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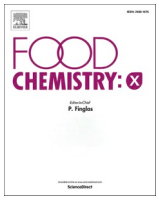

Green enrichment of argan oil (*Argania spinosa* L.) with thyme (*Thymus vulgaris* L.) and oregano (*Origanum vulgare* L.) leaves: Evaluating quality and stability improvements

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A R T I C L E I N F O

Chemical compounds:
Oleic Acid (PubChem CID: 445639)
Linoleic Acid (PubChem CID: 5280450)
Palmitic Acid (PubChem CID: 985)
Carnosic Acid (PubChem CID: 65126)
Carnosol (PubChem CID: 442009)
Rosmarinic Acid (PubChem CID: 5281792)
Thymol (PubChem CID: 6989)
Carvacrol (PubChem CID: 10364)
Apigenin (PubChem CID: 5280443)
Ursolic Acid (PubChem CID: 64945)

Keywords:
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Maceration
Medicinal and aromatic plants
Oxidative stability
Rancimat
Green products
Sustainable yield

A B S T R A C T

This study aimed to assess the impact of enriching argan oil (AO) (*Argania spinosa* L.) using the maceration technique with thyme (*Thymus vulgaris* L.) and oregano (*Origanum vulgare* L.) leaves (TL and OL) at two proportions (5 and 10%). The oxidative stability of the control and enriched oils was examined under accelerated conditions at a temperature of 60 °C for 120 days (4 months). Quality indices (Free fatty acids (FFA), peroxide value (PV), *p*-anisidine value (*p*-AV), ultraviolet absoptions (K232 and K270), Rancimat test, fatty acids composition, sensory attributes, simple phenolic contents (SPC) and antioxidant activity (DPPH•) were determined. As a simple, inexpensive and green method, enrichment by maceration yielded advantageous results. Compared to the control (68.05 ± 1.10 mg GAE/kg), the SPC significantly increased in enriched oils reaching notably 250.9 ± 9.1 mg GAE/kg when adding 10% of TL. Also, the enriched oil samples showed the lowest PV, *p*-AV and ultraviolet absorptions compared with the control. However, no noticeable changes were reported in fatty acids composition and iodine value. In terms of sensory attributes, enrichment by maceration masked the rancid odour caused by oxidation. These scientific discoveries inherently yield economic advantages by enabling the diversification of product offerings, simultaneously catering to a broader market seeking high-quality oils infused with herbs, including both AO and aromatic plants.

1. Introduction

Commonly recognized as the argan tree, *Argania spinosa* L. is a unique and valuable tree native to the south western regions of Morocco. Renowned for its hardy nature and ability to thrive in arid conditions, the argan tree has adapted to the challenging environment of the semi-desert areas of North Africa. AO is the most renowned product derived from the argan tree, often referred to as "liquid gold" (Guillaume et al., 2019). This oil has a rich history deeply rooted in Moroccan culture and has been utilized for various purposes for centuries. Historically, Berber communities, who are indigenous to North Africa, have used AO as a key component in their culinary and medicinal practices (Charrouf & Guillaume, 2008). The nutritional properties of AO, combined with its cultural significance, have contributed to its widespread recognition and utilization worldwide (Ruas et al., 2016). For ages, thyme (*Thymus vulgaris* L.) and oregano (*Origanum vulgare* L.) herbaceous Mediterranean plants of the Lamiaceae family have been integral parts of many cultures, fulfilling a variety of purposes in the culinary, medicinal, and

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aromatic domains (Santoro et al., 2007). Both plants are rich in essential oils, phenolic compounds and flavonoids, giving them powerful antioxidant, antimicrobial and anti-inflammatory properties. Thymol and carvacrol, the main constituents of thyme and oregano respectively, are particularly effective in neutralizing reactive oxygen species and alleviating oxidative stress. These bioactive compounds have been shown to enhance immune function, reduce inflammation and exhibit antimicrobial activity against a broad spectrum of pathogens. The synergistic effects of these bioactive components contribute to the overall therapeutic potential of these two plants, making them valuable in preventive and therapeutic applications of medicine (Gavaric et al., 2015). Vegetable oils oxidation is a natural and complex chemical reaction that takes place when the oils are exposed to air, heat, and light. This oxidative reaction involves the degradation of unsaturated fatty acids present in oils, giving birth to the formation of undesirable compounds such as peroxides, ketones and aldehydes. As oils undergo oxidation, their color, flavor, and nutritional quality can be significantly compromised, resulting in rancidity and off-putting tastes (Gharby et al., 2022). Oxidation also affects the oil's shelf life and nutritional benefits, making it imperative to store and handle vegetable oils properly. Antioxidants, either naturally occurring or added, play a crucial role in slowing down this process by neutralizing free radicals then prolonging the onset of oxidation. Hence the idea of taking advantage of the anti-oxidants provided by medicinal plants, instead of using synthetic antioxidants in the harmful effects are detected (Fadda et al., 2022). Moreover, the rationale for enriching AO with oregano and thyme lies at the intersection of scientific intrigue and practical significance. AO, renowned for its unique nutritional composition and versatile applications, can potentially undergo a transformative enhancement by incorporating the bioactive compounds present in these two medicinal plants. The synergistic interaction between the bioactive compounds of AO and these two specimens may lead to a harmonious amalgamation that not only enhances the overall nutritional value but also imparts novel functional attributes to the enriched oil. Especially with the green used enrichment process that holds promise for creating a product that not only capitalizes on the inherent benefits of each component but also introduces a new dimension of health and sensory appeal, catering to the evolving preferences of consumers seeking natural and efficacious solutions. This study was prompted by successful attempts to enrich AO by maceration with *Crocus sativus* L. (Oubannin, Asbbane, Bijla, et al., 2023; Oubannin, Asbbane, Elhaidag, et al., 2023) and enrichment during extraction with *Thymus vulgaris* L. (Oubannin, Asbbane, Bijla, et al., 2023; Oubannin, Asbbane, Elhaidag, et al., 2023). These endeavors yielded promising results, prompting further investigation into alternative enrichment techniques utilizing diverse aromatic and medicinal plants. The discoveries from these explorations are expected to contribute novel perspectives on the field of botanical extraction and oil enrichment methodologies (Oubannin et al., 2024).

