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Enrichment of refined sunflower oil with phenolic extracts from argan (*Argania spinosa* L. (Skeels)) co-products using ultrasound-assisted extraction

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ABSTRACT

Vegetable oils, rich in unsaturated fatty acids, are highly susceptible to oxidation, requiring antioxidants to ensure stability. In this study, phenolic-rich extracts from argan (*Argania spinosa L.* (Skeels)) co-products were obtained by ultrasound-assisted extraction (UAE) and incorporated into refined sunflower oil (100 and 200 ppm). The oxidative stability of enriched oils was compared to a non-enriched control and vitamin E-supplemented oils. Chemical characterization confirmed that argan co-products extracts contained high levels of phenolics and flavonoids with strong antioxidant activity. Quality indices (peroxide value, acidity, p-anisidine, K_{232}, K_{270}, and fatty acid profile) showed no significant changes between enriched and control oils, indicating that the ultrasound enrichment process did not compromise oil quality. Rancimat analysis demonstrated a marked improvement in oxidative stability, particularly for pulp extracts at 200 ppm, which yielded the longest induction period compared to both controls. These findings highlight the potential of argan co-products as sustainable sources of natural antioxidants, offering an efficient and safe alternative to synthetic stabilizers in edible oils.

1. Introduction

Vegetable oils are essential to the diet because they provide energy and important micronutrients, but their high content of unsaturated fatty acids makes them extremely sensitive to oxidation [1,2,3]. The oxidative degradation process leads to rancidity and unpleasant flavors, and decreases nutritional value, reducing commercial appeal and creating safety issues for consumers [4,5]. The food industry uses synthetic antioxidants like BHT, BHA and TBHQ to protect products from oxidation [6]. The use of these compounds for protective purposes has raised health concerns, prompting the market to seek natural antioxidant alternatives [7,8,9]. The interest in plant-derived phenolic compounds grows because they possess strong antioxidant capabilities and consumers tend to favor them [10,11,12]. Modern extraction methods enable the recovery of bioactive compounds from agro-industrial waste materials which brings economic advantages and environmental sustainability ** [13,14,15,16,17,18,19]. Ultrasound-assisted extraction (UAE) is recognized as a sustainable technique, offering high extraction yields at relatively low temperatures while reducing solvent consumption and preserving extract quality [16,20,21]. Research indicates that adding bioactive compounds extracted by ultrasound to sunflower oil creates a product with

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enhanced antioxidant properties and longer shelf life without compromising its taste or aroma [22,23,24,25,19]. The argan tree (*Argania spinosa* L. Skeels) native to southwestern Morocco produces valuable oil but its co-products such as leaves, pulp and press cake contain unexploited bioactive compounds, especially phenolic and flavonoid compounds with antioxidant properties [26,27,28,29]. Despite increasing interest in the valorization of these co-products, little is known about their direct application in stabilizing edible oils. To the best of our knowledge, no study has yet examined the enrichment of refined sunflower oil with argan co-product extracts obtained by UAE. The objective of this study was therefore to characterize the biochemical composition of argan co-products, extract their phenolic-rich fractions using UAE, and evaluate their potential to enhance the oxidative stability of refined sunflower oil. For comparison, vitamin E was used as a positive control. Oxidative stability was assessed through Rancimat analysis and quality indices in order to determine the effectiveness and suitability of argan co-product extracts as natural antioxidants in edible oils.

2. Material and methods

2.1. Raw materials

The plant material employed in this study includes argan leaves, pulp and press cake. The leaves were collected from Argan trees (*Argania spinosa L.* (Skeels)) during January 2024 in the Taroudant region. The pulp and press cake were supplied by the Tiout women's cooperative, also located in the Taroudant region. The various materials were dried and then ground to obtain uniform powders. The powders obtained were subjected to various analyses, to determine ash, moisture, protein and fibre contents, as well as mineral profile, oil yield, total phenolic compound content and antioxidant capacity. The vegetable oil used in this research was refined sunflower oil from "Souss Belhassan oil mills" company (Agadir, Morocco), purchased from a supermarket in Agadir. This edible oil has a free fatty acid level of 0.07 ± 0.03 g/100 g, and has a clear, pale-yellow color. The oil was preserved at 4 ± 1°C before being enriched, while the co-products were stored in a dark, dry place.

2.2. Proximate composition

The proximate composition of argan co-products (leaves, pulp, and press cake) was determined using standard analytical procedures. Moisture content was obtained by drying the samples at 103 °C until constant weight and calculating the loss in mass [30]. Lipid content was quantified by Soxhlet extraction with n-hexane for 8 h [31], while ash content was determined after incineration at 525 °C for 4 h in a muffle furnace. For mineral analysis, ash samples were digested with 4 mL of 65 % HNO_3 and 10 mL of HCl, and the resulting solutions were analyzed using inductively coupled plasma optical emission spectrometry (ICP-OES) [32,33]. Protein content was determined by nitrogen quantification with a LECO FP628 analyzer, applying the conventional conversion factor of 6.25 [32,33]. Fiber fractions, including crude fiber (CF), neutral detergent fiber (NDF), acid detergent fiber (ADF), and acid detergent lignin (ADL), were measured following the procedure of Van Soest et al., [34] with a FIWE 6 fiber extractor. Carbohydrate content was obtained by difference, calculated as 100 – (lipids + proteins + ash + moisture) [35]. The energy value was then estimated from the protein, lipid, and carbohydrate fractions and expressed in kcal per 100 g of dry matter, according to Bouzid et al. [35].

2.3. Ultrasound assisted extraction of bioactive compounds

Bioactive compounds were extracted using an ultrasonic probe system (Biobase Ultrasonic Cell Disruptor) equipped with a 6 mm cylindrical sonotrode. Sonication was performed at 50 Hz, with the power adjusted to 70 % of the device's maximum capacity (650 W). After setting the solvent/solid ratio, each sample, consisting of 50 g of powder, was combined with 500 ml of ethanol, used as the extraction solvent, in a beaker placed in an ice bath to maintain a temperature of 20 ± 2 °C. The extraction parameters were defined as follows: time 20 min, amplitude 70 %, temperature limit 25 °C, pulse on 1 s and pulse off 1 s. The obtained extracts were isolated by centrifugation at 3500 rpm for 15 min, followed by filtration using a Whatman filter, the solvent was subsequently removed with a rotary evaporator at 40 °C under reduced pressure until fully evaporated. The extract from each sample, which was semi-solid or viscous in nature depending on the quantity of compounds extracted, was then preserved at −4 °C until enrichment process.

2.4. Co-products bioactive compounds and antioxidant activity

2.4.1. Total phenolics content (TPC)

The total polyphenol content (TPC) was quantified through spectrophotometric analysis using the Folin–Ciocalteu assay as describe by Khatoon et al. [36] with some modification. In this procedure, 0.5 mL of the extract, diluted in a 1:10 ratio with distilled water, was combined with 2.5 mL of Folin–Ciocalteu reagent (also diluted 1:10 with distilled water) and 2.5 mL of a 7.5 % Na_2CO_3 aqueous solution in a 10 mL glass tube. This combination was then incubated at 45 °C for 30 min. Measurements of absorbance were carried out at 765 nm with a SCILOGEX SP-UV1100 spectrophotometer. A calibration curve was established for gallic acid and the TPC was reported in mg of gallic acid equivalent per g of dry weight.

2.4.2. Total flavonoids content (TFC)

The overall amount of flavonoids present within the extracts was quantified as per the method of Bijla et al. [2] with minor modifications. To summarize, 1 mL of the diluted extract was added to a 10 mL volumetric flask containing 0.3 mL of 5 % $NaNO_2$. A 10 % $AlCl_3$ solution of 0.3 ml was introduced after 5 min. After waiting for another 6 min, 2 mL of 1 M NaOH was added and made up to 10 mL using distilled water. The mixture was stirred and allowed to react for 30 min. The SCILOGEX SP-UV1100 was used to record the absorbance at 415 nm. Quercetin was employed to prepare the standard curve. Based on the regression equation, TFC was obtained in mg quercetin equivalents per g of dry weight (mg QE/g DW).

