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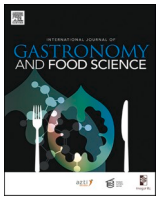

Evaluation of phenolic compounds, antioxidant activity and pigment content in emerging and traditional plant-based oils in Mediterranean gastronomy

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A B S T R A C T

EVOO is an indispensable ingredient in Mediterranean gastronomy that is known for its numerous benefits, such as reductions in cardiovascular risk and its anti-inflammatory effects due to the presence of bioactive compounds and their antioxidant activities. However, recently, other emerging vegetable oils are increasing their culinary presence because of their potential health benefits or interesting attributes for food preparation. The aim of this study was to determine and compare physicochemical parameters, such as the contents of phenolic compounds (TPC), antioxidant capacities, pigments, viscosity and surface tension, of the traditional and emerging plant-based oils used in Mediterranean gastronomy and evaluate the effects of short pan-frying on these parameters. EVOO, olive pomace oil (OPO), sunflower oil (SO), organic flaxseed oil (FO), avocado oil (AO) and extra virgin coconut oil (EVCO) were studied.

The results indicated that the TPC content ranged from 8.92 to 142.27 mg/kg. The highest antioxidant capacities were found in OPO and SO. AO had the highest pigment content.

Frying the oils resulted in greater losses in the TPC contents in EVOO (27.4%) and AO (21.5%), 59.3% of the antioxidant capacity of AO and between 12% and 40% of the pigments in the different oils studied.

Conclusion: EVOO and AO are highlighted for their TPC value, FO and AO for their pigment content and OPO and SO for their antioxidant capacities. In contrast, EVCO had low contents of all the compounds studied. Pigments and physical parameters of these vegetable oils were just lightly affected by short pan-frying, however, the nutritional value provided by its antioxidant capacity and its phenolic compounds was reduced, especially for AO and EVOO.

1. Introduction

Olive oil, especially extra virgin olive oil (EVOO), is widely known to be the main source of fat consumed in the Mediterranean Diet. According to the International Olive Council (IOC), EVOO's world demand has increased notably in recent years, reaching a consumption amount of 3,098 million tons in the years 2020 and 2021 (International Olive Council, 2023). However, palm, soybean, rapeseed, and sunflower oils continue to lead in the ranking of the most-consumed oils worldwide (Statista, 2022). The antioxidant and health benefits of EVOO have been widely described (Jimenez-Lopez et al., 2020;(Yubero-Serrano et al., 2019). Apart from EVOO, there are other oils obtained from olives available on the market, such as olive pomace oil (OPO), which is frequently used as a cheaper culinary alternative to virgin olive oil. Although rich in monounsaturated fatty acids (Table 1), OPO's minor compounds, antioxidant capacity, and health benefits have been less studied in comparison to those of other olive oil derivates (Mateos et al., 2020). Sunflower oil, despite not being especially suitable for frying due to its rich polyunsaturated fatty acid content, which makes it prone to oxidation (Martínez-Pineda et al., 2011), has long been used in the region.

Recently, Mediterranean traditional oils have shared attention with other emerging vegetable oils, such as avocado, coconut, or flaxseed oil, which were not previously consumed in the region. Similarly to EVOO, avocado oil is characterised by a high content of monounsaturated fatty acids (65.29-71.31%), which comprise more than 50% oleic acid and approximately 10% polyunsaturated fatty acids (Krumreich et al., 2018). It also contains a large proportion of biologically active compounds to which numerous beneficial effects in terms of health are attributed, such as reduced cardiometabolic risk, antidiabetic effects, and protection against age-related macular degeneration (Cervantes-paz & Yahia, 2021; Tan, 2019). Additionally, it presents relatively high levels of pigments, such as carotenoids, which also act as antioxidants, and chlorophylls, the presence of which provides a distinctive green colour considered by consumers to be a beneficial attribute and commercial advantage (Woolf et al., 2009). Flax oil is rich in essential fatty

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Table 1
Mean fatty acids profile (g/100g) of traditional and emerging Mediterranean plant-based oils.

	EVOO[a]	OPO[b]	SO[a]	AO[a]	FO[a]	CO[a]
C8:0	–	–	–	–	–	6.8
C10:0	–	–	–	–	0.008	5.39
C12:0	–	–	–	–	0.018	41.8
C14:0	0.01	0.01	–	–	0.077	16.7
C16:0	12.10	17.35	5.90	10.9	5.11	8.64
C16:1	1.03	2.19	–	2.66	0.06	0.016
C18:0	2.59	2.60	4.50	0.66	3.37	2.52
C18:1	67.80	57.97	19.50	67.9	18.3	6.27
C18:2	8.40	18.47	65.70	12.5	14.3	1.68
C18:3	0.65	0.68	0.163	0.96	53.4	0.019
C20:0	0.41	0.52	0.274	–	0.13	0.019
C20:2	–	–	0.05	–	0.03	–
C22:0	0.12	–	0.82	–	0.11	–
C22:1	–	–	0.01	–	0.03	–
C24:0	0.06	–	0.31	–	0.07	0.031
SFA %	15.40	19.52	11.80	11.6	8.98	82.5
MUFA %	69.20	60.37	19.51	70.6	18.4	6.33
PUFA %	9.07	19.15	65.91	13.5	67.8	1.70

