators. The existence of two separators allows the fractionation of extracts, but in this case, it is not necessary, therefore it is depressurized at the same pressure and the two oils are joined. In this study, the SC-CO_2 conditions were 250 bar of pressure, at 40 °C for 90 min, with CO_2 flows of 100 g/min.

The oil obtained from this extraction process will be named hereafter supercritical CO_2 OO (SCOO). The final by-product was an olive-defatted flour that was vacuum packed and stored under refrigeration at 4 °C. This process was carried out in triplicate.

To evaluate de efficiency of the two-step sequential extraction of OO, the extraction yield, oil recovery, and material balance were calculated for each process as described above (Eqs. (1), (2) and (3), respectively). Finally, the combined yield (CY) of the two methodologies, taking into account the total fat content of the dehydrated and pitted olives, was calculated as follows:

CY = ((Oil obtained by expeller + Oil obtained by supercritical CO_2) / Total Fat content of the sample) × 100 (4)

2.2.2. Particle size of the press cake

For this purpose, 100 g of the press cake were introduced into a TZA1491 electric sieve shaker (Puebla, Mexico) with four sieves with different pore sizes (>1000 μm, 1000–500 μm, 500–250 μm, 250–100 μm) and a container, in which particles less than 100 μm in size were collected. The samples were left in the sieve shaker with ultrasonic vibration for 6 min, with an amplitude of 1.8 mm and cycles of 9.0 s on and 1.0 s off. Finally, each fraction was collected and weighed.

2.2.3. Evaluation of the acidity, oxidation status and total phenolic compounds

Acidity was measured and expressed as percentage of free oleic acid. PV was expressed as milliequivalents of active oxygen per kilogram of oil (meq O_2 kg^{-1}). The extinction coefficients K_{232} and K_{270} (UV absorbance) were determined from the absorption at the exact λ wavelengths in nm, according to the analytical methods described in the European Commission Regulation EEC 2568/91 and subsequent amendments (the most recent being EU 1348/2013) (EEC, 2022). In addition, the extinction coefficients K_{266} and K_{274} were determined to calculate ΔK, to evaluate the adulteration ratio, as

$$\Delta K = K_{270} - ((K_{266} + K_{274}) / 2) \quad (5)$$

$$\text{With: } K_\lambda = Abs_\lambda / (C \cdot S) \quad (6)$$

where C is the molar concentration and S is the cuvette path length.

To measure the formation of oxidation secondary products, the p-anisidine value (AnV) was determined by applying the AOCS Official Method Cd 18–90 using an Oxitester at 366 nm (Firestone, 2009). These determinations were carried out in duplicate to evaluate the initial status of the oils.

In addition, the total oxidative deterioration was evaluated by calculating the TOTOX value according to:

$$TOTOX = AnV + 2\,PV \quad (7)$$

The total phenolic compounds (TPC) of the obtained oil and the commercial EVOO was determined by the Folin–Ciocalteu spectrophotometric method at 760 nm. A calibration curve was prepared in the range 0.2–1.0 mg/mL ($R^2 = 0.9986$), and results were expressed as mg hydroxytyrosol equivalents (HTE) per kg of oil (Figure S1) (Gutfinger, 1981). These determinations were carried out in duplicate for each sample.

2.2.4. UV-Vis spectra acquisition

Spectra of the commercial EVOO (23 mg mL^{-1}), eOO (11.8 mg mL^{-1}) and SCOO (10.4 mg mL^{-1}), dissolved in cyclohexane and some minority compounds (hydroxytyrosol, tyrosol, elenolic acid, α-tocopherol and luteolin-7-O-glucoside) dissolved in MeOH:H_2O (80/20, v/v), were collected using a UV-Vis spectrophotometer (Shimadzu UV-2450 Kyoto, Japan) equipped with a deuterium discharge lamp as a source for the ultraviolet wavelength range and a tungsten lamp for the visible range, and a resolution of 2.0 nm was used. Absorption spectra of the oils were acquired in the ultraviolet and visible ranges (200–800 nm).

2.2.5. Olive oil characterization external assessment

To check whether the characterization of the oils obtained in the laboratory was in accordance with official legislation, the same parameter characterization described above were carried out at the Spanish Association for Standardization and Certification (AENOR, Madrid, Spain) by following the quality standards established by Regulation (EEC) No. 2105/22 on the characteristics of olive and olive-pomace oils (EEC, 2022). The physicochemical parameters of the oils obtained were determined in accordance with COI/T.15/NC No. 3/Rev. 19. Free acidity (% oleic acid) and peroxide value (meq O_2/kg) were determined by volumetry, the extinction coefficients (K_{232} and K_{270}) by ultraviolet visible spectrophotometry (UV-Vis). Fatty acid composition was analyzed by gas chromatography with flame ionization detector (GC-FID) with a fused silica capillary column (50–60 m x 0.25–0.30 mm i.d.) coated with cyanopropylpolysiloxane phases (SP-2380) with 0.20–0.25 μm film thickness, determining fatty acid methyl esters as detailed in the IOC method COI/T.20/Doc. No 33/Rev.1 (COI,2019). The determination of fatty acid methyl esters (FAME) was carried out according to the IOC official method COI/T.20/Doc. No 33/Rev.1 (COI, 2019). FAMEs were identified and quantified using a certified standard mixture (e.g., Supelco 37 Component FAME Mix), injected under the same chromatographic conditions as the samples. Identification was based on retention time comparison, and quantification was performed by area normalization.