2.4.3. Condensed Tanins (CT)

The method of vanillin [35] was used to quantify the condensed tannins. In simple terms, 2 mL of freshly prepared methanol solution (1 % w/v) of vanillin was added to 2 mL of a 25 % v/v H_2SO_4 solution in methanol. Following this, the combination was added with the diluted sample extract (0.5 mL). The solution is stirred in the dark for 15 min to develop a red color. Using a SCILOGEX SP-UV1100 UV–Vis spectrophotometer, we recorded the absorbance at 500 nm. Results were measured against a control consisting of vanillin, sulphuric acid and methanol. The amount of CT was reported as mg of catechin equivalents per g of dry weight (mg CE/g DW).

2.4.4. In vitro total antioxidant activity

Three complementary assay procedures were done to determine the in vitro antioxidant properties of co-products of argan oil. Ferric Reducing Antioxidant Power (FRAP) assay [37], ABTS + radical cation decolorization assay [2] and DPPH (2,2-diphényl-1-picrylhydrazyle) radical scavenging assay [38]. Absorbance readings of FRAP, ABTS and DPPH were taken at 700 nm, 734 nm and 517 nm respectively with SCILOGEX SP-UV1100 UV– visible spectrophotometer. According to FRAP and ABTS data, provided as milligram of ascorbic acid equivalents (AAE) and trolox equivalents (TE)/gram of sample, respectively, and DPPH data as percentage of DPPH inhibited, calculated using formula (1):

$$\text{Inhibition of DPPH \%} = \frac{Ac - As}{\text{Ac}} \times 100 \quad (1)$$

"where 'Ac' denotes the absorbance of the control (a 1.2 mM DPPH solution in methanol) and 'As' signifies the absorbance of the sample."

2.5. Enrichement of sunflower oil

To assess the effect of enriching sunflower oil with phenolic extracts derived from argan co-products. Two concentrations of each extract (100 and 200 ppm) were partially dissolved in sunflower oil using a stirrer, then mixed vigorously using an ultrasonic probe system (Biobase Ultrasonic Cell Disirupter) with a 6 mm diameter cylindrical sonotrode, under the following conditions: time 15 min, amplitude 70 %, temperature limit 25 °C, pulse on 1 s and pulse off 1 s to complete total enrichment. Vitamin E served as a positive control at the same concentrations. After enrichment process, the different samples of enriched oil and negative control oil (non-enriched oil) were then separated into 30 ml clear glass bottles and kept at 4 °C for subsequent analysis. The negative control oil was a refined sunflower oil purchased from a supermarket in Agadir, and according to the manufacturer, it had not been previously enriched with any synthetic antioxidants. The important indicators of quality of the sunflower oil sample were evaluated according to the standard parameters. The free fatty acids (FFA), peroxide value (PV), p-anisidine value (p-AV), total oxidation value (TOTOX), pigment content (chlorophyll and carotenoids), oxidation potential (COX) and conjugated dienes (CD) and trienes (CT). Also, composition of fats and iodine value (IV) determine the degree of unsaturation.

2.6. Oil physico-chemical indices

2.6.1. Free fatty acids (FFA) value

The quantification of free fatty acids was carried out in accordance with ISO660, [39]. Briefly, 10 g of each oil sample was combined with 50 mL of neutral ethanol. The mixture was titrated against 0.1 N sodium hydroxide, with phenolphthalein serving as the endpoint indicator. Results were expressed as the percentage by weight of oleic acid relative to the total oil mass.

2.6.2. Peroxide value (PV)

The peroxide value was determined following the ISO3960, [40] protocol. For each test, 5 g of oil was dissolved in a 3:2 mixture of acetic acid and chloroform. Potassium iodide, added in excess, initiated the reaction in darkness for five minutes. The liberated iodine was titrated with 0.01 N sodium thiosulfate, using starch as an indicator. The values obtained were expressed in milliequivalents of active oxygen per kilogram of oil.

2.6.3. P-anisidine value (p-AV)

The p-anisidine value was measured according to ISO6885, [41]. Two grams of oil were diluted in 25 mL of isooctane to prepare solution A. Its absorbance was recorded at 350 nm against an isooctane blank. Subsequently, 5 mL of this solution was treated with 1 mL of p-anisidine reagent (0.25 % in glacial acetic acid), and absorbance was measured again after 10 min of incubation. The p-AV was then calculated using the standard equation:

$$\boldsymbol{p-\text{AV}} = \frac{25 \times (1.2 \times (\boldsymbol{A_s} - \boldsymbol{A_b}))}{\mathbf{m}} \quad (2)$$

Where, p-AV represents the p-anisidine value. A_s denotes the absorbance of the oil solution after its reaction with p-anisidine, while A_b refers to the absorbance of the unreacted oil solution. m corresponds to the mass of the oil sample in g. The number 25 signifies the volume of isooctane used to dissolve the oil sample, and the factor 1.2 serves as a correction to account for the dilution effect caused by the addition of the p-anisidine solution.

2.6.4. Cojugated Diens (CD) and conjugated Triens (CT) values

Conjugated dienes and trienes are present, according to the K_{270} and K_{232} values, respectively, were measured using a method outlined in ISO3656, [42]. These values correspond to the specific extinction coefficients of a 1 % (w/v) solution created by mixing cyclohexane with sunflower oil. The absorbance of this solution was recorded using a Shimadzu UV-1800 UV–Vis spectrophotometer, fitted with a 1 cm path length cuvette. The measurements were taken at specific wavelengths: 232 nm for conjugated dienes (K_{232}) and 270 nm for conjugated trienes (K_{270}).

2.6.5. Chlorophyll and carotenoid content

Pigment quantification was performed following the procedure of Nid Ahmed et al. [13,18]. For each sample, 7.5 g of oil was transferred to a 25 mL volumetric flask and diluted to the mark with cyclohexane. Absorbance was measured at 670 nm and 470 nm using a SCILOGEX SP-UV1100 spectrophotometer to determine chlorophyll and carotenoid contents, respectively. Calculations were performed using the equations provided by Gharby et al. [43]:

$$\textbf{Chlorophyll content}\,(\boldsymbol{mg/kg}) = \frac{(\boldsymbol{A}_{670} \times 10^6)}{(613 \times 100 \times \mathbf{L})} \quad (3)$$

$$\textbf{Carotenoid content}\,(\boldsymbol{mg/kg}) = \frac{(\boldsymbol{A}_{470} \times 10^6)}{(2000 \times 100 \times \mathbf{L})} \quad (4)$$

2.6.6. Fatty acids (FAs) composition

The fatty acid composition of the oils was analyzed by gas chromatography, following the ISO 12966-2, 2017 [44] protocol. Prior to injection, fatty acids were converted into their methyl esters. Approximately 0.1 g of oil was weighed into a vial, to which 2 mL of isooctane and 0.1 mL of 2 N methanolic potassium hydroxide was added. The mixture was shaken vigorously for one minute and left to stand for two minutes. Thereafter, 2 mL of 40 % sodium chloride solution was introduced to facilitate phase separation. The upper organic phase, containing the fatty acid methyl esters, was collected and dried over anhydrous sodium sulfate. Analysis was carried out using an Agilent HP chromatograph equipped with a DB-23 AG-TRANS capillary column (60.0 m × 320 μm × 0.25 μm) and a flame ionization detector. Helium was employed as the carrier gas, with a flow rate of 20 mL/min. The injector, detector, and oven temperatures were set at 260 °C. Fatty acid levels were expressed as the relative peak area percentages, in accordance with the method described by Gharby et al. [45,].