To the best of our knowledge, no prior research has investigated the enrichment of AO through maceration with oregano and thyme leaves (OL and TL). This study is original in evaluating how the incorporation of these leaves affects physicochemical characteristics, and oxidative stability of this gold liquid. Furthermore, enriching AO with aromatic and medicinal plants have a significant economical impact by diversifying argan products and increasing their added value, which can translate into higher profit margins and access to new markets. This practice also encourages the creation of local jobs in cultivation, harvesting and processing, while supporting farmers and enhancing the value of local resources for both AO and aromatic and medicinal plants.

2. Material and methods

– Argan oil, thyme and oregano samples

The base product used for this study is AO (*Argania spinosa* L). Argan nuts were harvested in the Tighanimine area of Agadir, Morocco (35° 12′ 40″ north, 6° 18′ 10″ west). Then, using a traditional crushing process, the women of the cooperative (Tighanimine cooperative) provide us with the kernels to extract AO using a mechanical extractor. Oregano (*Origanum vulgare* L.) and thyme (*Thymus vulgaris* L.) are cultivated and widely found in this region. Moreover, TL and OL are cleaned and prepared by the women of the cooperative (Targanine cooperative).

– Argan oil enrichment procedure

The maceration process used for enrichment consists of immersing TL and OL (previously ground and passed through a 125 µm sieve) in AO at two quantities: 5 and 10%. More precisely, for an enrichment with 5% of the plant, 30 g of the powder, was immersed in 600 g of AO previously placed in an erlenmeyer, similarly for 10%, 60 g of the powder is immersed in 600 g of AO, for the two powders of TL and OL separately, to obtain then 4 samples of enriched argan oils: Enriched oil with 5% of TL (EOTL 5%), enriched oil with 10% of TL (EOTL 10%), enriched oil with 5% of OL (EOTL 5%) and enriched oil with 10% of OL (EOOL 10%), then, the whole is stirred for 7 days (1 week) to allow optimal extraction of the bioactive compounds of OL and TL in the AO. During this time, the ambient temperature (25 ± 3 °C) is carefully controlled. The precision of these parameters is crucial to obtain a balanced and enriched AO with enhanced sensory and functional attributes. Specific values for time, temperature and ratios may vary according to the objectives of individual studies or production preferences. Finally, the macerate mixtures are filtered using a Whatman filter paper. The same mass (600 g) of non-enriched AO was also used as a control.

– Accelerated ageing study

Once filtered, the control (unenriched argan oil) and the oils enriched with TL and OL at different concentrations were placed in a series of 30 mL opaque glass bottles. After filling, the bottles were securely sealed to maintain the integrity of the contents, the number of vials filled with each sample was designed to be sufficient to be taken each month for all the necessary analyses (16 bottles for each of the fifth samples). Then, the accelerated ageing was carried out in an oven (BINDER GmbH, Bergstr 14, D-78532 Tuttlingen, Germany) set at 60 ± 1 °C for a period of 120 days (4 months). Once every 30 days during the 120 days the samples were taken to monitor oxidative stability by performing a series of analyses, namely; Peroxide value (PV), free fatty acids (FFA), *p*-anisidine value (*p*-AV), and chlorophylls content. While Rancimat test, fatty acids composition and iodine value (IV) were evaluated at the initial (Day 0) and final (120 days) states of the four months storage period. Simple phenolic content (SPC), total flavonoids content (TFC) and radical scavenging activity by DPPH• are conducted prior to the accelerated ageing process, whereas sensory attributes were monitored at the end.

2.1. Rancimat test

A Metrohm Professional Rancimat model 892 (Herisau, Switzerland) was employed to determine the resistance to oxidation of the samples under investigation. Each oil sample, weighing 3 g, was placed into the reaction tube and subjected to high temperature (120 °C) in the presence of an air flow rate of 20 L/h. Pure water was used in the conductivity cell. All the measurement vessels, electrodes, including air tubes, underwent thorough cleaning before and between tests, then purged with nitrogen before the experiment to prevent any contamination that could catalyze peroxidation, results are expressed by induction period (h) (IP) (Gagour, Ahmed, et al., 2022; Gagour, Oubannin, et al., 2022).

2.2. Total phenolic content and radical scavenging activity by DPPH• assay

2.2.1. Preparation of oils polar extracts

2.5 g of each oil sample were carefully measured into individual centrifuge tubes, followed by the addition of 5 mL of hexane along with 5 mL of methanol-water solution (60:40, *v*/v). After vigorous vortex mixing for 2 min, the tubes underwent centrifugation at 5000*g* for 5 min (Ballus et al., 2015). Leaves powder (LP) of each plant (1 g) were extracted with 10 mL of methanol-water (80:20) with stirring for 24 h. The mixture was then filtered through Whatman filter paper (Ait Bouzid et al., 2023). The polar phase at the bottom was extracted and used for both total phenolic content analysis and various antioxidant capacity assessments.