The same chromatographic method was used to determine the content of waxes, ethyl esters, the composition and content of sterols (including cholesterol, brassicasterol, campesterol, stigmasterol, δ-7-stigmastenol and apparent β-sitosterol, which is the sum of δ-5–23-stigmastadienol, clerosterol, β-sitosterol, sitostanol, δ-5-avenasterol and δ-5–23-stigmastadienol), erythrodiol, uvaol and triterpenic dialcohols (stigmasta-3,5-diene). Additionally, the difference between the actual and theoretical triacylglycerol content was determined using the

equivalent carbon number (ECN 42). Quantification of waxes and ethyl esters was performed using internal standards, lauryl arachidate and methyl heptadecanoate, respectively, in accordance with IOC method COI/T.20/Doc. No 28/Rev. 4.

All chromatographic analyses were performed following official methods established by the International Olive Council (IOC) and adopted by the European Union (COI,2019; EEC, 2022). These methods have been validated through international inter-laboratory studies in compliance with ISO 5725, providing data on method repeatability, reproducibility, and uncertainty. Although compound-specific parameters such as LOD, LOQ, matrix effect, and intra/interday precision are not individually reported in the IOC protocols, method robustness is ensured through standardization and the use of certified reference materials.

2.2.6. Data analysis

Internally, each extraction was carried out in duplicate, and all analytical measurements were performed in triplicate for each extraction. The data was presented as mean ± standard deviation. All statistical analyses were carried out using Origin (version 9.0 for Windows; OriginLab Corporation, Northampton, USA).

3. Results and discussion

3.1. Total fat, processing moisture and eficiency parameters

The olives processed have a moisture content of 65.4 %, which confirms that they are from the early harvest (October-November) (Alowaiesh et al., 2016). Dehydration has reduced the moisture content by 97 % and the oil content has become 51 % of the weight of the olive (Table 1), making it more accessible for extraction. When the olive is dehydrated, its plasticity decreases, leading to greater compression during extraction with the expeller press and, as a result, enhanced oil recovery (Bañares et al., 2022; Chabni et al., 2023b).

Table 1 also presents the extraction yields and oil recovery achieved through the expeller press and SC-CO_2 methods. These results consider the initial oil content of the starting materials: dehydrated and pitted olives for the press and press cake for SC-CO_2. The extraction yield of the expeller press was 27.4 %, which corresponds to the recovery of 55.6 % of the total oil contained in the olives. As for SC-CO_2, the extraction yield was 26.8 %, which corresponds to 75.2 % of the oil contained in the press cake. The results obtained by press are similar to those obtained previously for dehydrated olives of the Moroccan Picholine and Cornicabra varieties (Chabni et al., 2024, 2023b). The results obtained by SC-CO_2 were slightly lower, than in previous extraction probably caused by the fact that those were carried out in a stirred extraction cell (Bañares et al., 2022). Under agitated conditions, CO_2 is distributed improving the extraction yield. In contrast, without agitation, CO_2 takes preferential paths and is not transferred to the whole cake (Bañares et al., 2022; Vázquez et al., 2024).

The sequential extraction combining expeller pressing and supercritical CO_2 achieved a total yield of 87 %, significantly surpassing the yields of conventional methods and those reported by other authors for OO using SC-CO_2 (Al-Otoom et al., 2014). This process represents an innovative and efficient approach, yielding two distinct types of oils that can be processed individually or separately, along with a defatted flour rich in nutrients and bioactive compounds, with multiple potential applications, such as food industry or development of biodegradable packaging films (Chabni et al., 2025a). Moreover, these integrated clean extraction techniques are safe and environmentally friendly. They eliminate the need for water, avoid the production of pomace and aqueous residues, and do not rely on organic solvents to extract residual oil from the press cake.

Evaluating the granulometry of the material prior to initiating extraction with SC-CO_2 is crucial, as the initial particle size of the press cake can significantly influence the extraction process. Therefore, this parameter was analyzed before proceeding with the extraction. Table 1 reveals that the press cake primarily consists of particles larger than 500 μm (69.2), followed by particles between 500 and 250 μm (25.7 %), and 250–100 μm (5.1 %). Despite this particle size distribution, it was found to be suitable for extracting the residual oil in the press cake using SC-CO_2 technology.