EVOO: Extra virgin olive oil; OPO: Olive pomace oil; SO: Sunflower oil; AO: Avocado oil; FO: Flaxseed oil; CO: Cocconut oil.
[a] (USDA, 2023).
[b] (Bouaziz et al., 2010).

acids, primarily omega 3 fatty acids, with levels higher than those in any other vegetable oil. Approximately 73% of the lipid contents in flax oil are polyunsaturated fatty acids, of which 50% correspond to α-linolenic acid (ALA) (Herchi et al., 2014). Flax oil's growing popularity is due to its health benefits, such as the reduced risk of cardiovascular disease and certain types of cancer, reductions in anti-inflammatory activities, and relief from the symptoms of menopause and osteoporosis (Goyal et al., 2014). On the contrary, coconut oil's popularity has increased in recent years because of its oxidation stability, related to its high saturated fatty acid content (Azevedo et al., 2021). Virgin coconut oil comprises more than 90% saturated fatty acids, with medium-chain fatty acids (MCFAs) being the most abundant. Especially important are the lauric and myristic acids that are present in virgin coconut oil (Zeng et al., 2022). MCFAs are associated with energy metabolism regulation and bacterial inhibition (Coelho et al., 2019). However, coconut oil's effects on human health remain unclear (Jayawardena et al., 2021; Wallace, 2019).

The high prices of EVOO and emerging vegetable oils such as avocado, flaxseed, and coconut oils cause them to be used in small amounts for raw consumption or for occasional culinary pan-frying techniques.

The popularity of these emerging oils has increased in recent years; however, there is scarce knowledge about important aspects such as their antioxidant capacities and how they act under different culinary techniques. Changes in oil properties have mainly been studied in deep-fat frying. However, few studies have analysed what occurs during pan frying. Although both culinary techniques imply high temperature, oil degradation is different between pan frying and deep-fat frying. The higher surface-to-volume ratio and exposure to oxygen induce faster oil degradation in pan frying compared to deep-fat frying (Andrikopoulos et al., 2002).

The aim of this work was to compare some relevant physico-chemical parameters among the traditionally consumed Mediterranean plant-based oils (EVOO, sunflower, and olive pomace oils) and emerging consumed oils (avocado, flaxseed, and coconut oils) in raw states and after an occasional domestic heat treatment, like short-time pan frying. Due to their contributions to the organoleptic characteristics and nutritional value, the total phenolic content, antioxidant capacity, peroxide value, free fatty acids, pigments, viscosity, and surface tension were analysed.

2. Materials and methods

2.1. Oil samples

In this research, six different types of plant-based oils were studied: extra virgin olive oil (EVOO) (a mix of the Arbequina, Hojiblanca, Manzanilla, and Picual varieties) (Maeva, Aceites Maeva S.L.U., Granada, Spain), olive pomace oil (OPO) (Carrefour, SA, Spain), refined sunflower oil (SO) (Coosol, Aceites del Sur-Coosur, S.A., Jaen, Spain), organic flaxseed oil (FO) (La Masía, OLEO MASÍA, S.A., Sevilla, Spain), avocado oil (AO) (Ethnos, Pietro Coricelli S.p.a., Spoleto, Italy), and extra virgin coconut oil (EVCO) (La Masía, OLEO MASÍA, S.A., Sevilla, Spain). All of the oils were purchased from a local supermarket, and all experiments were performed at least six months before the oil's expiration date.

2.2. Frying process

The frying process was carried out in duplicate for each type of oil. For this, 100 ml of oil was added to a 14 cm diameter frying pan (Ibili Menaje S.A., Gipuzkoa, Spain). Then, the pan was placed on an induction hob model VIN155 (Electrodomésticos JATA, S.A., Tudela, Spain) and heated at low power. A Type K thermocouple probe connected to a Data Logger Testo 177-T4 and Testo Comfort-Software (Testo SE and Co. KGaA, Titisee-Neustadt, Germany) were used to control the temperature.

Once the oil reached 150 °C, 20 g of 1 × 1 cm stick-cut potatoes was added and fried for 5 min. During the frying process, the power of the induction hob was controlled to minimise temperature fluctuations (Fig. 1).

Immediately after the frying process, the oil was removed and collected in a 100 ml borosilicate glass bottle for analysis.

2.3. Physical properties

The surface tension of the oils was determined by the Du Noüy ring method using a K6 tensiometer (Kruss, Hamburg, DE) at 25°C.

The oils' viscosities were determined using a Brookfield CAP-2000+ rotational cone–plate viscometer equipped with a Peltier thermoelectric controller and CAP-CALC/CAP266Y (v. 3) software (AMETEK Brookfield, Middleboro, MA, USA). Tests were performed at 25°C and with a shear rate of 100 s^{-1}.

2.4. Free fatty acids and peroxide value

The total free fatty acid level was determined by titration according to the standard method AOCS, 2017) and expressed as the percentage of free fatty acids (% FFA).

The peroxide value, expressed as milliequivalent active oxygen per kg of oil (mEq O2 active/kg oil), was determined according to the standard volumetric titration method (ISO, 2012).