2.6.7. Oxidizability value (COX)

The susceptibility of the oil to oxidation, expressed as the coefficient of oxidizability (COX), was calculated using a theoretical equation derived from its fatty acid composition. The formula applied, presented as equation (5), was originally proposed by Abril et al. [46]:

$$\textbf{COX} = \frac{1 \times (\textbf{C16:1} + \textbf{C17:1} + \textbf{C18:1} + \textbf{C20:1}) + 10.3 \times (\textbf{C18:2}) + 21.6 \times (\textbf{C18:3} + \textbf{C20:3})}{100} \quad (5)$$

2.6.8. Total oxidation value (TOTOX)

The total oxidation (TOTOX) value offers a comprehensive indicator of the oxidative state of an oil, reflecting both primary and secondary oxidation products. It is calculated theoretically by combining the peroxide value and the p-anisidine value, as shown in equation (6).

$$\mathbf{TOTOX} = 2 \times \mathbf{PV} + \boldsymbol{p} - \mathbf{AV} \quad (6)$$

2.6.9. Saponification value

The saponification value (SV) was estimated from the fatty acid composition using the equation (7) presented below. The calculation began with determining the molecular weight fraction of each fatty acid, obtained by multiplying its relative proportion (expressed as a decimal) by its molecular weight. The mean molecular weight of the sample was then derived by summing the fractional contributions of all identified fatty acids [47].

$$\boldsymbol{SV}(\boldsymbol{mgKOHpergofoil}) = 3 \times \boldsymbol{n} \times 56 \times 1000 \quad (7)$$

With

$$\boldsymbol{n} = \frac{1}{\boldsymbol{M_{TG}}} \text{ and } .\boldsymbol{M_{TG}} = (\boldsymbol{meanmolecularweight} \times 3) + 92.09 - (3 \times 18)$$

2.6.10. Iodine value (IV)

The iodine value (IV) is a key parameter for evaluating the degree of unsaturation in vegetable oils. It reflects the grams of iodine that can react with 100 g of oil, with higher values indicating a greater proportion of unsaturated fatty acids. The IV was calculated from the relative abundance of these unsaturated components, according to equation (8) [48]:

$$\mathbf{IV} = (\%\boldsymbol{C}16:1 \times \boldsymbol{C_{O1}}) + (\%\boldsymbol{C}18:1 \times \boldsymbol{C_{O2}}) + (\%\boldsymbol{C}18:2 \times \boldsymbol{C_{O3}}) + (\%\boldsymbol{C}18:3 \times \boldsymbol{C_{O4}}) \quad (8)$$

Where, C16:1 represents palmitoleic acid, C18:1 refers to oleic acid, C18:2 corresponds to linoleic acid, and C18:3 denotes linolenic acid. The iodine value coefficients for these respective fatty acids are CO1 (1.001), CO2 (0.899), CO3 (1.814), and CO4 (2.737).

2.7. Rancimat test

The oxidative stability (OS) of the oil samples was assessed by the Rancimat test, in accordance with the ISO standard [49]. This method measures OS as the oxidative induction time (hours). Briefly, 3 g of each oil sample (enriched or native) were placed in the reaction vessel of a Rancimat instrument (Metrohm, Herisau, Switzerland). The vessel was then heated at different temperatures (383 K and 393 K) with an airflow rate of 20 L/h. The volatile compounds generated during oil oxidation were captured in a flask containing 60 mL of distilled water. The conductivity of this solution, which increases with oxidation, was continuously measured and recorded by the Rancimat software. The OS of each oil sample was calculated using the resulting conductivity curves using the software's extrapolation method. This method estimates the induction time at typical storage temperature at room temperature 25 °C (298 °K). Separate induction times were measured for the oil samples at the two elevated test temperatures (383 K and 393 K).

2.8. Data statistical analysis

Data are expressed as mean values ± standard deviation (SD), with n = 3 replicates per treatment. Statistical differences between mean values were evaluated using Tukey's test at a significance level of $\alpha = 0.05$. All statistical analyses were conducted using Origin Pro 2024 (OriginLab Corporation, Northampton, MA, USA).

Table 1
Proximate composition, fibers content, total phenolic content (TPC), total flavonoids content (TFC), condensed tannins (CT) and antioxidant activities of the argan co-product studied.

	Leaves	Pulp	Press Cake
Ash (g/100 g)	4.26 ± 0.14^{b}	4.89 ± 0.01 ^{a}	3.69 ± 0.16^{c}
Moisture (g/100 g)	8.23 ± 0.14^{b}	10.23 ± 0.62 ^{a}	6.27 ± 0.11^{c}
Protein (g/100 g)	15.53 ± 0.40^{b}	9.28 ± 0.13^{c}	46.84 ± 1.37 ^{a}
Oil (g/100 g)	–	8.47 ± 0.30^{b}	15.9 ± 0.4 ^{a}
Carbohydrates (g/100 g)	71.98 ± 0.96 ^{a}	67.13 ± 0.57^{b}	27.30 ± 0.37^{c}
EV (Kcal)	–	378.10 ± 0.34 ^{a}	370.47 ± 4.31^{b}
CF (%)	10.30 ± 0.05^{b}	12.70 ± 0.06 ^{a}	9.00 ± 0.04^{c}
NDF (%)	16.50 ± 0.50^{c}	19.20 ± 0.6^{b}	38.00 ± 0.75 ^{a}
ADF (%)	14.09 ± 0.09^{c}	15.18 ± 0.17^{b}	27.30 ± 0.35 ^{a}
ADL (%)	11.59 ± 0.04 ^{a}	10.39 ± 0.02^{b}	6.20 ± 0.02^{c}
TPC (mg GAE/g DW)	24.05 ± 1.53 ^{a}	10.14 ± 0.57^{b}	2.28 ± 0.06^{c}
TFC (mg QE/g DW)	44.01 ± 1.29 ^{a}	19.50 ± 3.54^{c}	33.04 ± 4.24^{b}
CT (mg CE/g DW)	28.51 ± 0.95^{b}	54.19 ± 0.19 ^{a}	5.54 ± 0.15^{c}
FRAP (mg AAE/g DW)	16.18 ± 2.53 ^{a}	13.54 ± 0.38 ^{a}	3.71 ± 0.73^{b}
ABTS (mg TE/g DW)	20.09 ± 2.55 ^{a}	9.23 ± 0.45^{b}	8.38 ± 0.76^{b}
DPPH (%)	51.16 ± 1.64 ^{a}	7.18 ± 0.33^{b}	5.09 ± 2.62^{b}

EV: Energy Value; CF: Crude Fiber; NDF: Neutral Detergent Fiber; ADF: Acid Detergent Fiber; ADL: Acid Detergent Lignin; TPC: Total Phenolic Content; TFC: Total Flavonoid Content; CT: Condensed Tannins; FRAP: Ferric Reducing Antioxidant Power; ABTS: ABTS radical scavenging capacity; DPPH: DPPH radical scavenging capacity. Values are presented as mean ± standard deviation. Different superscript letters indicate significant differences between samples ($P < 0.05$), as determined by one-way ANOVA followed by Tukey's test.

3. Results and discussion

3.1. Proximate composition, phenolic compounds and antioxidant activity

The proximate composition of argan co-products (leaves, pulp, and press cake) revealed marked differences in their nutritional profiles (Table 1). Press cake was distinguished by its high protein content (46.84 g/100 g) and residual oil (15.9 g/100 g), in agreement with previous reports highlighting argan press cake as a valuable protein-rich by-product [50,30]. Leaves contained the highest proportion of carbohydrates (71.98 g/100 g) and lignocellulosic fibers, comparable to other plant by-products such as olive and palm leaves, which are commonly investigated for biomass or food additive applications [51,52]. Pulp displayed an intermediate composition with moderate levels of proteins (9.28 g/100 g), lipids (8.47 g/100 g), and carbohydrates (8.47 g/100 g), in line with earlier analyses of argan fruit pulp [53,54,55].