2.2.2. Simple Phenolic Content (SPC)

Simple phenolic content (SPC) was assessed using the method of Folin-Ciocalteu. Where, 0.5 mL of the oil polar extract or LP extract was combined with 2.5 mL of diluted Folin-Ciocalteu reagent (1:10 in ultrapure water). Allow the oil mixture to stand in the dark for 5 min. Subsequently, for the oil extract, 2 mL of 7.5% sodium carbonate (Na_2CO_3) was introduced, and the tubes were then incubated in darkness for 2 h. While, for the LP extract, add 4 mL of Na_2CO_3 (7.5%) and incubate at 45 °C for 30 min The used wavelength to mesure absorbance of the oil extract was 760 nm and the LP extract at 765 nm, quantitative results were derived from an analytical curve constructed with gallic acid, expressed as milligrams of gallic acid equivalents per kilogram of oil (mg GAE/kg oil) or (mg GAE/g DM) (Ait Bouzid et al., 2023; Ballus et al., 2015).

2.2.3. Total Flavonoid Content (TFC)

TFC of oil or LP samples were determined through a colorimetric method. 1 mL of the polar solvent from the oil or LP were combined with 0.3 mL of $NaNO_2$ (0.5 M) is added to a 10 mL test flask and left to stand for 5 min at room temperature. Subsequently, the mixture of oil or LP were introduced to 0.3 mL of $AlCl_3$ (10%). After six minutes, the solution was transferred to 1 mL of NaOH (2 M) and topped up to the mark with distilled water. The absorbance of the samples was measured at 415 nm using a SCILOGEX SP-UV1100 spectrophotometer. The TFC was quantified and expressed as quercetin equivalent per gram of oil or dry metter (DM) (mg QE/g oil or mg QE/g DM) following the methodology described by Park et al. (2019) (Park et al., 2019).

2.2.3.1. Radical scavenging activity by DPPH• assay. This assay involves introducing the DPPH• (2,2-diphenyl picryl hydrazine) radical, characterized by a purple color, into a solution containing molecules referred to as "antioxidants" to assess their ability to counteract this radical. The neutralized form, indicated by a yellow color, no longer absorbs light, resulting in a reduction of absorbance at the corresponding wavelength. In this context, antioxidants act to convert the purple DPPH• into a yellow compound, DPPH•, with the color intensity inversely proportional to the concentration of antioxidants present in the medium (Aljaiyash et al., 2018). Absorbance of the oil extract or LP extract values were measured at 517 nm using a spectrophotometer (SCILOGEX SP-UV1100) and were then converted into the percentage of antioxidant activity (AA, %) using a specific eq. (1). Antioxidant activity of LP extract was expressed as mg equivalents of ascorbic acid per g of dry material (mg AAE/g DM).

$$\%AA = 100 - \left[\left(\left(\mathbf{Abs_{sample}} - \mathbf{Abs_{blank}}\right) \times 100\right)/\mathbf{Abs_{control}}\right] \quad (1)$$

where $\mathbf{Abs_{blank}}$ is the solvent absorbance, $\mathbf{Abs_{control}}$ is the DPPH• absorbance solution and $\mathbf{Abs_{sample}}$ is the sample absorbance.

2.2.4. Parameters for monitoring oxidative stability

The evaluation of oil stability was conducted through the measurement of free fatty acids (FFA), (g/100 g of oleic acid), peroxide value (PV) (milliequivalents (mEq) O_2/kg oil), ultraviolet absorptions (K232 and K270) and para-anisidine value (*p*-AV) in accordance with the analytical methods described by the International Standard Organization (ISO 3960, 2017; ISO 660, 2020; ISO 6885, 2006) respectively,

Briefly, the FFA was determined by the titration of a solution of oil in ethanol with ethanolic NaOH (0.1 N). The PV was determined by iodine titration with a sodium thiosulfate solution (0.01 N) of oil in isooctane/acetic acid 2:3 that had been left in darkness in the presence of potassium iodide. K232 and K270 were expressed as the specific extinctions of a 1% (*w/v*) solution of cyclohexane-oil measured in a 1 cm cuvette, using a SCILOGEX SP- UV1100 spectrometer (Rocky Hill, CT 06067, USA). These coefficients are obtained by measuring the absorbance of the oil sample at 232 nm for K232 and 270 nm for K270.

For *p*-anisidine value, into a 25 mL flask, 2 g of the oil sample is placed then diluted with 25 mL of isooctane to prepare solution A. The absorbance (Ab) of solution A at 350 nm is obtained using a SCILOGEX SP-UV1100 spectrometer, with isooctane as the blank. Subsequently, 5 mL of solution A is moved into the initial tube, then 1 mL of *p*-anisidine solution (0.25 % w/v in glacial acetic acid) is added. Incubated for a 10-min, the absorbance (As) at 350 nm, with the blank reference being the second tube where isooctane and *p*-anisidine. The *p*-AV is computed based on the following formula (2):

$$\mathbf{p-AV} = (25x(1.2x(As - Ab)))/m \quad (2)$$

where **As** is the oil solution sample post-reaction with *p*-anisidine absorbance. **Ab** is the the non-reacted test solution absorbance. **m** is the mass in grams of the test sample. **25** is the equivalent volume of isooctane dissolving the oil. **1.2** is a correction factor used to adjust for the dilution caused by adding 1 mL of *p*-anisidine solution to the test sample (Nid Ahmed, Abourat, et al., 2024; Nid Ahmed, Gagour, et al., 2024).