Table 1
Characteristics of the starting materials (dehydrated olives and olive press cake): Fat content of the olives, processing moisture, extraction yield, oil recovery, combined yield, and material balance of the extractions; expressed in percentage (%). Values for the expeller OO (eOO) and supercritical OO (SCOO) are included. Different letters within a row indicate statistically significant differences ($p < 0.05$).

Dehydrated stoneless olives (100 %)		
Fat content	50.9 ± 1.2	
Processing moisture	2.22 ± 0.55	
Processing parameters		
	Expeller press extraction (eOO)	SC-CO_2 extraction (SCOO)
Extraction yield	27.4 ± 1.3	26.8 ± 0.8
Oil recovery	55.6 ± 1.2b	75.2 ± 2.4a
Combined yield	86.9 ± 1.7	
Material balance	93.8 ± 3.5	
Olive press-cake		
Fat content	35.6 ± 2.8	
	Diameter (μm)	Percentage (%)
Granulometry	> 500	69.2 ± 1.9
	500–250	25.7 ± 2.0
	250–100	5.1 ± 0.4

3.2. Quality indices and oxidative status of extracted oils (laboratory assessment)

The quality indices systematically measured (acid value (AV), peroxide value (PV), K_{232}, K_{270} and ΔK) of the OO produced are presented in Table 2. It shows the results determined just after extraction of the different oils, before taking them to AENOR. The *p*-anisidine value was also determined to see secondary oxidation, tand TOTOX was calculated. The results of each parameter were compared to the limits defined by the European Legislation (EEC) No. 2104/22 (EEC, 2022).

3.2.1. Quality indices

In the acidity index (AV), the eOO complies with the legislated limit (≤ 0.80), registering a value of 0.66 %. On the other hand, the SCOO exceeds this limit with a value of 1.01 %. Regarding the peroxides index (PV), both oils are well below the maximum allowable limit (≤ 20), with values of 2.90 for eOO and 4.82 meq O_2/kg oil for SCOO. This reflects an acceptable state of primary oxidation in both oils, although SCOO presents a slightly higher value, suggesting a possible lower initial oxidative stability.

Table 2
Quality indices determined internally: acidity value (AV), peroxide value (PV), extinction coefficients (K_{232} and K_{270}), $^{\Delta K}$ (adulteration ratio), *p*-anisidine value (AnV), and TOTOX of expeller OO (eOO) and supercritical OO (SCOO). Different letters within a row indicate statistically significant differences ($p < 0.05$).

	eOO	SCOO	EVOO*
AV (% oleic acid)	0.66 ± 0.11b	1.01 ± 0.03a	≤ 0.80
PV (meq O2/kg oil)	2.90 ± 0.75	4.82 ± 1.34	≤ 20
K232	2.48 ± 0.00	2.84 ± 0.05	≤ 2.50
K270	0.38 ± 0.00	0.35 ± 0.01	≤ 0.22
ΔK	−0.007 ± 0.00b	−0.016 ± 0.0a	≤ 0.01
AnV	3.41 ± 0.79	3.28 ± 0.78	-
TOTOX	9.22 ± 0.79	12.92 ± 2.22	-

*EVOO: extra virgin OO quality criteria, value limits set by International Oil Council.

For spectrophotometric parameters (K_{232} and K_{270}), eOO oil complies with the legislated limit for K_{232} ($\leq$ 2.50), with a value of 2.48, while SCOO oil slightly exceeds it (2.84). For K_{270}, both oils exceed the allowable limit ($\leq$ 0.22), with values of 0.38 in eOO and 0.35 in SCOO. In the ΔK parameter, none of the oils complies with the established limit ($\leq$ 0.01), which could be due to spectrophotometric variations specific to the extraction methods used.

High K_{232} values are usually attributed to a higher presence of compounds derived from primary oxidation, such as hydroperoxides. And those of K_{270} indicate the presence of compounds formed from the decomposition of the initial hydroperoxides (Velasco and Dobarganes, 2002). As noted previously, these results should be interpreted with caution. Whereas the peroxide and *p*-anisidine values are low in both oils, suggesting good oxidative stability, the high K_{232} and K_{270} values indicate other possible causes. It is important to note that compounds other than dienes and trienes, such as certain phenolic compounds, can also absorb at these wavelengths. Peroxides and *p*-anisidine values are low in both oils so this suggests other possible causes make K_{232} and K_{270} high. Dienes and trienes are not the only compounds that absorb at these wavelengths (Chabni et al., 2024; Fuentes et al., 2012). Fig. 1 presents the absorbance spectra as a function of wavelength (230–320 nm) of different compounds and oils: hydroxytyrosol (HT), tyrosol (TYR), elenolic acid (EA), α-tocopherol, luteolin-7-O-glucoside, commercial EVOO, eOO and SCOO. These spectra allow comparison of the optical properties of pure compounds and oils. HT (blue curve) and TYR (black curve) absorb between 220 and 295 nm, characteristic of this phenolic compound, known for its antioxidant properties (Choe and Min, 2006). On the other hand, EA (yellow) shows a high absorbance in the region close to 250 nm, attributable to its polycyclic aromatic structure, which underlines its antioxidant potential different from that of other compounds. Alfa-tocopherol (pink), a lipophilic antioxidant, exhibits a unique spectrum indicating its characteristic activity and chemical structure (Choe and Min, 2006; Psomiadou et al., 2000). Also noteworthy is luteolin-7-O-glucoside, a flavonoid whose presence is related to the ripening process and the variety of olive and absorbs in a range between 235 and 275 nm.