The analysis of bioactive compounds showed that leaves were the richest source of phenolics (24.05 mg GAE/g dw) and flavonoids (44.01 mg QE/g dw). These values are higher than those reported by Fahmi et al., [56], but lower than those obtained with methanolic extractions [57,27], highlighting the influence of solvent choice. Pulp exhibited the highest condensed tannin content (54.19 mg CE/g dw), however our findings for the pulp are lower than those reported by other authors [26,58,59], since they used methanol as the solvent for extraction. Press cake, despite its protein richness, contained relatively low levels of phenolics and tannins.

The antioxidant activity followed similar trends. Leaves displayed the strongest activity across DPPH, ABTS, and FRAP assays, in agreement with their high phenolic and flavonoid levels. Pulp extracts showed intermediate activities, while press cake presented the lowest antioxidant performance. These results confirm the positive correlation between phenolic content and antioxidant capacity, as widely reported [60,61]. Nevertheless, the contribution of other compounds, such as chlorophylls, or carotenoids, cannot be excluded and may act synergistically with phenolics. However, these findings demonstrate that argan co-products, especially leaves and pulp, are rich sources of bioactive compounds with antioxidant potential. Their composition and activity are comparable to other agro-industrial by-products valorized for similar purposes, reinforcing their interest as natural additives for

Table 2
Mean concentrations of mineral elements (mg/kg dry weight).

	Leaves	Pulp	Press Cake
Ca	7535.48 ± 17.24^{b}	2526.09 ± 5.89^{c}	20887.65 ± 162.70 ^{a}
Na	1258.44 ± 3.39 ^{a}	1059.84 ± 1.52^{b}	203.51 ± 1.08^{c}
K	11001.32 ± 99.83^{b}	20592.25 ± 109.56 ^{a}	5174.71 ± 16.88^{c}
Mg	3113.55 ± 3.29^{b}	695.60 ± 2.01^{c}	4308.31 ± 22.87 ^{a}
Fe	192.21 ± 0.04 ^{a}	91.68 ± 0.32^{c}	121.69 ± 0.96^{b}
Mn	17.22 ± 0.07^{b}	4.59 ± 0.01^{c}	70.43 ± 0.35 ^{a}
Cu	2.77 ± 0.01 ^{a}	1.45 ± 0.01^{b}	1.45 ± 0.04^{b}
Zn	9.88 ± 0.19^{b}	2.79 ± 0.03^{c}	18.15 ± 0.16 ^{a}
B	32.62 ± 0.10^{b}	11.67 ± 0.08^{b}	327.38 ± 5.93 ^{a}
P	798.03 ± 4.50^{b}	497.64 ± 0.01^{c}	1273.52 ± 0.76 ^{a}

All values are reported as means ± standard deviation based on two replicates. Different letters in the same column signify statistically significant differences ($p < 0.05$).

stabilizing edible oils [13,18,62–64].

3.2. Mineral composition of co-products of argan

The mineral profile of argan co-products (Table 2) showed considerable variation between leaves, pulp, and press cake. Pulp was particularly rich in potassium (20592.25 mg/kg), while press cake contained the highest concentrations of calcium (20887.65 mg/kg), magnesium (4308.31 mg/kg), and zinc (18.15 mg/kg). Leaves exhibited intermediate to high levels of most minerals, with a predominance of potassium (11001.32 mg/kg) and calcium (7535.48 mg/kg), along with notable amounts of sodium (1258.44 mg/kg) and iron (192.21 mg/kg). These significant differences observed between samples for each element highlight the heterogeneity of mineral distribution in the various argan co-products, which was also observed in a previous study on the mineral composition of argan co-products [32,33]. Generally, the mineral composition complements the proximate and phytochemical profiles, confirming that argan co-products are not only sources of bioactive phenolics but also valuable reservoirs of essential macro- and microelements.

3.3. Principal component analysis

Principal Component Analysis (PCA) was applied to visualize the relationships between the compositional and antioxidant parameters of argan co-products (Fig. 1). The first two principal components explained

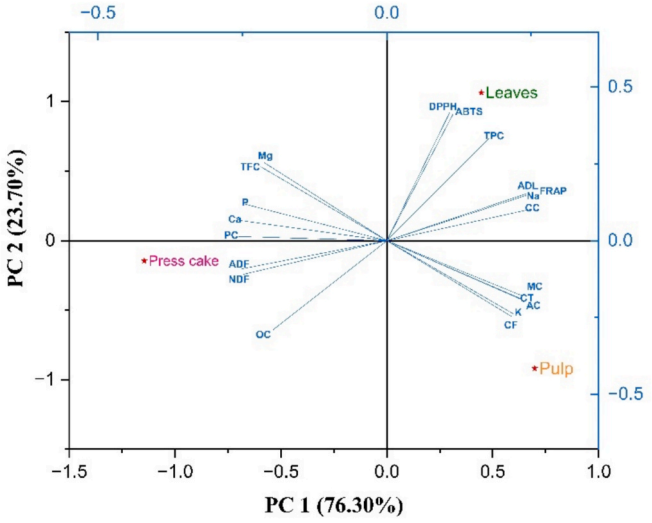


Fig. 1. Principal component analysis (PCA) of the argan co-products studied with regard to proximal composition, mineral elements and antioxidant activity.

100 % of the total variance, with PC1 alone accounting for 76.3 %. Variables such as total phenolic content (TPC), flavonoids, DPPH, ABTS, and FRAP were strongly and positively correlated with PC1, clearly separating the leaves from the other co-products. This confirms that leaves are the richest in phenolics and exhibit the highest antioxidant activity. Pulp was positioned closer to the positive side of PC1 but was mainly influenced by condensed tannins, ash, moisture, potassium, and fiber content. In contrast, press cake clustered on the negative side of PC1, reflecting its distinctive composition characterized by higher protein, residual oil, and detergent-insoluble fibers (NDF, ADF). These findings highlight the biochemical complementarity of the different co-products. Leaves emerged as the primary reservoir of antioxidant compounds, while the pulp was distinguished by its high tannin and potassium content, and the press cake by its richness in proteins and residual oil. Comparable clustering patterns of plant by-products, based on polyphenol and nutrient profiles, have been observed in PCA studies of olive leaves, spent coffee grounds, and various oilseed cakes [31,48,62,65,66]. Collectively, the PCA highlights the compositional diversity of argan co-products and supports their targeted valorization: leaves for antioxidant applications, pulp for tannin exploitation, and press cake for nutritional and feed purposes.

3.4. Quality indices

3.4.1. Free faty acids content

The content of free fatty acids (FFAs) serves as an indicator of oil quality, reflecting its susceptibility to rancidity [67]. Free fatty acids (FFAs) are primarily generated through the action of lipase enzymes, which hydrolysis triacylglycerols to produce diacylglycerols, monoacylglycerols and FFAs [68]. In this study, free acidity was determined in order to assess the impact of adding phenolic extracts from argan co-products on the quality of refined sunflower oil. The results show that the free fatty acid (FFA) content of the control sunflower oil (SC) was 0.07 ± 0.03 g/100 g. Oils enriched with argan co-product extracts displayed values ranging from 0.04 to 0.07 g/100 g, with no significant differences compared to the control ($P > 0.05$) (Table 3). These results indicate that the ultrasound enrichment process did not promote hydrolysis of triacylglycerols and therefore preserved the initial quality of the oil. Comparable findings have been reported for linseed oil enriched with carotenoids via ultrasound-assisted extraction, where no significant changes in acidity were observed [69]. Similarly, Sousa et al. [70] showed that the addition of natural flavorings to olive oil did not alter FFA levels. In contrast, some studies have noted increased acidity when natural additives were incorporated, such as thyme in argan oil [71], highlighting the importance of the matrix–extract interaction. However, for refined edible oils, the Codex Alimentarius sets the maximum free fatty acid content, expressed as acid value, at 0.6 mg KOH per gram of oil, equivalent to 0.3 g/100 g FFA [72]. In this study, the values obtained were clearly below this regulatory threshold, confirming the chemical stability of argan co-product extracts during ultrasonic enrichment. Among the treatments, pulp extracts at 100 and 200 ppm tended to yield slightly lower acidity values, although differences were not statistically significant. This observation suggests a possible stabilizing effect of pulp phenolics on the hydrolytic stability of sunflower oil.