Total oxidation (TOTOX) value was also evaluated using the eq. (3):

$$\mathbf{TOTOX} = (\mathbf{2} \times \mathbf{PV}) + \mathbf{p-AV} \quad (3)$$

where **PV** is the peroxide value and ***p*-AV** is the para-anisidine value (Tavakoli et al., 2019).

2.2.5. Total chlorophylls

Approximately, 7.5 g of oil sample was added and then diluted with cyclohexane in a 25 mL volumetric flask. The chlorophyll content was determined by measuring the absorbance at 670 nm, and the calculation was performed using the provided eq. (4) (Bijla et al., 2024):

$$\mathbf{Total\ chlorophylls\ (ppm)} = \frac{\boldsymbol{A}670 \times 10^6}{613 \times 100 \times \boldsymbol{L}} \quad (4)$$

where **A** represents the measured absorbance, and **L** denotes the thickness of the spectrophotometer cell, which is set at 10 mm.

2.2.6. Fatty acids composition and iodine value (IV)

The fatty acids underwent transmethylation to convert them into their corresponding fatty acid methyl esters (FAME), according the International Standards Organization (ISO) method (ISO 12966-2, 2017). Fatty acids composition was conducted through gas chromatography using a CP-Wax 52 CB column (30 m × 0.25 mm inner diameter, 0.25 μm film thickness). Helium served as the carrier gas with a flow rate of 1 mL min^{-1}. The temperature settings for the detector, oven, and injector were 230, 170, and 200 °C, respectively. Each test involved injecting a volume of 1 μL with a split ratio of 1:50. The iodine value (IV) was computed based on the percentages of unsaturated fatty acids using the provided eq. (5) (Gharby et al., 2017):

$$\mathbf{IV} = (\%\mathbf{C16} : \mathbf{1} \times \mathbf{1.001}) + (\%\mathbf{C18} : \mathbf{1} \times \mathbf{0.899}) + (\%\mathbf{C18} : \mathbf{2} \times \mathbf{1.814}) + (\%\mathbf{C18} : \mathbf{3} \times \mathbf{2.737}) \quad (5)$$

2.2.7. Sensory attributes

A group of 10 trained evaluators, which includes an expert professor, participated in the evaluation. All evaluators, between the ages of 20–50 years and nonsmokers. The sensory assessment involved the five samples (Control, EOOL 5%, EOOL 10%, EOTL 5% and EOTL 10%). The latters were presented in amber glasses labeled with numerical codes and maintained at the tasting room temperature (22 ± 2 °C). The assessed parameters included the assessment of the overall acceptance of the jury, the oil rancidity due to prolonged storage, as well as the intensity of flavor, color and aroma resulting from the addition of OL and TL. Ratings were assigned on a 4 points scale, with 0 indicating absence, 1 representing the least intensity, and 4 indicating the strongest intensity, referencing to Mihaylova et al., 2020 with some modifications (Mihaylova et al., 2020).

2.2.8. Statistical analyses

The findings are expressed as mean ± standard deviation ($n = 3$). Some results were analyzed using one-way analysis of variance (ANOVA) and other using the Principal Component Analysis (PCA) using R software version 4.1.2. Significance was established at a 5% probability level. Graphs, correlation matrix and sensory radar map, were created using OriginPro 2024 software.

3. Results and discussion

3.1. Improving the initial conditions of argan oil

3.1.1. SPC, TFC, and antioxidant activitiy of plants' extracts

Lamiaceae species are renowned for containing a range of secondary metabolites, notably phenolic compounds (Ramos da Silva et al., 2021). Therefore, assessment of these compounds for these two investigated plant extracts is indispensable. Table 1 represents SPC, TFC and antioxidant activity using the DPPH• (2,2-diphenyl-1-picrylhydrazyl) test that delivers insight into free radical scavenging capacity of *Thymus vulgaris* L. and *Origanum vulgare* L. leaves. In the studied plants, thyme recorded significantly higher SPC and TFC with values of 50.38 ± 0.12 mg GAE/g DM and 95.52 mg QE/g DM respectively. Followed by oregano which registered a SPC of 43.84 mg GAE/g DM and 90.41 mg QE/g DM for the total flavonoids.

Concerning the DPPH• antioxidant activity test, *Thymus vulgaris* L. leaves powder always ranks first with 34.27 ± 0.14 mg AAE/g DM (Table 1). The same finding has been reported by other studies that have worked on Moroccan medicinal and aromatic plants such as the one of a Ait Bouzid et al. (2023) (Ait Bouzid et al., 2023). While in an another study of Amarowicz et al. (2009) where thyme and oregano are from Poland, the highest phenolic content and antioxidant activity were determined for oregano then the thyme comes in the second position (Amarowicz et al., 2009).