Regarding the oils, EVOO (dark green) shows a lower absorbance compared to the extracted oils, which may indicate a lower content of specific phenolic compounds and, consequently, a greater susceptibility to oxidation. SCOO (red) and eOO (light green) have higher overall absorbance in the 250–290 nm range, which may be indicative of a significant concentration of specific PC and/or initial oxidation products, such as hydroperoxides, which tend to absorb at these wavelengths (Orfanakis et al., 2023), both parameters are high in both oils (eOO and SCOO). The total of phenolic content obtained in this study for commercial EVOO, eOO and SCOO is $297^{\pm 27}$, $1013^{\pm 31}$ and $1855^{\pm 210}$ mg/kg of oil, respectively. These eOO and SCOO results are much higher than those obtained by other authors for oils of the same olive variety, extracted by traditional methods (~500 mg/kg) (Pardo et al., 2019), which could explain this variation in absorbance with respect to the EVOO.

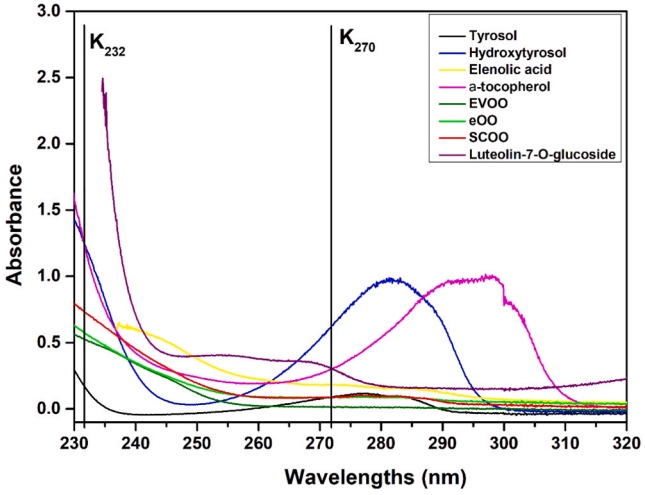

Fig. 1. Absorbance spectra as a function of wavelength (230–320 nm) of hydroxytyrosol (blue), tyrosol (black) elenolic acid (yellow), α-tocopherol (pink), luteolin-7-O-glucoside (purple), commercial EVOO (dark green), eOO (light green) and SCOO (red).

Particularly sensitive is the region between 230 and 240 nm in the UV-Vis spectra of oils and related compounds. The varying absorbances observed in this region may be due to several reasons, such as the presence of secondary oxidation products (conjugated aldehydes and ketones). The presence of specific PC, such as HT and TYR derivatives, show absorbance in this region due to their electron transitions (π-π*) in aromatic rings conjugated to functional groups (Guclu et al., 2021; Tsimidou et al., 1992). The concentration and composition of these compounds can vary in oils depending on the extraction method and the amount of water present, which can entrain or degrade certain antioxidants during the process (Chabni et al., 2023b; Novoselić et al., 2021). Oils may also contain trace amounts of compounds that are not detectable in other regions of the spectrum, but have significant absorbance between 230 and 240 nm. This could include oxidized pigment residues or even lipid contaminants from the environment or extraction process (Borello et al., 2021). Furthermore, since EU regulations do not set legal limits for the composition of phenolic compounds, an oil can be classified as EVOO regardless of its content of phenolic compounds. It is known that higher quality commercial OOs, such as EVOO and VOO, usually contain low amounts of polyphenols (López-Huertas et al., 2021).

However, it should be noted that the oils obtained by these extraction methods were previously characterized by chromatographic techniques (HPLC for phenolic compounds and GC for lipid profile). These analyses confirmed the absence of conjugated dienes and trienes, supporting the reliability of the extinction coefficient data (Chabni et al., 2025b, 2024). Although current legislation does not require chromatographic analyses for OO classification, these complementary techniques may improve the interpretation of oxidative behavior and will be considered in future work.