3.4.2. Peroxide value (PV)

The peroxide value (PV) is a basic and crucial parameter used to evaluate the oxidative state of vegetable oils [73]. It enables the determination of the concentration of primary products, which are formed in the early stages of oxidation [48]. Table 3 presents the peroxide value of supplemented and non-supplemented sunflower oils. The peroxide value (PV) of the control sunflower oil was 0.68 ± 0.08 mEq O_2/kg, while the oils enriched with pulp and press cake extracts maintained low PVs, particularly SEP-200 (0.53 ± 0.19 mEq O_2/kg) and SEC-100 (0.32 ± 0.10 mEq O_2/kg), indicating effective protection against primary oxidation. In contrast, oils enriched with leaf extracts showed

Table 3
Mean values of quality indices results of the enriched sunflower oil by argan co-products extract and by vitamin E; Free fatty acids (FFA), Peroxide values (PV), *p*-anasidine values (*p*-AV), Carotenoids and Chlorophylls Content, Conjugated dienes (CD) and Conjugated trienes (CT).

	FFA (g/100 g)	P V (mEq O_2/kg)	*p*-AV	Carotenoids (mg/kg)	Chlorophylls (mg/kg)	CD (K_{232})	CT (K_{270})
SC	0.07 ± 0.03 ^{a}	0.68 ± 0.08^{b}	7.75 ± 0.01^{b}	0.12 ± 0.01^{b}	0.27 ± 0.01^{c}	3.04 ± 0.04 ^{a}	2.43 ± 0.15 ^{ab}
SEL-100	0.06 ± 0.02 ^{a}	3.08 ± 0.53 ^{a}	9.70 ± 1.30 ^{a}	0.12 ± 0.01^{b}	0.72 ± 0.05 ^{ab}	3.06 ± 0.01 ^{a}	2.82 ± 0.09 ^{a}
SEL-200	0.05 ± 0.01 ^{a}	2.36 ± 0.06 ^{a}	9.08 ± 0.21 ^{ab}	0.19 ± 0.01 ^{a}	0.86 ± 0.01 ^{a}	2.91 ± 0.01 ^{a}	2.81 ± 0.05 ^{a}
SEP-100	0.04 ± 0.01 ^{a}	0.82 ± 0.27^{b}	8.67 ± 0.12 ^{ab}	0.13 ± 0.01 ^{ab}	0.20 ± 0.03^{c}	2.87 ± 0.04 ^{a}	2.68 ± 0.12 ^{a}
SEP-200	0.04 ± 0.01 ^{a}	0.53 ± 0.19^{b}	7.32 ± 0.27^{b}	0.08 ± 0.01 ^{bc}	0.33 ± 0.06^{c}	2.86 ± 0.04 ^{a}	2.67 ± 0.13 ^{a}
SEC-100	0.05 ± 0.01 ^{a}	0.32 ± 0.10^{b}	8.66 ± 0.05 ^{ab}	0.05 ± 0.01^{c}	0.37 ± 0.15 ^{bc}	2.94 ± 0.04 ^{a}	2.82 ± 0.25 ^{a}
SEC-200	0.07 ± 0.02 ^{a}	0.50 ± 0.17^{b}	8.34 ± 0.24 ^{ab}	0.04 ± 0.01^{c}	0.48 ± 0.15 ^{bc}	3.00 ± 0.03 ^{a}	2.67 ± 0.16 ^{a}
SEV-100	0.07 ± 0.03 ^{a}	0.43 ± 0.01^{b}	7.84 ± 0.47 ^{ab}	0.05 ± 0.01^{c}	0.25 ± 0.08^{c}	3.06 ± 0.10 ^{a}	2.88 ± 0.15 ^{a}
SEV-200	0.04 ± 0.00 ^{a}	0.36 ± 0.01^{b}	9.14 ± 0.27 ^{ab}	0.09 ± 0.01^{bc}	0.51 ± 0.15 ^{abc}	2.96 ± 0.01^{a}	1.93 ± 0.05^{b}

Unenriched sunflower oil served as a negative control, whereas vitamin E was utilized as a positive control. Values are expressed as mean ± standard deviation (±SD), and mean values within the same column that are followed by different superscript letters indicate significant differences ($P < 0.05$, Tukey's test). SC: Negative Control, SEL-100: Sunflower enriched with leaves extract 100 ppm, SEL-200: Sunflower enriched with leaves extract 200 ppm, SEP-100: Sunflower enriched with pulp extract 100 ppm, SEP-200: Sunflower enriched with pulp extract 200 ppm, SEC-100: Sunflower enriched with cake extract 100 ppm, SEC-200: Sunflower enriched with cake extract 200 ppm, SEV-100: Sunflower enriched with Vitamin E 100 ppm, SEV-200: Sunflower enriched with Vitamin E 200 ppm.

significantly higher PVs, reaching 3.08 ± 0.53 mEq O_2/kg at 100 ppm and 2.36 ± 0.06 mEq O_2/kg at 200 ppm, suggesting that leaf phenolics were less efficient in inhibiting hydroperoxide formation. These values remain below the Codex Alimentarius limit for refined oils (10 mEq O_2/kg), confirming the overall oxidative quality of all samples [72]. Similar trends have been observed with numerous natural extracts that have been tested on several vegetable oils and have been found to offer better protection [15,74,75]. This trend suggests that enrichment with natural argan co-products extracts offers a natural option for stabilizing oils against primary oxidation while maintaining efficacy comparable to that of vitamin E. However, the increase in PV with leaf extracts may be related to the presence of chlorophylls, known to act as pro-oxidants [76].

3.4.3. *Para-anisidine value (p-AV)*

The *p*-anisidine value (*p*-AV) is a parameter used to evaluate the extent of secondary oxidation in oils [45,77]. Evaluating this parameter in parallel with the PV is very useful, particularly as the PV is typically effective for evaluating oxidation in its early stages, due to the reactivity of unstable products that convert spontaneously into secondary peroxides. Thus, the *p*-AV provides further information, offering a complete insight into the overall oxidation state of vegetable oils [78,79]. As shown in Table 3, the *p*-anisidine value of the control sunflower oil was measured at 7.75 ± 0.01. However, most enriched samples showed slightly higher values, ranging between 8.3 and 9.7, while the pulp extract at 200 ppm (7.32 ± 0.27) remained close to the control. The highest p-AV was recorded for SEL-100 (9.70 ± 1.30), indicating that leaf extracts were less effective at preventing the formation of aldehydes and other secondary compounds. All values, however, remained below the commonly accepted threshold of 10 for edible oils [47], which suggests that none of the treatments led to deterioration incompatible with oil quality standards. Nevertheless, similar increases in p-AV have been described when olive mill by-products extracts were incorporated into vegetable oils [80]. Conversely, natural antioxidant extracts α- and δ-tocopherol have been shown to reduce secondary oxidation during storage [81].