3.1.2. SPC, TFC, and DPPH• of oils

We initially assessed the variation in simple phenolic content and antioxidant activity by comparing control and enriched oils with aromatic herbs (thyme and oregano). It is important to estimate these level of phenolics (SPC and TFC), since they are known for their antioxidant properties, stemming from their capacity to chelate metals, inhibit lipoxygenase, and neutralize free radicals, which provides a role to play in the long-term stability of vegetable oils (Decker & A., 1997). A significant increase in SPC with the addition of OL and TL after one week under agitation. SPC of the control was 68.05 becoming 140.9, 190.9, 187.9, and 250.9 mg GAE/kg oil after enrichment with 5 and 10% of TL and OL, respectively (Table 2). The same applies to TFC, where the enriched oils have the largest amounts ($p < 0.05$) (Table 2). In particular, EOTL (10%) reached a value of 0.30 mg QE/g oil which is five times higher than the TFC of the control (0.06 mg QE/g). Polyphenols and flavonoids, as bioactive compounds, are abundant in oregano and thyme, which will certainly improve the quality of the AO. Incorporating these plants into a carrier matrix such as AO facilitates the direct infusion of these compounds into the solution. More specifically, oregano is a source of polyphenols such as rosmarinic acid and carvacrol and flavones as apigenin which has strong antioxidant, anti-inflammatory, antimicrobial and anti-cancer properties (Gutiérrez-Grijalva et al., 2018), while thyme is rich in thymol and ursolic acid. Integrating these polyphenols and flavonoids into the lipid matrix of AO considerably increases the overall concentration of antioxidants in the oil.

DPPH• test is considered to be a representative of a stable lipophilic radical that is started when antioxidants bind to DPPH•, they reduce its molecules with an amount that is equivalent to the number of accessible hydroxyl groups (Roby et al., 2013). As it can be seen in Table 2, a clearly significant difference ($p < 0.05$) between control (0.08%) and enriched oils either with thyme or oregano at 10% (0.29 and 0.27%) is reported showing a concentration-dependent effect. This improvement in the antioxidant activity of AO after enrichment by maceration is very important in extending its shelf life, since free radicals take a major part in the lipid peroxidation reaction. Then, subsequently avoiding the problems caused by lipid oxidation, i.e. reduced nutraceutical properties and deteriorated food quality, including unpleasant flavours and odors (Machado et al., 2023). The variation in DPPH• activity can be ascribed to the hydroxyl groups within thym and oregano flavonoid compounds (Fig. 1), which are proven to play a role in its potent radical scavenging effectiveness (Zeghad & Merghem, 2013). Similar results are reported when evaluating antioxidants in oils after enrichment with diffrents plants, such as the study by Şahin et al. (2017) using olive leaves to enrich corn oil (Şahin et al., 2017). This growth could be explained by the high phenolic compound content of these two plants belonging to the Lamiaceae family (previously mentioned in the section above).

3.2. Principal component analysis (PCA)

The PCA presented in Fig. 2, illustrates the correlation between TFC, DPPH•, and SPC variables and the samples. The interpretation of the principal components is based on the search for the variables most strongly correlated with each component (Gewers et al., 2021). In present study, we retained the first two principal components (PCs) as they accounted for nearly 100% of the total variance in our dataset. This high percentage indicates significant variability explained by this model as depicted in Fig. 2, all three variables studied correlate positively with PC1, which accounts for over 92% of the variance, leading to a clear

Table 1
Simple phenolic content (SPC), total flavonoid content (TFC) and antioxidant activity (DPPH•) of the plants investigated. In each column, values followed by the same letter are not significantly different at $p < 0.05$.

	SPC (mg GAE/g DM)	TFC (mg QE/g DM)	DPPH• (mg AAE/g DM)
Thymus vulgaris L	$50.38^{a} \pm 0.12$	$95.52^{b} \pm 0.90$	$34.27^{b} \pm 0.14$
Origanum vulgare L	$43.84^{b} \pm 1.56$	$90.41^{c} \pm 0.10$	$31.77^{c} \pm 0.26$

Table 2
SPC, TFC and DPPH• of control and enriched oils with oregano leaves (EOOL) and thyme leaves (EOTL) with 5 and 10% amounts. In each line, values followed by the same letter are not significantly different at $p < 0.05$.

Parameters	Control	EOOL (5%)	EOOL (10%)	EOTL (5%)	EOTL (10%)
SPC (mg GAE/kg oil)	$68.05^{a} \pm 1.10$	$140.9^{b} \pm 11.1$	$190.9^{c} \pm 21.1$	$187.9^{cd} \pm 11.1$	$250.9^{e} \pm 9.1$
TFC (mg QE/g oil)	$0.06^{a} \pm 0.01$	$0.19^{b} \pm 0.01$	$0.19^{b} \pm 0.01$	$0.16^{c} \pm 0.01$	$0.30^{d} \pm 0.01$
DPPH• (%)	$0.08^{a} \pm 0.01$	$0.25^{b} \pm 0.03$	$0.27^{bc} \pm 0.03$	$0.19^{d} \pm 0.03$	$0.29^{e} \pm 0.03$

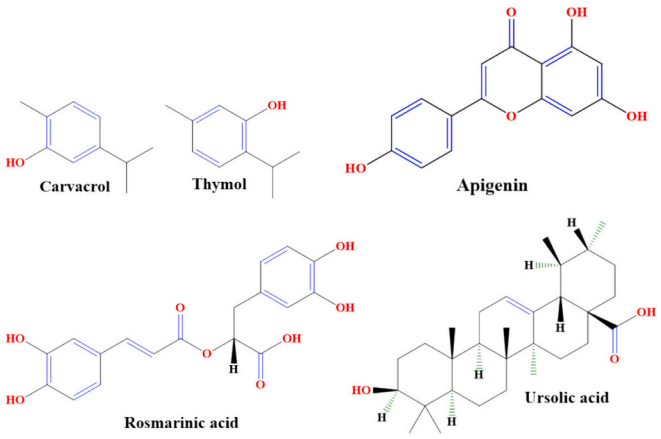

Fig. 1. Oregano and Thyme bioactive compounds.