3.2.2. *Secondary oxidation parameters*

Regarding secondary oxidation, OO quality regulations, especially in the European context (e.g., EC Regulation No. 2104/22 and its amendments), do not include parameters such as AnV or TOTOX index to assess oxidation (COI,2019; EEC, 2022). These indices are more commonly used for refined or seed oils, where secondary oxidation is a more frequent issue. AnV measures aldehydes formed during secondary oxidation, which are responsible for rancid odors. Since VOO are not refined, secondary oxidation is less relevant, therefore, the focus is on primary oxidation (peroxides) and natural antioxidants that reflect freshness and initial quality (Labrinea et al., 2001). Similarly, the TOTOX index, which combines PV and AnV, is more appropriate for refined oils. The OO standard gives priority to parameters such as primary oxidation (peroxides index, UV absorbance) and stability, because it considers they represent better the natural properties and freshness of VOO, where secondary oxidation is naturally attenuated by antioxidants such as polyphenols and tocopherols. However, hydroperoxides are transient chemical compounds and, despite being an obligatory parameter of the oxidative state of packaged olive and vegetable oils, they do not always correlate directly with the oxidation of the sample. AnV, despite being a more empirical determination, is well correlated with the level of secondary oxidation products (aldehydes), which are much more stable than hydroperoxides (Casal et al., 2010). Therefore, for an accurate estimation of the oxidation state, both parameters must be interpreted simultaneously.

No significant differences were observed between the AnV of eOO

and SCOO, with values of 3.41 and 3.28, respectively. These results are similar to those obtained by other authors for EVOO before being subjected to frying processes (2.5) (Baiano et al., 2005; Casal et al., 2010), and much lower than those obtained by Galanakis et al. (2018) for fresh OO (9−10). Furthermore, the AnV are within the limits established by legislation for vegetable or refined oils (<10), indicating the almost absence of secondary oxidation products. However, the TOTOX value, which reflects the sum of primary and secondary oxidation, is higher in SCOO (12.92) compared to eOO (9.22), and much lower than those obtained by other authors for VOO (~20) (Galanakis et al., 2018).

3.2.3. *Visible spectrum of olive oil*

The absorption spectra of oils and other compounds within the visible range (400–750 nm) are presented in Fig. 2. Commercial oil catalogued as EVOO (dark green) shows smaller absorption peaks despite doubling the analysis concentration (23 mg mL^{-1}) of eOO (11.8 mg mL^{-1}) and SCOO (10.4 mg mL^{-1}). In contrast to the spectra observed in the literature on pure EVOO, which usually shows carotenoid peaks at 415, 456 and 485 nm and a peak at 671 nm corresponding to chlorophyll (Lagouri et al., 2017; Philippidis et al., 2017). The Low absorption peaks indicate that they could indicate degradation of the oil during processing and storage (Ruiz-Méndez et al., 2013). In contrast, eOO (light green) and SCOO (red) show pronounced peaks in the regions corresponding to β-carotene, lutein and pheophytin a, confirming the presence of these natural pigments that provide antioxidant properties and are indicators of unrefined oils (Lagouri et al., 2017; Moyano et al., 2010).

These findings highlight the usefulness of spectroscopic techniques, such as visible and fluorescence spectroscopy, for identify adulterations and assess the authenticity of OOs. Visible spectroscopy can detect the presence or absence of these key compounds in the oil, providing a rapid and non-destructive tool to differentiate virgin from refined oils. Moreover, it aligns with previous observations on the advantages of these optical methods to evaluate quality parameters such as oxidative status and phenolic antioxidant contents, eliminating the need for more complex chemical techniques (Borello et al., 2021; Gonçalves et al., 2018; Guzmán et al., 2015). Thus, this spectroscopic approach can complement or replace traditional methods to guarantee the authenticity and quality of OO.

3.3. *Olive oil characterization (external assesment)*

3.3.1. *Quality parameters*

The oils obtained were analyzed using the same methods by the Spanish Association for Standardization and Certification (AENOR) (Table 3) and some discrepancies were identified compared to the internally obtained results for acidity, peroxide values, and extinction coefficients (K_{232} and K_{270}). Regarding AV and PV, both measurements are within the limits established for EVOO (≤0.80). The differences could be attributed to variations in analytical conditions or changes in the sample due to the time elapsed between analyses (one week after extraction and internal analysis). As shown in Table 2, the initial oxidation level in eOO is lower than the value reported by AENOR for the same oil sample (Table 3). This trend is not observed in SCOO, as, according to a previous study, the oxidation of eOO progresses slightly faster due to differences in the composition of antioxidants (Chabni et al., 2024). As for the spectrophotometric indexes, the initial values are at the limit of what is considered EVOO (Table 2). However, the results reported by AENOR exceed the limits established for VOO. This discrepancy could suggest the onset of secondary oxidation, as well as the presence of absorbing compounds at these wavelengths, as previously mentioned.