3.4.4. *Conjugated dienes (CD) and conjugated trienes (CT) values*

Conjugated dienes (CD) and conjugated trienes (CT) are widely recognized as markers of primary and secondary oxidation in oils [82]. The specific extinction values (K) at 232 and 270 nm provide insight into these changes, as they correspond to the formation of CD and CT resulting from the oxidation of polyunsaturated fatty acids [83]. Spectrophotometric measurements at these wavelengths thus offer an indirect means of assessing the extent of oxidation: absorbance at 232 nm indicates the accumulation of primary oxidation products, whereas absorbance at 270 nm reflects the presence of secondary compounds such as short-chain fatty acids, ketones, and aldehydes [19]. In the present study, the control sunflower oil exhibited CD and CT values of 3.04 ± 0.04 and 2.43 ± 0.15, respectively (Table 3). Oils enriched with argan co-product extracts or vitamin E showed values in the same range, with no significant differences compared to the control ($P > 0.05$). The only exception was SEV-200, which recorded a slightly lower CT value (1.93), suggesting a stronger inhibition of secondary oxidation by vitamin E at this concentration. These findings are consistent with earlier reports showing that the incorporation of natural extracts, such as saffron stigma, prickly broom or sesame, does not markedly modify K_{232} and K_{270} at the initial stage of enrichment [14,84,85]. The stability of CD and CT values across most treatments indicates that argan co-product extracts do not negatively affect primary or secondary oxidation markers at the baseline. In summary, the conjugated diene and triene data support the conclusion that enrichment with argan extracts preserves the oxidative status of refined sunflower oil during the early phases of oxidation. The slightly superior performance of vitamin E at 200 ppm on CT values reinforces its role as a benchmark antioxidant, but the comparable outcomes obtained with argan pulp and press cake extracts highlight their potential as natural alternatives.

3.4.5. *Chlorophylls and carotenoids content*

Among other factors, the quality of vegetable oils is determined by their pigment content, such as chlorophylls and carotenoids. Given their strong antioxidant activity, these pigments influence the oxidative stability of oils by preventing auto-oxidation [15]. The findings of chlorophyll and carotenoid content are presented in Table 3. The control sunflower oil contained 0.27 ± 0.01 mg/kg of chlorophylls and 0.12 ± 0.01 mg/kg of carotenoids. Enrichment with leaf extracts markedly increased chlorophyll levels, reaching 0.72 ± 0.05 mg/kg at 100 ppm and 0.86 ± 0.01 mg/kg at 200 ppm, while pulp and press cake extracts produced only moderate increases. Vitamin E supplementation had little effect on pigment content, as expected. Regarding carotenoids, leaf extracts at 200 ppm induced a slight increase (0.19 ± 0.01 mg/kg), whereas pulp and cake extracts generally yielded lower values than the control. A significant reduction was observed with cake extracts, particularly at 200 ppm (0.04 ± 0.01 mg/kg). Our results regarding chlorophyll and carotenoid contents in enriched sunflower oils show both similarities and discrepancies with previous studies. For chlorophylls, the observed increase in leaf-enriched oils agrees with earlier reports on pigment-rich extracts [19,86]. However, it contrasts with Nid Ahmed et al. [13,18], who reported variable effects of vitamin E on chlorophyll levels in supplemented sunflower oils. Concerning carotenoids, our findings are consistent with Corbu et al. [87], who observed higher carotenoid content in oils enriched with by-product extracts rich in these compounds. These outcomes emphasize the complex interplay between plant extracts and bioactive molecules in edible oils, and

highlight that variability in pigment transfer largely depends on the type of extract and its concentration. Overall, while leaf extracts contribute more pigments to the oil, this may be a double-edged effect, enhancing nutritional value but potentially increasing susceptibility to oxidation.

3.4.6. Total oxidation (TOTOX)

Total Oxidation (TOTOX) values was determined to assess the global oxidative stability of refined sunflower oil and to quantify both primary and secondary oxidation compounds [88]. A more complete indicator of oil Oxidative state is the Total Oxidation Index, which integrates the PV with the *p*-AV to measure the oxidation products. Lower TOTOX values indicate greater oxidative stability and better oil quality, with a value below 10 generally considered to be an indicator of high quality oil [89]. In the present study, the TOTOX value of control sunflower oil was 9.09 ± 0.16 (Table 4). Enrichment with pulp extract at 200 ppm yielded the lowest value (8.38 ± 0.64), indicating superior protection against oxidative deterioration. By contrast, leaf-enriched oils showed significantly higher values, reaching 15.85 ± 2.35 at 100 ppm, suggesting weaker antioxidant performance. Other treatments, including press cake and vitamin E, resulted in intermediate values, generally close to the control. These findings align with previous studies showing that natural extracts can reduce TOTOX values when their phenolic profile is suited to lipid stabilization [62,64,90,91]. The elevated TOTOX values in leaf-enriched oils likely reflect the combined effects of higher PV and p-AV, possibly influenced by chlorophyll content acting as a pro-oxidant [92,93].

3.4.7. Oxidizability (COX) and oxidative susceptibility (OS) values

Vegetable oils' oxidative stability primarily relates to their FAs content, especially their unsaturation level, and the antioxidants they contain [94]. The COX value was determined based on the percentage of UFAs. It serves as an indicator of an oil stability and oxidation susceptibility indicator; oils that are stable typically exhibit lower oxidation values [95]. Our findings for the COX values of sunflower oils, whether enriched or not, ranged from 5.87 ± 0.01 (SEP-100) to 6.03 ± 0.01 (SEC-100) (Table 4). These values are lower than those reported for other polyunsaturated oils such as soybean (6.89), grape seed (7.3), or wheat germ oil (7.8) [95], confirming that sunflower oil is relatively more stable than these highly unsaturated matrices. Among the enriched samples, pulp extract at 100 ppm (SEP-100) conferred the lowest COX value, suggesting a favorable balance between its fatty acid profile and its phenolic composition. In contrast, press cake extracts (SEC-100) were associated with higher COX and OS values, reflecting their greater degree of unsaturation and consequently higher oxidation susceptibility. Comparable findings were reported by Athanasiadis et al. [81], who observed no significant differences in COX among sunflower oils supplemented with various antioxidants, underlining that antioxidant enrichment does not directly modify theoretical oxidizability. Nevertheless, when comparing OS values, sunflower oil appeared substantially more sensitive to oxidation than olive oil (453–922) [96], which is

Table 4
Main fatty acids composition, iodine value, COX value, oxidative susceptibility value, saponification value and TOTOX value of sunflower oil enriched by argan co-products extracts and by vitamine E.