2.5. Total phenolic content (TPC)

The phenolic compounds were extracted from the samples according to the method of (Capannesi et al., 2000), where 2.5 mL of hexane and 2.5 mL of methanol were added to 2.5 g of the oil samples, then vigorously mixed with a vortex and centrifuged at 3600g for 5 min (Orto Alresa Digicen 20-R; Madrid, Spain). The extraction was completed with methanol (2.5 mL) and repeated two times. Then, 2.5 mL of Folin–Ciocalteu's reagent and 5 mL of Na_2CO_3 (7,5%, w/v) were added to the methanolic extract solution in a 50 mL volumetric flask. Distilled water was then added until the volume reached 50 mL, and the solutions were left in the dark for 90 min. The solutions were filtered (25 mm diameter filter with 0.45 μm pores) to remove the suspended particles that formed during frying and used for the spectrophotometric

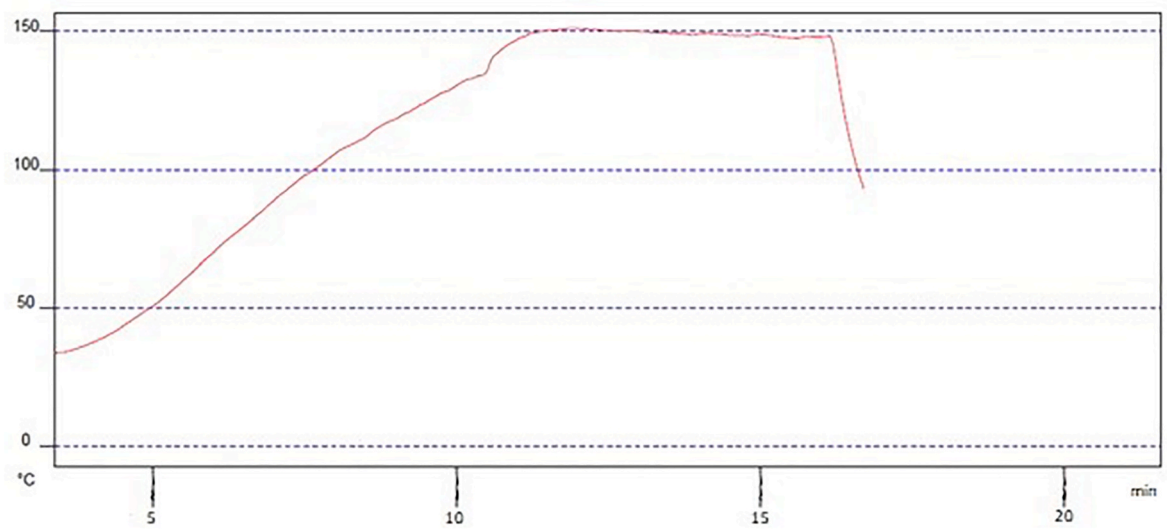

Fig. 1. Oil temperature profile during heating and frying.

determination of the total phenolics at 765 nm using a Jasco UV–Vis spectrometer (Easton, MD, USA/Madrid, Spain). The TPC was expressed as milligrams of caffeic acid equivalents per kilogram of sample (mg CAE/Kg).

2.6. DPPH radical scavenging effect

A spectrophotometric analysis that used DPPH as a stable radical was performed (Tuberoso et al., 2007). Samples (20 μL) were added to a DPPH ethyl acetate solution (3 mL, 0.05 mM). The decrease in absorbance of the resulting solution was measured at 517 nm for 15 min using a Jasco UV–Vis spectrometer (Easton, MD, USA/Madrid, Spain). The difference in absorbance at 15 min was used as a measure of the antioxidant capacity of the sample. A Trolox calibration curve in the range of 0.1–0.4 mM was prepared, and the results were expressed as the Trolox equivalent antioxidant capacity per Kg (mM TEAC/Kg) and as the percentage of inhibition using the following equation:

$$\%Inhibition = \frac{(A_b - A_t)}{A_b} \times 100$$

where A_b refers to the absorbance of a blank sample and A_t refers to the absorbance of an analysed sample.

2.7. Pigment content

The carotenoid and chlorophyll contents were measured spectrophotometrically using the method proposed by (Motilva et al., 1998). The oil samples were dissolved in cyclohexane (O:C) until they reached a final volume of 3 ml and then analysed via spectrophotometry at 472 and 670 nm. The carotenoids were quantified at an absorbance of 472 nm, a measure that corresponds to lutein, which was the largest component of the fraction, while the chlorophylls were measured at an absorbance of 670 nm, which corresponds to pheophytin a, a major constituent.

The concentration of chlorophyll pigments and carotenoids was calculated by applying the following formula:

$$C = \frac{E\ x\ V_f}{E_{1\%}\ x\ W}\ x\ 10000$$

where C refers to the final pigment concentration (mg of chlorophylls or carotenes per Kg of oil), E is the absorbance measured at the corresponding λ, V_f is the final volume of pigment extract (ml), $E_{1\%}$ is the specific absorbance of a 1% solution measured in a 1 cm cuvette ($E_{1\%}$ lutein = 2000 and $E_{1\%}$ pheophytin a = 613), and W is the oil sample's weight (g).