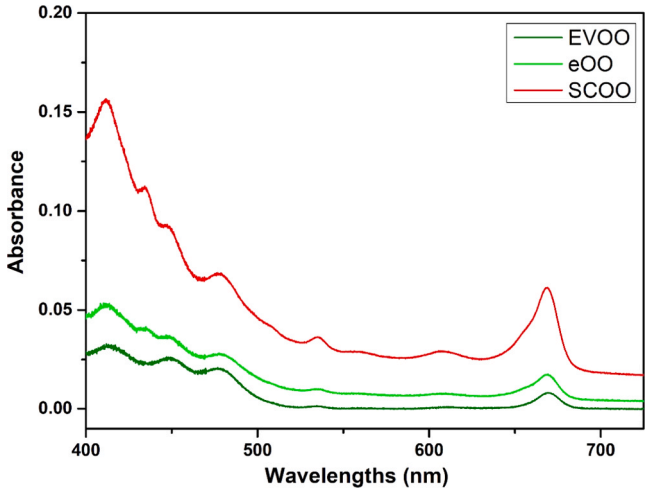

Fig. 2. Absorption spectra in the visible region (400–750 nm) of commercial EVOO (dark green), eOO (light green) and SCOO (red).

3.3.2. *Fatty acid profile and wax content*

The fatty acid composition of the oils analyzed (eOO and SCOO) is representative of the typical characteristics of an EVOO. As for saturated fatty acids, the levels of palmitic acid (13.11 % in eOO and 13.52 % in SCOO) and stearic acid (~2 %) are within the permitted ranges, while myristic acid is found in negligible traces (0.01 %). Monounsaturated fatty acids, led by oleic acid, are predominant, with values of 76.71 % for eOO and 76.26 % for SCOO, reflecting the high quality of the oil in terms of health benefits. Also, palmitoleic acid is present in typical proportions (~1.38 %). As for polyunsaturated fatty acids, linoleic acid and linolenic acid present low levels (5.13–5.14 % and 0.69–0.70 %, respectively), which may contribute positively to the oxidative stability of the oils. On the other hand, the presence of trans isomers is minimal (≤ 0.03 %), indicating that the applied extraction methods do not promote the formation of undesired compounds, thus maintaining the nutritional and functional quality of the oils. Finally, minority fatty acids, such as arachidic, behenic and lignoceric, are present in very low amounts, complying with international standards for EVOO. This fatty acid profile supports the results obtained in previous studies for oils from other varieties, that both the expeller extraction process and the SC-CO_2 process preserve the characteristic properties of OO, guaranteeing its quality and functionality (Chabni et al., 2024, 2023b, 2023a).

Other parameters, such as wax content and fatty acid ethyl esters (FAEE), have been adopted as quality indicators under European Union directives (EEC, 2022). Wax content serves as a purity criterion for VOO and EVOO (Diarte et al., 2021). FAEE, on the other hand, is used to differentiate the “extra virgin” category from lower-quality oils, which may result from damaged olives or improper practices during extraction and storage (Beltran et al., 2021). In other hand, ethanol, the alcohol precursor of ethyl esters, can be produced by microbial fermentation during storage. This ethanol reacts with free fatty acids in VOO, leading to increased FAEE levels. The wax contents in the oils were 49 mg/kg for eOO and 89 mg/kg for SCOO, both below the established maximum limit of 150 mg/kg suggesting no adulteration with other oils. The ethyl ester content in eOO was 8 mg/kg, well below the legal limit of 35 mg/kg for EVOO. Ethyl ester content was not determined for SCOO as supercritical fluid technology was not considered as a valid method of VOO extraction (EEC, 2022). The low concentration of ethyl esters in eOO reflects adequate control during extraction, minimizing ethanol formation from microbial fermentation. This suggests that handling and processing practices (olive dehydration) ensured quality raw material and appropriate storage conditions. Overall, these purity parameters confirm the authenticity of the oils obtained by the proposed extraction methods and support their potential as high quality, unadulterated products.

Table 3
Quality indices, fatty acid profile, wax and ethyl ester content, and determination of the composition and content of sterols and triterpenic dialcohols of eOO and SCOO, by Spanish Association for Standardization and Certification (AENOR). ß-Sitosterol (apparent)* = δ-5–23-stigmastadienol + clerosterol + β-sitosterol + sitostanol + δ-5-avenasterol + δ-5–24-stigmastadienol. Results from external analysis provided by AENOR as single values; standard deviations were not reported. Non detected (n.d.).