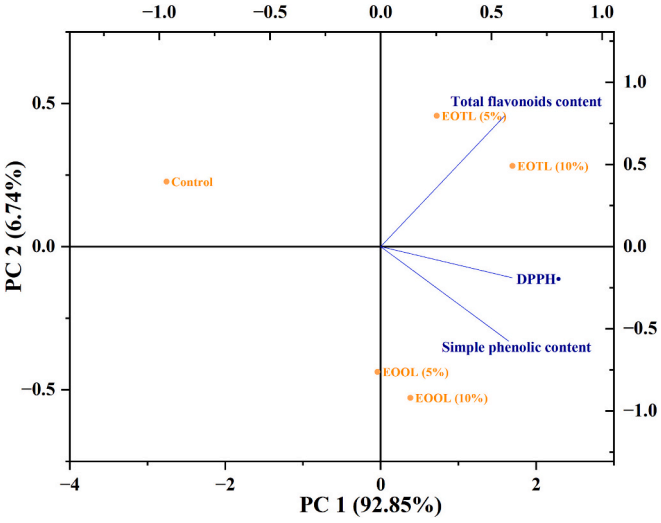

Fig. 2. Principal component projections for the first two PCs of DPPH•, Simple phenolic content and total flavonoid content. Blue segments represent dependent variables, while points plotted are mean values of the studied samples. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

separation of the samples. Samples located on the positive side of PC1 exhibited higher concentrations of the targeted variables compared to those on the negative side. Specifically, the sample 'EOTL 10%' being more correlated with the PC1 which displayed higher concentrations of the studied variables. A noticeable grouping of samples, namely "EOOL (5%)" and "EOOL (10%)", appears in the bottom area of the PC2. This specific categorization highlights their strong positive correlation and moderate similarity, hence their proximity in terms of antioxidant activity profile. Whereas the control sample recorded the lowest values for all mentioned variables. This analysis gave a clear picture of the strong difference between the unenriched and enriched oils in terms of the assessed variables.

3.3. Accelerated ageing study

3.3.1. Rancimat test

The Rancimat method is an accelerated ageing test (Almoselhy, 2021). By constantly increasing the temperature in the reaction vessel, air is passed through the sample. The fatty acids are then oxidised and volatile compounds are produced in the water in which the electrical conductivity is measured (Aparicio et al., 1999). The rancimat test is carried out on the first day of enrichment and after the 120 days heat treatment (Fig. 3). First of all, As the IP increases, the oil becomes more stable. The same IP value (13 h) has already been found by evaluating the IP at the same temperature (120 °C) of a freshly prepared AO (Gharby et al., 2012). Initialy, the control with EOOL (10%) and EOTL (10%) statistically indicated comparable IP. While 5% of EOOL and EOTL presented a higher stability with values of 15.46 and 14.56 h respectively. After four months of storage in an oven set at 60 °C, the Rancimat analysis is redone. Compared with control oil (4.95 h), the IP of enriched oils is two times greater when both aromatic and medicinal plants have been added (8.25 and 8.57 h for EOOL (5%) and EOTL (10%), respectively) at $p < 0.05$. Since the principle of Rancimat is based on the measurement of oxidation products due to the oxidation of oils, the results found at the end of accelerated ageing can be interpreted by the fact that the two plants added were able to delay the hydrolysis of fatty acids, i.e. less release of peroxides and greater oxidation stability, which may be linked to their antioxidant composition. The two plants under study are abundant in polyphenolic compounds, particularly flavonoids, which are known for their effective radical-scavenging properties due to their hydrogen-donating ability. This activity is thought to be influenced by several structural features of the flavonoids such as; the 3-OH group in the C ring also contributes to the antioxidant activity of flavonoids, the presence of a 3′,4′-dihydroxy group in the B ring, which has electron-donating properties and targets radicals, the combination of 3-OH and 5-OH groups with a 4-carbonyl function and a C2-C3 double bond and the C2-C3 double bond conjugated with a 4-keto group enhances electron delocalization in the B ring, further boosting radical scavenging ability (Amic et al., 2003). Similar findings were documented by Artajo et al. (2006), who note a substantial rise in IP following the enrichment of refined olive oils with various phenolic compounds found in fruits and olive oil (Artajo et al., 2006).

3.3.2. Free fatty acids (FFA)

FFA expressed as a percentage by weight of oleic acid, this parameter allows the classification of extra virgin, virgin and lampante oils such as the determination of their deterioration status (Oubannin et al., 2022). From a statistical point of view, the initial results did not give rise to significant variance (Fig. 4 (A)). However, within the first month a discrepancy began to appear ($p < 0.05$); the control recorded a FFA value of 0.29 ± 0.02 g/100 g, while all enriched oils notably; 10% of OL and TL have established values that have risen to 0.35 ± 0.01 and 0.34 ± 0.01 g/100 g respectivly. This observed rise in the enriched AOs