2.8. Statistical analysis

The analyses were conducted in triplicate for each type of oil, both for the raw state and after frying. The measurements obtained for the total phenolic compounds, antioxidant activities, and pigment contents were analysed via one-way ANOVA, followed by a Tukey's post hoc test, and values of $P < 0.05$ were considered significant. An unpaired *t*-test was used to evaluate the effect of frying on each type of oil, with the same threshold for statistical significance. I statistical analyses were performed using GraphPad Prism 5.0 software (GraphPad Software, Inc., San Diego, CA).

3. Results and discussion

3.1. Physical properties

The viscosity and surface tension analysis results of the different oils, raw and after frying, are shown in Table 2. As expected, no significant differences were observed in either parameter before and after one cycle of frying. Changes in the physical properties of an oil or fat have been previously observed, mainly due to products generated during the process and differing in molecular size from triacylglycerols (i.e., oxidation, polymerisation, hydrolysis, and fission products), but only after several repeated frying and heating cycles (Kalogianni et al., 2011).

Table 2
Physical properties (viscosity and surface tension) of oils before and after frying.

	Viscosity (P)		Surface tension (mN/m)	
	Raw	After frying	Raw	After frying
EVOO	0.76 ± 0.02 [a]	0.75 ± 0.02 [a]	37.50 ± 0.50 [a]	37.80 ± 0.29 [a]
OPO	0.75 ± 0.01 [a]	0.75 ± 0.01 [a]	35.70 ± 0.58 [a]	34.50 ± 0.50 [a]
SO	0.61 ± 0.02 [a]	0.63 ± 0.01 [a]	35.70 ± 0.58 [a]	34.50 ± 0.50 [a]
AO	0.70 ± 0.01 [a]	0.71 ± 0.01 [a]	35.70 ± 0.58 [a]	35.80 ± 0.76 [a]
FO	0.49 ± 0.00 [a]	0.50 ± 0.01 [a]	37.00 ± 1.00 [a]	36.00 ± 0.50 [a]
EVCO	0.51 ± 0.00 [a]	0.53 ± 0.01 [a]	34.30 ± 1.15 [a]	34.30 ± 0.58 [a]

EVOO: Extra virgin olive oil; OPO: Olive pomace oil; SO: Sunflower oil; AO: Avocado oil; FO: Flaxseed oil; EVCO: Extra virgin cocconut oil. Mean values ± standard deviations. Letters in each raw indicate significant differences ($p<0.05$) within each type of oil before and after frying process.

3.2. Peroxide value and free fatty acids

The oxidative stability of the oils, expressed as the peroxide value, and the content of free fatty acids are shown in Table 3. The results were similar to those previously reported (Bhatnagar et al., 2009; Giuffrè, et al., 2018; Nasri et al., 2021; Pérez-Saucedo et al., 2021). No significant increases ($p < 0.05$) were observed in the peroxide value or free fatty acids after short-time pan frying. In all samples, both parameters remained under the established limits. According to the Codex Alimentarius, general virgin and cold press oils should not exceed 4.0 mg KOH/g oil of free fatty acids and 15 mEq O_2 active/kg oil. In the case of virgin olive oil and blends of refined olive pomace oil and virgin olive oil, the limits are 1,5% acidity and 20 mEq O2 active/kg oil, respectively (FAO, 2001). European legislation, to the contrary, limits free fatty acids at ≤0,8% in extra virgin olive oil and ≤1% in olive pomace oil, while the peroxide value must be ≤20 or ≤15 mEq O2 active/kg oil, respectively (European Commission, 2022). It is generally accepted that over 20 mEq O2 active/kg oil, a rancid flavour is perceived.

3.3. Total phenolic content (TPC)

The total phenolic content results of the different raw oil analyses are shown in Table 4. All of the samples contained phenolic compounds, with EVOO highlighted for having the highest content (142 mg CAE/Kg), followed by avocado oil (64 mg CAE/Kg). EVCO and SO were the oils with the lowest contents, and there were no significant differences ($p<0.05$) between them. Depending on the food matrix, the oil extraction method was a determining factor in the final TPC. According to the Codex standard for named vegetable oils (2015), virgin oils are those obtained by mechanical procedures, e.g., expelling or pressing, and the application of heat only, while for cold-pressed oils, mechanical procedures are permitted but heating is forbidden. In both cases, purification may have been performed by washing with water, settling, filtering, and centrifuging only (Codex Alimentarius Commision Amended (2015)). If other procedures are applied during oil extraction, such as the use of chemical solvents, the final product is considered to be refined. According to the manufacturer's indications, the flaxseed oil was obtained by first cold-pressing the flaxseed, and it was not refined. The observed TPC was double that previously found by other authors for commercial cold-pressed flaxseed oil (but not for first cold-pressed flaxseed oil). On the contrary, our refined SO showed a similar TPC value to cold-pressed sunflower oil (12 mg CAE/Kg) (Siger et al., 2008). The TPCs found in the EVCO oil samples were close to those found previously in commercial refined, bleached, and deodorised coconut oil (Marina et al., 2009). The cultivar, region, environmental conditions, and whether the extraction procedure included testa, in addition to the endosperm of the coconut kernel, were observed as the determining factors in the coconut oil samples' phenolic compounds (Jayathilaka and Seneviratne, 2022; Robles-Ozuna et al., 2021).