Parameters	Results		EVOO limits	Method
	eOO	SCOO		
Acidity Value	0.43 % oleic acid	0.63 % oleic acid	≤ 0.80	Volumetry
Peroxide Value	6.0 meq O2/kg oil	4.2 meq O2/kg oil	≤ 20	Volumetry
Spectrophotometric Assay				
K232	3.30	3.72	≤ 2.50	UV-Vis
K270	0.51	0.49	≤ 0.22	UV-Vis
ΔK	−0.011	−0.008	≤ 0.01	Calculation
Fatty acid gas chromatography (Cis+Trans isomers)				
Miristic acid (C14:0)	0.01 %	0.01 %	≤ 0.05	GC-FID
Palmitic acid (C16:0)	13.11 %	13.52 %	9.4–19.5	GC-FID
Palmitoleic acid (C16:1)	1.38 %	1.39 %	0.6–3.2	GC-FID
Margaric acid (C17:0)	0.04 %	0.04 %	0.07–0.13	GC-FID
Margaroleic acid (C17:1)	0.07 %	0.08 %	0.17–0.24	GC-FID
Stearic acid (C18:0)	2.00 %	2.01 %	1.4–3.0	GC-FID
Oleic acid (C18:1)	76.71 %	76.26 %	63.1–79.7	GC-FID
Linoleic acid (C18:2)	5.13 %	5.14 %	6.6–14.8	GC-FID
Linolenic acid (C18:3)	0.70 %	0.69 %	0.46–0.69	GC-FID
Arachidic acid (C20:0)	0.37 %	0.38 %	≤ 0.60	GC-FID
Gadoleic acid (C20:1)	0.30 %	0.30 %	≤ 0.30	GC-FID
Behenic acid (C22:0)	0.12 %	0.11 %	≤ 0.20	GC-FID
Erucic acid (C22:1)	n.d.	n.d.	≤ 0.30	GC-FID
Lignoceric acid (C24:0)	0.06 %	0.09 %	≤ 0.20	GC-FID
Trans-oleic acid	0.02 %	0.03 %	≤ 0.05	GC-FID
Trans-linoleic + trans-linolenic acids	0.01 %	0.01 %	≤ 0.05	GC-FID
ECN42 (actual and target value difference)	0.05	0.07	≤ 0.20	Calculation
Waxes (C42 +C44 +C46)	49 mg/Kg	89 mg/Kg	≤ 150	GC-FID
Ethyl esters	8 mg/Kg	-	35	GC-FID
Determination of the composition and content of sterols and triterpenic dialcohols				
Cholesterol	0.1 %	0.1 %	≤ 0.50	GC-FID
Brassicasterol	Traces	Traces	≤ 0.10	GC-FID
Campesterol	2.0 %	2.1 %	≤ 4.00	GC-FID
Stigmasterol	1.1 %	1.3 %	< Campesterol	GC-FID
β-sitosterol (apparent) *	95.7 %	95.5 %	≥ 93.0	GC-FID
δ−7-Stigmastenol	0.1 %	0.2 %	≤ 0.5	GC-FID
Absolute sterols	1510 mg/Kg	1732 mg/Kg	≥ 1000	GC-FID
Erythrodiol absolute	37 mg/Kg	151 mg/Kg		GC-FID
Uvaol absolute	2 mg/Kg	5 mg/Kg		GC-FID
Erythrodiol + uvaol absolute	39 mg/Kg	156 mg/Kg		GC-FID
Erythrodiol + uvaol	2.5 %	8.3 %	≤ 4.5 %	GC-FID
Steroid hydrocarbon test (Stigmasta−3,5-diene)	0.01 mg/Kg	0.04 mg/Kg	≤ 0.05	GC-FID

3.3.3. Determination of the composition and content of sterols and triterpenic dialcohols

Moreover, the results of the determination of the composition and content of sterols and triterpenic dialcohols in eOO and SCOO reveal important quality and authenticity characteristics compared to the limits established for EVOO. Starting with sterols, cholesterol (0.1 %) and brassicasterol (in traces) values in both oils largely comply with legal limits (≤ 0.5 % and ≤ 0.1 %, respectively). This confirms the absence of adulterations with seed oils, as these compounds are usually found at higher levels in adulterated products. As for campesterol levels, they are similar between oils (2.0 % in eOO and 2.1 % in SCOO), within the permitted range (≤ 4.0 %). The stigmasterol content is also adequate (1.1 % in eOO and 1.3 % in SCOO) and, as stated in the regulations, is lower than the campesterol content. The β-sitosterol (apparent), 95.7 % in eOO and 95.5 % in SCOO, exceeds the minimum required value (≥ 93.0 %), which supports the purity and quality of the oils. And as for δ-7-Stigmastenol, both oils present low levels of this compound (0.1 % in eOO and 0.2 % in SCOO, ≤ 0.5 %). This is consistent with an extraction process that does not degrade the original compounds. Moreover, absolute sterols are 1510 mg/kg in eOO and 1732 mg/kg in SCOO, both above the required 1000 mg/kg. This reflects a composition rich in bioactive compounds, especially in the SCOO oil, due to a more efficient extraction of unsaponifiable compounds by using supercritical CO_2, as already demonstrated in previous studies.

Regarding to triterpenic dialcohols, the absolute values of erythrodiol and uvaol are higher in SCOO (151 mg/kg and 5 mg/kg, respectively) than in eOO (37 mg/kg and 2 mg/kg). This generates a combined erythrodiol + uvaol content of 156 mg/kg in SCOO, higher than the 39 mg/kg in eOO. The high concentration of erythrodiol and uvaol in OO does not represent a risk of toxicity, yet the legislation for EVOO limits their presence (maximum 4.5 %) (COI,2019). This is because these compounds are extracted more efficiently with solvents, so high levels could indicate adulteration. However, in our study, SCOO shows 8.3 % erythrodiol + uvaol, significantly higher than the 2.5 % found in eOO. This increase is explained by the superior ability of supercritical CO_2 to extract bioactive compounds, ruling out any concerns about toxicity or solvent adulteration.