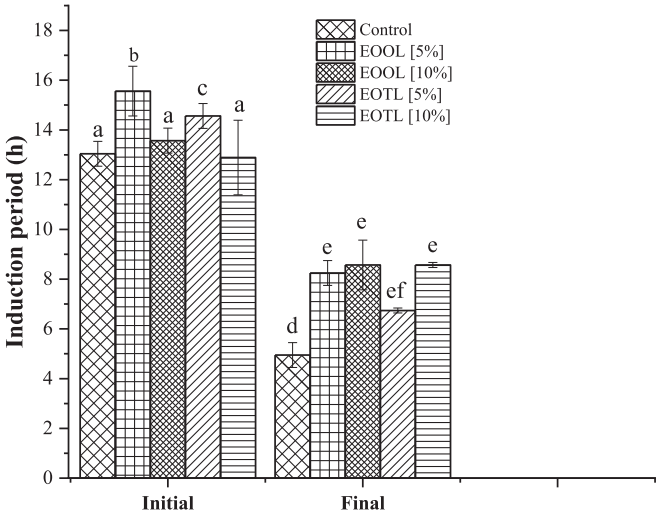

Fig. 3. Induction period (h) of control, enriched oil with oregano leaves (EOOL) and enriched oil with thyme leaves (EOTL) over 120 days of storage under accelerated ageing conditions (60 °C) at two concentrations (5 and 10%). Bars with different letters are significantly different at $p < 0.05$.

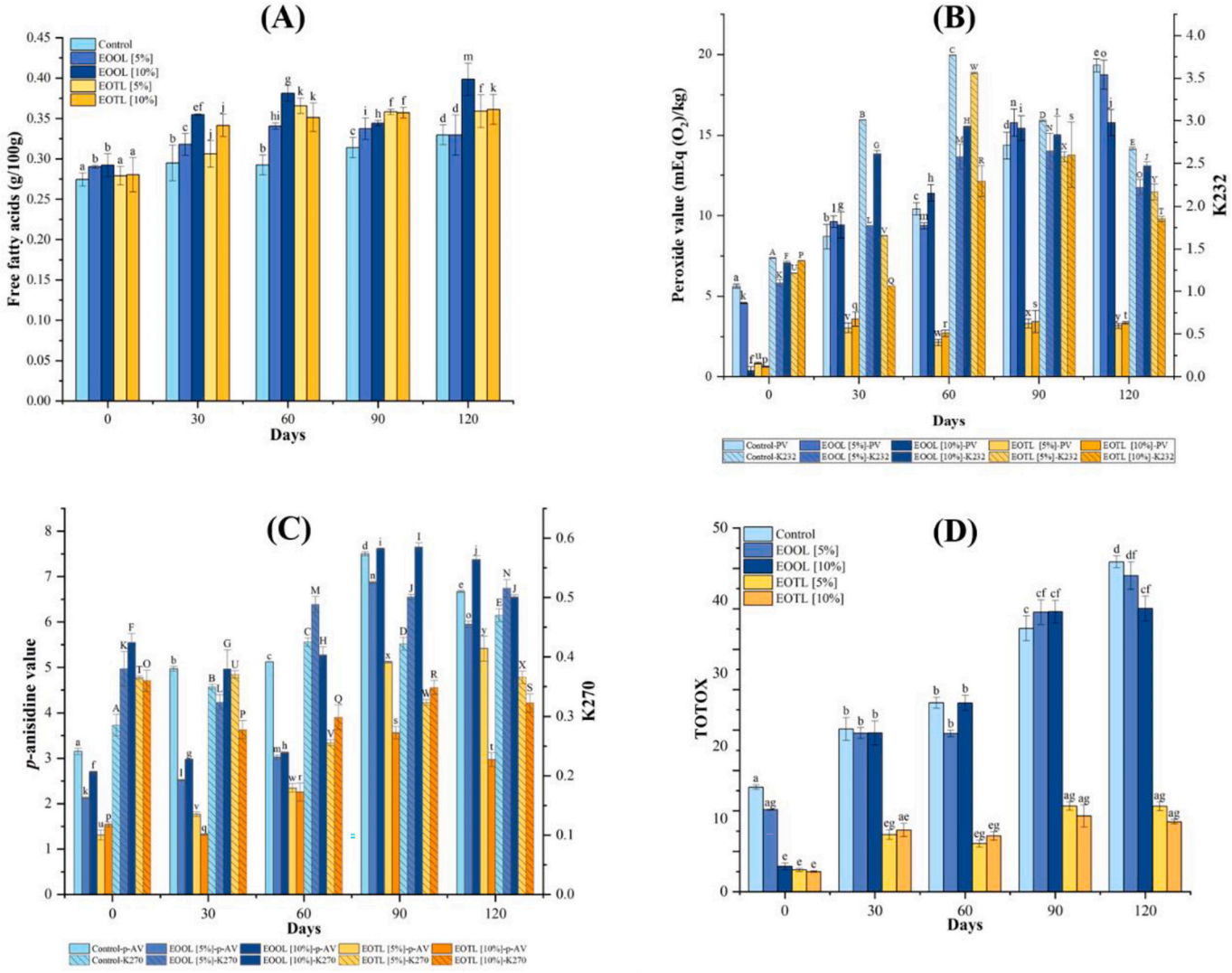

Fig. 4. (A) free fatty acids (B), peroxide value and K232 (C), *p*-anisidine value and K270 (C) and TOTOX (D) of control, enriched oil with oregano leaves (EOOL) and enriched oil with thyme leaves (EOTL) over 120 days of storage under accelerated ageing conditions (60 °C) at two concentrations (5 and 10%). Bars with different letters are significantly different at $p < 0.05$ (the lower-case letters refer to PV (B) and p-AV (C), and the upper-case letters refer to K232(B) and K270 (C)).