Higher TPCs were expected in the EVCO samples since lower amounts of phenolic compounds are expected in refined oils compared to those that are not refined (Kostadinovic Velickovska and Mitrev, 2013). This fact could be related to the extraction procedure applied. Previous studies have shown that high temperatures used in the hot extraction of virgin coconut oil promote the release of bound phenols and favour the incorporation of more thermally stable phenolic antioxidants in the final coconut oil product (Seneviratne et al., 2009; Zeng et al., 2022). The scarce literature indicated that the TPCs in olive pomace oil are lower than 100 mg/kg (Mateos et al., 2020), which was in accordance with the levels found in this study.

Fig. 2 shows a comparison of the TPCs in each oil before and after the frying process. Some of the raw oils with lower phenolic contents—OPO, SO, and FO—held up better under the effect of frying and did not show significant reductions in their TPCs. Despite retaining the highest phenolic contents, EVOO and AO suffered the greatest reductions in TPC after frying (27.4% and 21.5%, respectively). These results are in accordance with previous results that showed significant reductions in the phenolic compounds in EVOO after a few minutes of frying (Quiles et al., 2002). Other authors have also demonstrated that for EVOO, great amounts of the TPC are lost during initial cycles of frying (Abenoza et al., 2016; Casal et al., 2010). The effects of cooking, particularly frying, on avocado oil have scarcely been studied. There is little evidence available about the changes in avocado oil's TPC after frying (Samaniego-Sánchez, et al., 2021); however, the food matrices and frying parameters differed from those of our study, which could explain the different results obtained.

EVCO's total phenolic content was raised by 6.4 mg CAE/Kg after frying. A phenomenon similar to that observed by Seneviratne et al. (2009) for the hot extraction of coconut oil could explain this result. High temperatures applied during the frying process could release thermally stable phenolic compounds, increasing their accessibility. The observed TPC increase could be also explained by the particular composition of virgin coconut oil. The presence of other compounds, such as amino acids, sugars, or bounded forms, could cause interference during colorimetric Folin–Ciocalteau assay, causing a disparity. This, along with an unusually high TPC, was previously reported in virgin coconut oil by other authors (Jayathilaka and Seneviratne, 2022).

3.4. Pigments

The carotenoid and chlorophyll contents of the oils for both the raw state and after frying are shown in Table 5 and Table 6, respectively. As can be observed, AO presented the highest carotenoid and chlorophyll contents, followed by FO and EVOO. Chlorophylls are the predominant pigments that affect the colour of avocado oil (Wong, et al., 2008), and their concentration and colour variation depend on the methods used to extract the oil (Ashton et al., 2006). The chlorophyll contents reported by some authors for virgin avocado oil extracted by three different methods, including cold-pressing, reached values of 1.23-69.8 mg/kg (Tan, 2019). Despite the fact that the avocado oil analysed in this study did not belong to the category of virgin oils, the results were within that range. Carotenoids are responsible for yellow, red, and orange oil colours, and they also offer antioxidant capacity by inhibiting free radicals.

Table 3
Peroxide value (PV) and free fatty acids (FFA) of oils before and after frying.

	PV (mEq O2 active/kg oil)		FFA (%)	
	Raw	After frying	Raw	After frying
EVOO	13.35 ± 0.59 [a]	13.50 ± 0.71 [a]	0.39 ± 0.01 [a]	0.39 ± 0.00 [a]
OPO	3.66 ± 0.41 [a]	3.94 ± 0.76 [a]	0.11 ± 0.00 [a]	0.11 ± 0.00 [a]
SO	1.98 ± 0.27 [a]	2.56 ± 0.45 [a]	0.30 ± 0.04 [a]	0.32 ± 0.02 [a]
AO	13.66 ± 0.44 [a]	13.71 ± 0.01 [a]	0.83 ± 0.05 [a]	0.90 ± 0.06 [a]
FO	3.08 ± 0.11 [a]	3.46 ± 0.29 [a]	0.32 ± 0.02 [a]	0.36 ± 0.02 [a]
EVCO	0.00 ± 0.00 [a]	0.00 ± 0.00 [a]	0.48 ± 0.02 [a]	0.50 ± 0.04 [a]

EVOO: Extra virgin olive oil; OPO: Olive pomace oil; SO: Sunflower oil; AO: Avocado oil; FO: Flaxseed oil; EVCO: Extra virgin cocconut oil. Mean values ± standard deviations. Letters in each raw indicate significant differences ($p<0.05$) within each type of oil before and after frying process.

Table 4
Total phenolic content (TPC) and antioxidant capacity (AA) in raw oil.

	TPC (mg CAE/Kg)	AA (mM TEAC/Kg)	AA (% Inhibition)
EVOO	142.27 ± 2.75 [a]	1.21 ± 0.26 [a]	25.9 ± 5.0 [a]
OPO	17.68 ± 2.96 [b]	2.87 ± 0.25 [b]	64.2 ± 13.1 [b]
SO	11.60 ± 2.36 [c]	1.97 ± 0.16 [c]	43.5 ± 3.8 [c]
AO	64.33 ± 4.29 [d]	0.83 ± 0.06 [d]	17.1 ± 1.3 [a]
FO	24.49 ± 3.57 [e]	1.40 ± 0.11 [a]	30.9 ± 2.1 [d]
EVCO	8.92 ± 0.66 [c]	0.05 ± 0.02 [e]	4.4 ± 1.9 [e]

EVOO: Extra virgin olive oil; OPO: Olive pomace oil; SO: Sunflower oil; AO: Avocado oil; FO: Flaxseed oil; EVCO: Extra virgin coconut oil. Mean values ± standard deviations. Different letters in each column indicate significant differences ($p<0.05$) between oils.