	SC	SEL-100	SEL-200	SEP-100	SEP-200	SEC-100	SEC-200	SEV-100	SEV-200	Norm [72]
C14:0	0.08 ± 0.01 ^{a}	0.08 ± 0.01 ^{a}	0.08 ± 0.01 ^{a}	0.08 ± 0.01 ^{a}	0.07 ± 0.01 ^{a}	0.09 ± 0.01 ^{a}	0.10 ± 0.01 ^{a}	0.01 ± 0.01 ^{a}	0.08 ± 0.01 ^{a}	ND −0.2
C16:0	6.91 ± 0.10^{b}	6.60 ± 0.10^{c}	6.74 ± 0.10 ^{bc}	6.86 ± 0.10 ^{bc}	6.65 ± 0.10 ^{bc}	6.10 ± 0.10 ^{d}	7.49 ± 0.10 ^{a}	6.64 ± 0.10^{c}	6.75 ± 0.10 ^{bc}	5.0–7.6
C16:1	0.14 ± 0.01^{c}	0.09 ± 0.01^{b}	0.10 ± 0.01 ^{ab}	0.10 ± 0.01 ^{ab}	0.10 ± 0.01 ^{ab}	0.1 ± 0.01 ^{ab}	0.12 ± 0.01 ^{ab}	0.12 ± 0.01 ^{ab}	0.15 ± 0.01 ^{a}	ND −0.2
C18:0	3.94 ± 0.10 ^{bc}	3.97 ± 0.10 ^{bc}	3.84 ± 0.10^{c}	4.05 ± 0.10 ^{ab}	3.90 ± 0.10 ^{bc}	3.88 ± 0.10 ^{bc}	3.79 ± 0.10^{c}	3.94 ± 0.10 ^{bc}	4.24 ± 0.10 ^{a}	2.7–6.5
C18:1	34.44 ± 0.10^{c}	34.72 ± 0.10^{b}	34.14 ± 0.10 ^{d}	34.97 ± 0.10 ^{a}	34.48 ± 0.10^{c}	34.73 ± 0.10^{b}	34.15 ± 0.10 ^{d}	34.43 ± 0.10^{c}	34.23 ± 0.10 ^{d}	14.0–39.4
C18:2	53.77 ± 0.10 ^{e}	53.81 ± 0.10 ^{de}	54.40 ± 0.10^{b}	53.45 ± 0.10^{f}	54.17 ± 0.10^{c}	54.63 ± 0.10 ^{a}	53.98 ± 0.10 ^{cd}	54.13 ± 0.10^{c}	53.82 ± 0.10 ^{de}	48.3–74.0
C18:3	0.11 ± 0.01 ^{bc}	0.12 ± 0.01^{b}	0.12 ± 0.01^{b}	0.06 ± 0.01^{c}	0.12 ± 0.01^{b}	0.27 ± 0.01 ^{a}	0.12 ± 0.01^{b}	0.11 ± 0.01 ^{bc}	0.16 ± 0.01^{b}	ND-0.3
C20:0	0.31 ± 0.01 ^{a}	0.30 ± 0.01 ^{ab}	0.28 ± 0.01 ^{ab}	0.11 ± 0.01^{c}	0.30 ± 0.01 ^{ab}	0.15 ± 0.01^{c}	0.25 ± 0.01^{b}	0.3 ± 0.01 ^{ab}	0.30 ± 0.01 ^{ab}	0.1–0.5
C20:1	0.29 ± 0.01 ^{a}	0.30 ± 0.01 ^{a}	0.29 ± 0.01 ^{a}	0.31 ± 0.01 ^{a}	0.30 ± 0.01 ^{a}	0.13 ± 0.01^{b}	0.10 ± 0.01^{b}	0.29 ± 0.01 ^{a}	0.27 ± 0.01 ^{a}	ND-0.3
SFA	11.24 ± 0.10^{c}	11.25 ± 0.10^{c}	11.23 ± 0.10 ^{cd}	11.41 ± 0.10^{b}	11.22 ± 0.10 ^{cd}	10.35 ± 0.10 ^{e}	11.63 ± 0.10 ^{a}	11.18 ± 0.10 ^{d}	11.64 ± 0.10 ^{a}	8–15
MUFA	34.87 ± 0.10 ^{ab}	34.81 ± 0.10 ^{bc}	34.24 ± 0.10^{f}	35.07 ± 0.10 ^{a}	34.48 ± 0.10 ^{cde}	34.73 ± 0.10 ^{bcd}	34.27 ± 0.10^{f}	34.55 ± 0.10 ^{de}	34.38 ± 0.10 ^{ef}	14–39
PUFA	53.88 ± 0.10 ^{e}	53.93 ± 0.10 ^{e}	54.52 ± 0.10^{b}	53.51 ± 0.10^{f}	54.29 ± 0.10 ^{bc}	54.90 ± 0.10 ^{a}	54.10 ± 0.10 ^{cde}	54.24 ± 0.10 ^{cd}	53.98 ± 0.10 ^{de}	48–74
UFA	88.75 ± 0.10^{b}	88.74 ± 0.10^{b}	88.76 ± 0.10^{b}	88.58 ± 0.10^{b}	88.77 ± 0.10^{b}	89.63 ± 0.10 ^{a}	88.37 ± 0.10^{b}	88.79 ± 0.10^{b}	88.36 ± 0.10^{b}	62–113
UFA/ SFA	7.90 ± 0.07^{b}	7.89 ± 0.07^{b}	7.90 ± 0.07^{b}	7.76 ± 0.07^{b}	7.91 ± 0.07^{b}	8.66 ± 0.08 ^{a}	7.60 ± 0.07^{b}	7.94 ± 0.07^{b}	7.59 ± 0.07^{b}	7.53–7.75
IV	128.82 ± 0.08^{b}	129.23 ± 0.07 ^{ab}	129.80 ± 0.32 ^{ab}	128.61 ± 0.02^{b}	129.69 ± 0.09 ^{ab}	131.16 ± 0.09 ^{a}	129.07 ± 0.09 ^{ab}	129.57 ± 0.09 ^{ab}	128.99 ± 0.09^{b}	118–141
COX	5.91 ± 0.01 ^{e}	5.92 ± 0.01 ^{de}	5.97 ± 0.02 ^{d}	5.87 ± 0.01^{f}	5.95 ± 0.01 ^{bc}	6.03 ± 0.01 ^{a}	5.93 ± 0.01 ^{cde}	5.95 ± 0.01 ^{bcd}	5.92 ± 0.01^{de}	–
OS	2465.52 ± 2.06 ^{d}	2467.81 ± 1.43^{d}	2494.24 ± 7.79^{b}	2446.32 ± 2.06 ^{e}	2484.27 ± 1.87 ^{bc}	2520.08 ± 2.06 ^{a}	2475.37 ± 2.06 ^{cd}	2481.40 ± 2.06^{c}	2472.28 ± 2.06 ^{cd}	–
SV	191.52 ± 0.81 ^{a}	191.46 ± 0.81 ^{a}	191.50 ± 0.81 ^{a}	191.54 ± 0.81 ^{a}	191.29 ± 0.81 ^{a}	191.29 ± 0.81 ^{a}	191.47 ± 0.81 ^{a}	191.49 ± 0.81 ^{a}	191.48 ± 0.81 ^{a}	188–194
TOTOX	9.09 ± 0.16^{b}	15.85 ± 2.35 ^{a}	13.80 ± 0.32 ^{a}	10.30 ± 0.65^{b}	8.38 ± 0.64^{b}	9.31 ± 0.15^{b}	9.34 ± 0.10^{b}	8.70 ± 0.45^{b}	9.87 ± 0.27^{b}	–

SFA: Saturated Fatty Acids; MUFA: Monounsaturated Fatty Acids; PUFA: Polyunsaturated Fatty Acids; UFAs: Unsaturated Fatty Acids. Mean values ± standard deviations. Different letters are significantly different ($P < 0.05$) values with same letters indicate no significant difference ($P > 0.05$). SC: Negative Control, SEL-100: Sunflower enriched with leaves extract 100 ppm, SEL-200: Sunflower enriched with leaves extract 200 ppm, SEP-100: Sunflower enriched with pulp extract 100 ppm, SEP-200: Sunflower enriched with pulp extract 200 ppm, SEC-100: Sunflower enriched with cake extract 100 ppm, SEC-200: Sunflower enriched with cake extract 200 ppm, SEV-100: Sunflower enriched with Vitamine E 100 ppm, SEV-200: Sunflower enriched with Vitamin E 200 ppm.

consistent with its higher polyunsaturated fatty acid content. However, the combined evaluation of COX and OS indicates that pulp-enriched oils (SEP-100) show the best theoretical resistance to oxidation, while press cake enrichment increases susceptibility. These results highlight that the effectiveness of co-product extracts in stabilizing oils depends not only on their antioxidant compounds but also on the fatty acid composition of the enriched matrix.