Finally, the content of steroid hydrocarbons was determined, specifically stigmasta-3,5-diene, which is formed as a product of the dehydration of β-sitosterol during the physical refining of OO (Cert et al., 1994; M. León-Camacho, 2004). Therefore, measuring the concentration of this compound allows detecting the presence of refined oil in virgin oils. Since these oils come from dehydrated olives, a higher concentration of this compound was expected. However, the values obtained were lower than the limit of 0.05 mg/kg established for EVOO, with concentrations of 0.01 mg/kg in eOO and 0.04 mg/kg in SCOO, confirming that both oils meet the quality standards for this category.

Categorizing oils, especially those obtained by methods not covered by legislation such as supercritical fluid extraction (SCOO), requires careful interpretation of the quality and authenticity parameters established by international regulations, such as those of the EU and IOC (COI,2019; EEC, 2022). Especially questionable are extinction coefficients (K_{232} and K_{270}). As a result, since they exceed the established limits, a sensory analysis of eOO could not be performed. However, as mentioned above, this high value may be due to several factors and should be taken with caution. eOO meets the established standards for EVOO in all the parameters analyzed, including sterols, waxes, ethyl esters and steroidal hydrocarbon content such as stigmasta-3,5-diene. Its lipid profile, low content of oxidation compounds and high overall quality qualify it to be categorized as EVOO. Alternatively, since supercritical extraction is not covered by current legislation, SCOO could be classified in a different category or as an innovative product outside the traditional regulatory framework. As an alternative, gourmet or premium VOO recognizing innovative extraction methods, advanced process technologies, sustainability and highlighting unique characteristics such as higher content of bioactive compounds. As a functional or

enriched oil, since its high content in TPC (Chabni et al., 2024), erythrodiol and uvaol (for their bioactive properties), SCOO could be positioned as a functional oil with additional health benefits. In addition to being used as an antioxidant for other oils or even blended with refined OO for marketing as ordinary OO. This positioning as a high-value product could help offset higher production costs. It should also be noted that there is currently no legislation regulating OO derived from dehydrated olives extracted by traditional methods, even though it is commercialized in local markets in North Africa (Chabni et al., 2023a; Guillaume et al., 2018).

Legislation should be updated and upgraded to accurately categorize OOs and consider new environmentally friendly technologies such as supercritical technology, a suitable and valid method for OO extraction. Therefore, development of reliable, accurate and precise analytical methods for each parameter to ensure the quality of OO should be prioritized. Addressing this regulatory gap is essential to raise awareness among consumers and the industrial sector about the existence, unique characteristics, and potential benefits of this oil. Understanding these aspects will be critical for the market acceptance and integration of this innovative product.

4. Conclusions

The combined extraction process, using expeller pressing and extraction with supercritical CO_2 (SC-CO_2), showed a significant improvement in oil recovery, reaching a combined yield of 87 %, value much higher than conventional methods. In addition to the extracted oils, the process generated a defatted flour with potential applications in the food industry, maximizing raw material utilization and eliminating waste. The oils obtained by both press and SC-CO_2, showed a remarkably high concentration of phenolic compounds (1013 and 1855 mg/kg, respectively), compared to commercial extra virgin olive oil (297 mg/kg). These compounds correlated with excellent oxidative stability, positioning these oils as ideal candidates for functional applications and premium markets. However, some quality parameters, such as K_{232} and K_{270}, exceeded the limits established for EVOO, suggesting the need to interpret these results with caution due to the particularities of the extraction method.

The absence of undesirable compounds, such as trans isomers or high levels of waxes, confirms the purity and nutritional quality of the oils obtained. The fatty acid and sterol profiles are representative of a high quality EVOO, supporting the ability of the methods used to preserve the characteristic properties of olive oil. Finally, the results underscore the need to update current regulatory regulations to incorporate innovative and sustainable technologies such as extraction with SC-CO_2. This advance would not only allow these oils to be properly categorized and marketed, but would also increase consumer acceptance by recognizing their unique environmental and health benefits.

Statements and declarations

None.

Author agreement statement

I declare that this manuscript is original, has not been previously published, and is not being considered for publication elsewhere.

I confirm that the manuscript has been read and approved by all named authors and that there are no other persons who meet the criteria for authorship but are not listed. I also confirm that the order of authors listed in the manuscript has been approved by all.

The Corresponding Author is the sole contact for the Editorial process. He/she is responsible for communicating with the other authors about progress, submission of revisions, and final approval of proofs.

CRediT authorship contribution statement

Celia Bañares: Writing – review & editing, Validation. **Assamae Chabni:** Writing – original draft, Validation, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Torres Carlos:** Writing – review & editing, Supervision, Resources, Methodology. **Luis Vázquez:** Methodology.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jfca.2025.108249.

Data availability

Data will be made available on request.

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