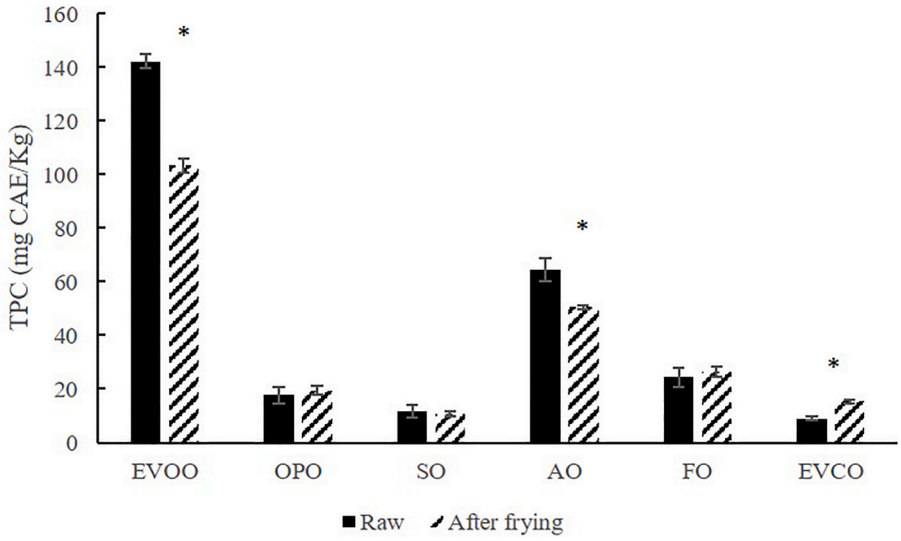

Fig. 2. Total phenolic content (TPC) before and after frying. * indicates significant differences ($p<0.05$) in each type of oil before and after frying.

Table 5
Oil carotenoids content (mg lutein/Kg oil) before and after frying.

	Raw	After frying	p_{fry}
EVOO	1.88 ± 0.06 [a]	1.41 ± 0.09 [a]	<0.0001
OPO	0.38 ± 0.02 [b]	0.25 ± 0.02 [b]	<0.0001
SO	0.00 ± 0.00 [c]	0.00 ± 0.00 [c]	–
AO	6.00 ± 0.79 [d]	3.58 ± 0.31 [d]	<0.0001
FO	4.38 ± 0.26 [e]	3.74 ± 0.45 [e]	0.0007
EVCO	0.00 ± 0.00 [c]	0.00 ± 0.00 [c]	–

EVOO: Extra virgin olive oil; OPO: Olive pomace oil; SO: Sunflower oil; AO: Avocado oil; FO: Flaxseed oil; EVCO: Extra virgin coconut oil. Mean values ± standard deviations. Letters in each column indicate significant differences ($p<0.05$) between oils. *p* column indicates significant differences ($p<0.05$) within each type of oil before and after frying process.

Table 6
Oil chlorophyll content (mg pheophytin a/Kg oil) before and after frying.

	Raw	After frying	p_{fry}
EVOO	1.68 ± 0.22 [a]	1.43 ± 0.23 [a]	0.0832
OPO	0.00 ± 0.00 [b]	0.00 ± 0.00 [b]	–
SO	0.00 ± 0.00 [b]	0.00 ± 0.00 [b]	–
AO	15.89 ± 2.08 [c]	13.98 ± 1.32 [c]	0.0924
FO	0.49 ± 0.20 [d]	0.36 ± 0.61 [d]	0.5021
EVCO	0.00 ± 0.00 [b]	0.00 ± 0.00 [b]	–

EVOO: Extra virgin olive oil; OPO: Olive pomace oil; SO: Sunflower oil; AO: Avocado oil; FO: Flaxseed oil; EVCO: Extra virgin coconut oil. Mean values ± standard deviations. Letters in each column indicate significant differences ($p<0.05$) between oils. *p* column indicates significant differences ($p<0.05$) within each type of oil before and after frying process.

The results for the raw AO were within the range previously reported for virgin avocado oil, ranging from 3.08 to 75 mg of β-carotene/kg (Tan, 2019).

The total pigment amounts found in this study for the raw multivarietal EVOO were lower than those previously reported for Arbequina, Hojiblanca, and Picual monovarietal types of EVOO, which ranged between 1.9 and 9.3 mg/kg for lutein and 4.5 and 21.7 mg/kg for pheophytin a (Gandul-Rojas and Minguez-Mosquera, 1996; Gandul-Rojas et al., 1999). These differences could be associated with the olives' ripeness stages, as both pigment contents decrease markedly during ripening, with the losses nearing 98% for chlorophyll and 82% for carotenoids (Gutiérrez et al., 1999). In addition to the olive variety, the growing area is another critical factor in the final pigment content (Criado et al., 2004). The proportion of each variety of olive oil used in the creation of this multivarietal EVOO was not indicated in its labelling; however, the chlorophyll/carotenoid ratio observed (0.90) indicated that the Arbequina variety was predominant. Previous studies have shown a chlorophyll/carotenoid ratio of 1.09 for Arbequina EVOO, ratios of between 1.08 and 1.28 for Picual varieties, and a ratio of 1.28 for the Hojiblanca variety (Gandul-Rojas and Minguez-Mosquera, 1996).