3.4.8. Iodine value (IV)

The iodine value (IV) is used to measure the degree of unsaturation in the fatty acids that compose triacylglycerols [97]. It is recognized that FAs with two double bonds may oxidize 10–40 times more rapidly than those with one double bond [98]. As indicated in Table 4, the control sunflower oil showed an IV of 128.82 ± 0.08 g I_2/100 g. Oils enriched with argan extracts generally displayed slightly higher IVs, with the exception of pulp-enriched oil at 100 ppm (SEP-100), which remained close to the control (128.61 ± 0.02 g I_2/100 g). This similarity is consistent with the superior oxidative stability of SEP-100 observed in other assays. By contrast, press cake enrichment at 100 ppm (SEC-100) yielded the highest IV (131.16 ± 0.09 g I_2/100 g), suggesting a greater contribution of polyunsaturated fatty acids, particularly linoleic acid. This higher unsaturation likely explains the greater susceptibility to oxidation noted in SEC samples. Importantly, all IV values remained within the Codex Alimentarius range for edible oils (118–141 g I_2/100 g) [72], confirming compliance with international quality standards. Taken together, the IV results indicate that argan pulp extracts preserve a balance between unsaturation and oxidative stability, while press cake extracts tend to increase oil susceptibility. These findings underline that the impact of enrichment depends not only on antioxidant compounds but also on the lipid composition of the enriched oil.

3.4.9. Saponification value (SV)

Vegetable oils with shorter fatty acids typically exhibit higher saponification values (SV). Significantly, a high saponification value indicates a high triglyceride content [99]. The obtained SV of supplemented or non-supplemented sunflower oils are presented in Table 4. The findings indicate that no significant variation was reported in SV between the control oil and all the supplemented samples, ranging from 191.29 ± 0.81 mg KOH/g obtained in SEP-200 & SEC-100 to 191.54 ± 0.81 mg KOH/g obtained in SEP-100.

3.5. Fatty acids composition

Fatty acids (FAs) are the main constituents of vegetable oils [99]. This composition not only indicates their stability but also serves as a crucial measure of their nutritional value, especially for edible oils [100]. The results of FAs values of supplemented or non-supplemented sunflower oils are presented in Table 4. Linoleic acid (C18:2) was the most abundant, ranging from 53.45 ± 0.10 g/100 g in SEP-100 to 54.63 ± 0.10 g/100 g in SEC-100, followed by oleic acid (C18:1) (34.14–34.97 g/100 g). Together, these two unsaturated fatty acids accounted for over 85 % of the total profile, confirming sunflower oil as a typical linoleic–oleic oil. Linolenic acid (C18:3), although highly prone to oxidation, was present only in trace amounts (0.06–0.27 g/100 g), which is consistent with the authenticity of the samples, since higher levels would suggest adulteration with oils such as rapeseed or soybean [13,18,101]. Among the saturated fatty acids, palmitic (6.10–7.49 g/100 g) and stearic acids (3.79–4.24 g/100 g) were predominant, while minor fatty acids (myristic, palmitoleic, arachidic, gadoleic) were detected below 1 g/100 g. Generally, the high levels of linoleic and oleic acids resulted in a proportion of unsaturated fatty acids (UFAs) exceeding 88 % in all samples, with the maximum value observed in SEC-100 (89.63 ± 0.10 g/100 g). These results are comparable to those reported for sunflower oils enriched with vitamin E [79], and remain within the Codex Alimentarius standards for edible oils (62–113 g/100 g) 72[22]. Monounsaturated fatty acids (MUFAs) varied between 34.27 g/100 g (SEC-200) and 35.07 g/100 g (SEP-100), remaining well below the Codex upper limit of 39 g/100 g. Saturated fatty acids (SFAs) ranged from 10.35 g/100 g (SEC-100) to 11.64 g/100 g (SEV-200). The ratio UFA/SFA, an indicator of nutritional quality and oxidative behavior, was highest in SEC-100 (8.66 ± 0.08), while the control oil (7.90 ± 0.07) showed values similar to SEL-100, SEL-200, and SEP-200. Interestingly, these ratios surpass those typically reported for Moroccan olive oils [102], underlining the high unsaturation degree of sunflower oil. Overall, the fatty acid profiles confirm that enrichment with argan extracts did not substantially modify the intrinsic lipid composition of sunflower oil. However, the variations observed, particularly in SEC samples, help explain the higher oxidizability (COX, OS) recorded for these oils, compared with the relative stability of pulp-enriched samples.

3.6. Oxidative stability using Rancimat analysis

The Rancimat accelerated oxidation test is a standard method for assessing the oxidative stability of edible oils by measuring the induction time (IT), i.e., the period before a rapid increase in volatile acid formation occurs [47,100,103]. Longer IT values indicate greater resistance to oxidation and provide useful insights into the shelf life of oils [104]. As shown in Table 5, the control sunflower oil displayed the lowest induction times, confirming its high susceptibility to oxidation. Enrichment with argan extracts markedly improved stability, although the effect varied with the type of co-product. Pulp extracts showed the strongest protective effect, with SEP-200 and SEP-100 recording ITs of 5.37 h and 5.14 h at 283.15 K, and 2.72 h and 2.65 h at 293.15 K, respectively. Oils enriched with press cake extracts ranked second, followed by leaf extracts, which still outperformed the control. In contrast, vitamin E enrichment did not significantly improve stability, and in some cases (SEV-100) even reduced IT compared to the control. These results are consistent with previous findings showing that natural extracts can enhance the oxidative resistance of sunflower or soybean oils [103,105]. In contrast, Oubannin et al. [106] reported no significant effect of saffron enrichment on argan oil stability, which was attributed to thermal degradation of sensitive antioxidants. In the present study, the higher effectiveness of pulp, cake and leaves extracts suggests that argan phenolics are more resistant to degradation and capable of prolonging oil stability under accelerated conditions. Altogether, the Rancimat data confirm that enrichment with argan co-products improves the oxidative stability of refined sunflower oil, with pulp extracts providing the most significant extension of induction time. These findings reinforce the potential of argan co-products as sustainable and efficient natural antioxidants, offering an alternative to conventional stabilizers such as vitamin E.

4. Conclusion

This study demonstrates that argan co-products are rich sources of nutrients and bioactive phenolics, and their ethanolic extracts obtained by ultrasound-assisted extraction can effectively enhance the oxidative

Table 5
Induction time (in hours) of control sunflower oil and oil enriched with various argan co-products and vitamin E at 383 K and 393 K measured by the Rancimat test.

	Induction time (hours) at 383 K	Induction time (hours) at 393 K
SC	4.66 ± 0.13	2.39 ± 0.1
SEL-100	5.3 ± 0.15	2.58 ± 0.16
SEL-200	5.06 ± 0.16	2.56 ± 0.13
SEP-100	5.14 ± 0.11	2.72 ± 0.14
SEP-200	5.37 ± 0.2	2.65 ± 0.12
SEC-100	5.15 ± 0.17	2.64 ± 0.15
SEC-200	5.21 ± 0.13	2.61 ± 0.16
SEV-100	3.59 ± 0.12	2.3 ± 0.14
SEV-200	4.59 ± 0.14	2.48 ± 0.12

stability of refined sunflower oil. The ultrasonic enrichment process did not negatively affect the main quality indices of the oil, while Rancimat analysis revealed a significant improvement in resistance to oxidation, particularly with pulp extracts at 200 ppm. These findings highlight the potential of argan co-products as sustainable and natural alternatives to synthetic antioxidants for edible oil stabilization. Future research should further investigate their application in other lipid matrices and assess sensory and technological properties to support industrial use.

CRediT authorship contribution statement

Otmane Hallouch: . **Bouchra Bounasser:** . **Abderrahim Asbbane:** . **Angelo Maria Giuffrè:** . **Waleed Al Abdulmonem:** . **Abdelhakim Bouyahya:** Writing – review & editing, Visualization, Validation, Investigation, Formal analysis. **Khalid Majourhat:** . **Said Gharby:** .

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.

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Data Availability Statement.

The data are contained within the article

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