A previous study analysed the pigments present in 15 samples of cold-pressed linseed oils, concluding that their carotenoid contents ranged from 18.34 to 67.97 mg/kg, while their chlorophyll contents ranged from 0.06 to 3.93 mg/kg (Symoniuk et al., 2017). According to our results, the chlorophyll contents were within this range, but the carotenoid contents were four times lower.

Both SO and EVCO stand out for having an absence of these components. In SO extraction, during the bleaching stage of the oil refining process, the SO undergoes a series of physicochemical reactions, the objectives of which are to reduce the pigments and the consequent discoloration of the oils (Gharby, 2022). The EVCO pigment results were as expected as it is a colourless vegetable oil.

A significant reduction in the carotenoid contents of the AO, EVOO, and FO samples was observed after the frying process, while no significant changes were noted for their chlorophyll contents. It was previously demonstrated that carotenoids are more susceptible to degradation under heating conditions than chlorophylls (Resende et al., 2019). The short-time frying treatment applied in this study and chlorophyll's higher stability could explain these results.

Regarding carotenoids, previous studies have observed that the thermal degradation of lutein, β-carotene, and β-cryptoxanthin follows a first-order kinetic mechanism. Competitive and parallel reactions occur with lutein in olive oil, which yields reactions of isomerisation and subsequent degradation to a colourless product (Aparicio-Ruiz et al., 2011).

3.5. DPPH radical scavenging effect

The antioxidant activities of the different raw oils analysed are shown in Table 4. Although EVOO and AO presented the highest TPCs, their resulting antioxidant properties were lower than those of the other oils. It is known that the antioxidant properties of phenolic substances vary significantly depending on their functional groups (Bayram et al., 2012; Rice-Evans et al., 1996), which could explain these results. The OPO and SO samples, despite being or containing refined oils, presented the highest values. We must consider the presence of other common

components in plant-based oils, such as tocopherols and carotenoids, that also possess antioxidant capacities. Additionally, it should be noted that, in accordance with European legislation, during the production process of plant-based refined oils, such as seed oils or olive pomace oil, the external addition of these antioxidant compounds or even of additives with this function is allowed to preserve the oil's properties, increase its stability, and avoid rancidity due to a high unsaturated fatty acid content (European Parliament and of the Council, 2011). The DPPH radical scavenging demonstrated by the EVOO non-monovarietal sample in the present study was 1.21 mM TEAC/Kg, while that of other monovarietal EVOO samples reported previously ranged between 0.20 and 2.72 mM TEAC/Kg (Dugo et al., 2020; Samaniego Sánchez et al., 2007). We also considered that because the OPO samples were a blend of refined and extra virgin oils, the antioxidant capacity observed could be due not only to the antioxidant agents from the virgin olive oil but also to the contribution of other agents or ingredients, such as antioxidant additives, used during the refining process of refined olive pomace (but not in the OPO final product).

Previous studies on cold-pressed oils indicated radical scavenging capacity values of 1.58 mM TEAC/Kg for flaxseed oil and 0.58 mM TEAC/Kg for avocado oil (Prescha et al., 2014), and these results are similar to those determined in the present study. In contrast, the percentage of inhibition of free radical DPPH for cold-pressed virgin avocado oils in other studies was much lower (0.61%) (Tan, 2019) than that determined in this study (17.1%).

The EVCO samples, despite being extra virgin, showed barely any radical scavenging ability. Similar results were reported by Bhatnagar et al. (2009) for raw coconut oil. This could have been related to the fact that a low total phenolic content was detected, as were low levels of other antioxidant molecules such as carotenoids. A strong correlation between the total phenolic content and scavenging capacity of virgin coconut oil was previously reported (Marina et al., 2009).

The effect of frying on antioxidant capacity is shown in Fig. 3. Frying significantly reduced the antioxidant capacity. The highest reduction was observed in avocado oil (59.3%), followed by OPO (37.9%) and EVOO (33.1%). It was previously reported that these oils are rich in tocopherols. Avocado oil's α-tocopherol contents range between 32.4 and 45.0 mg/kg, while EVOO and SO contain between 5 and 300 mg/kg and between 44 and 1200 mg/kg, respectively (Gil Hernández, Artacho Martín-Lagos, & Ruiz López, 2017; Manaf et al., 2018). Tocopherols belong to a class of phenolic antioxidants that can inhibit lipid autoxidation by scavenging free radicals and by reacting with singlet oxygen; after processing, approximately 60-70% of the tocopherols remain in the oil (Frankel, 1989). However, α- and δ-tocopherols lose their antioxidant capacity when cooking temperatures reach 150 °C (Reblova, 2006). As this was the frying temperature used, it could explain the high antioxidant capacity loss observed in the studied oils.

Regarding minor compounds, OPO is highlighted for its content of tocopherols, especially α-tocopherol, aliphatic and triterpenic alcohols, and squalene (Holgado et al., 2021). A previous study found that the most relevant losses after frying were in the tocopherol and squalene contents in OPO (Ruiz-Méndez et al., 2021). The rapid degradation of α-tocopherols, the reductions in antioxidant activity at high temperatures (Barrera-Arellano et al., 2002), and the oxidation-prone nature of squalene (due to its high number of double bonds) (Shimizu et al., 2019) were exposed as the main causes, and these could also explain the results of our study.

Previous studies also showed a marked decrease in tocopherol and polyphenol contents in EVOO after frying (Olivero-David et al., 2014), as well as a reduction in its antioxidant capacity after exposure to different heating conditions (Giuffrè et al., 2018).

It should be noted that along with tocopherols and other molecules with antioxidant activities, the evidence also indicated that the total phenolic compounds play an important role in an oil's stability during frying, as they have the ability to inhibit the oxidation of oil under frying conditions (Wu et al., 2019). Consequently, their reduction during the frying process might have strongly contributed to the antioxidant capacity loss.

It should be noted that the loss of antioxidant capacity is greater when using a frying pan because there is greater oxygen absorption per unit of oil with a thin layer of oil, as compared to that with a larger amount of oil, when frying with a pan (Kobyliński et al., 2016).

4. Conclusions

In their raw states, EVOO and avocado oil showed the highest contents of total phenolic compounds. However, there were relevant losses observed after short-time pan frying.

Among the traditional Mediterranean oils, EVOO presented the best characteristics for raw consumption. However, the sunflower oil samples best maintained their antioxidant properties after frying. Olive pomace oil, because of its lower price compared to other olive oils and its relatively high antioxidant capacity, represents a relevant alternative.

Flaxseed oil could be considered an interesting oil since it presented high carotenoid contents and its relevant antioxidant capacity was not affected by short-time pan frying.

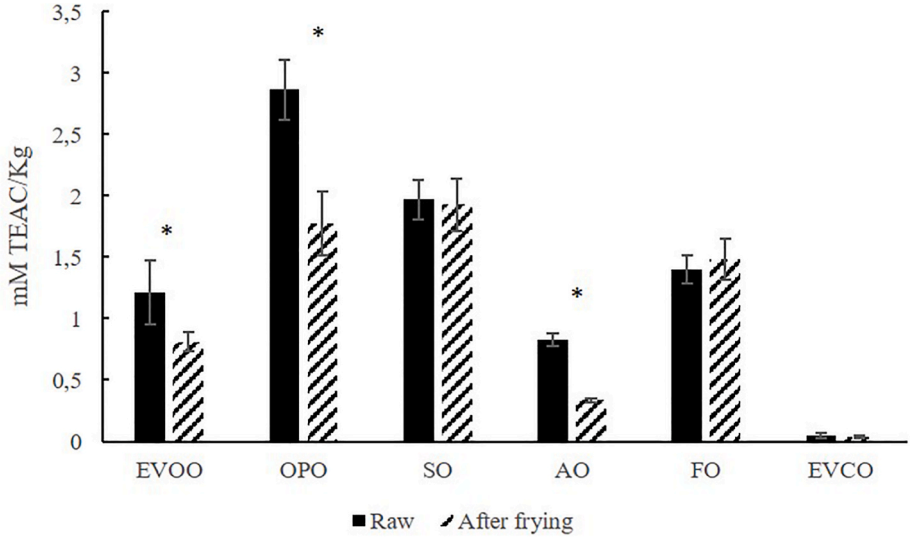

Fig. 3. Antioxidant capacity before and after frying. * indicates significant differences ($p<0.05$) in each type of oil before and after frying.

Despite being extra virgin and stable because of its high saturated fatty acid content, coconut oil did not stand out for its contribution of antioxidant agents. It showed low contents for all of the analysed parameters.

It was observed that after short-time pan frying, the pigments and physical parameters of these vegetable oils were lightly affected; however, the nutritional value provided by their antioxidant capacity and phenolic compounds was reduced. According to the obtained results, among the traditional Mediterranean oils, extra virgin olive oil could be considered the most interesting oil because it retained the highest TPC after short-time pan frying; among the emerging oils, flaxseed oil properly maintained its nutritional value.

The present study outlines a general view of the impact of short-time pan frying on some of the nutritional and organoleptic properties of the oils. However, further studies are necessary to elucidate specific changes in the complete phenolic profile.

Implications for gastronomy

In the Mediterranean area, olive oils and some seed oils, such as sunflower oil, have traditionally been the most common oils used in culinary applications. However, in recent years, new emerging vegetables oils, such as avocado, flaxseed and coconut oils, are trending in the region. Their use has acquired great relevance due to the positive effects on health that are attributed to them. These oils are usually directly consumed in a raw state. Nevertheless, there is scarce information about their behaviors during culinary processes. This research attempts to provide knowledge about the changes caused by short pan-frying to some valuable molecules such as total phenolic compounds and pigments, as well their interesting parameters such as the antioxidant capacities of these emerging oils compared to traditional oils.

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Author statement

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Declaration of competing interest

The authors declare no conflict of interest.

Data availability

Data will be made available on request.

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