continued throughout the four months, and was more marked in the EOOL (10%) which reached at the end of storage 0.39 ± 0.02 g/100 g, where the control did not exceed 0.32 ± 0.01 g/100 g. Nevertheless, it's crucial to mention that that even the addition of these leaves resulted in a slight excess of FFA but all the oil kept the label of "extra virgin" since they did not exceed 0.8 g/100 g, the limit set by the Moroccan standard (SNIMA, 2003). It should be pointed out that, in addition to chemical hydrolysis, FFA also gives an idea of the degeneration of oils by enzymatic hydrolysis to form non-volatile components, which could be the cause of this slight increase caused by plants (Zahran & Najafi, 2020). This rise in acidity can also be attributed to the release of organic acids from aromatic plants, in particular rosmarinic acid and carnosic acid. During the maceration process, these organic acids are solubilised in the oil, resulting in a high concentration of free acids. This high concentration of free acids contributes directly to the overall increase in the oil's acidity. Similar findings were obtained after the enrichment during extraction of AO with *Thymus vulgaris* L (Oubannin, Asbbane, Bijla, et al., 2023; Oubannin, Asbbane, Elhaidag, et al., 2023). Unlike the enrichment by maceration of AO using saffron petals, which delayed the increase in FFA for six months of storage (Oubannin, Asbbane, Bijla, et al., 2023; Oubannin, Asbbane, Elhaidag, et al., 2023). However, other authors have not reported significant differences between enriched and unenriched olive oils (Clodoveo et al., 2016).

3.3.3. *Primary oxidation: Peroxide value and K232*

The peroxide value (PV) and the specific extinction coefficient (K232) are two parameters that can be used to indicate the presence of primary oxidation products (Gharby & Charrouf, 2022).

The concentration of hydroperoxides, which are one of the principal products of primary oxidation, is indicated by the peroxide value (PV). It is an extremely useful method for determining the early stages of oxidative degradation with satisfactory sensitivity (Gagour, Ahmed, et al., 2022; Gagour, Oubannin, et al., 2022). Clearly, a progressive and significant ($p < 0.05$) increase in PVs was observed across all samples throughout the experimental period, correlating with storage time, indicating enhanced production of hydroperoxides caused by the temperature of 60 °C (Fig. 4 (B)). The highest values were observed in the control and the lowest value was noticed in the samples treated with 10% of TL powder. From 30 days onwards, the same behaviour is always reported, EOTLs with EOOLs showed lower PVs compared to the control. Surprisingly, the progress in PVs of enriched oils increased differently depending on the plant deployed for enrichment (thyme or oregano) and the concentration used (5 or 10%). After 60 days EOTLs had PVs equal to 2.13, and 2.70 $mEqO_2/kg$ for the 5 and 10% concentrations respectively.

While the control reached 10.42 $mEqO_2/kg$. At the end, this unenriched oil finished with a value of 19.33 $mEqO_2/kg$ considered to be the maximum value for the entire storage period and six times higher than that of EOTL 10%. Since the peroxide value is a measurement of the quantity of oxygen found in an environment, the results found can be explained by the oxygen trapping capacity of these compounds (previously evaluated (Table 1)) of the two used Lamiaceae. The formation of peroxides in oils begins with the generation of free radicals such as peroxyl radicals (ROO•). These radicals react with molecular oxygen (O_2) to produce lipid peroxides, including hydroperoxides (ROOH) (Gharby et al., 2022). This oxidation process often propagates through a chain reaction, where the products of oxidation generate additional free radicals, accelerating lipid degradation. Thus, by macerating OL and TL powders in AO, an inhibition of free radicals is implemented; the antioxidants present in these plants, namely thymol and carvacrol, react with free radicals (such as ROO•) before they can attack unsaturated lipids. By neutralizing these radicals, the antioxidants slow down the formation of peroxides. Furthermore, they also scavenge reactive oxygen species (ROS), reducing their concentration and further inhibiting oxidative damage (Chroho et al., 2024). Comparable results were achieved previously by Moczkowska et al. (2020), who found a significant decrease in lipid peroxides after incorporating rosemary extract into hemp seed oil (Moczkowska et al., 2020). Also, similarly to Gambacorta et al. (2007) observed that oils enriched with Oregano (*Origanum vulgare* L.) extract demonstrated PVs six times lower at all concentrations compared to olive oil (Gambacorta et al., 2007).

The most appropriate method for checking the results of PV is the Ultraviolet absorption at 232 nm (K232), since it also indicates the rate of primary oxidation (Gharby et al., 2020). As shown in Fig. 4 (B), K232 rises significantly for all oils with increasing time, indicating an elevation in the production of primary oxidation compounds, the hydroperoxides. Crucially, from the first 30 days, all AOs supplemented with TL and OL powder displayed lower K232 values than the control sample. After 120 days of the accelerated ageing, a significant difference is reported between the K232' samples, showing absorptions of 2.16, 1.85, 2.22, 2.47 and 2.67 for EOTL (5%), EOTL (10%), EOOL (5%), EOOL (10%), and control respectively. This behaviour continued until the end of the 2 months of the thermoxidation process at almost all time points, supporting the results previously found by PV. Though, after 90 days K232 showed a drop ($p < 0.05$) which may be interpreted by the start of the degradation of primary oxidation products and the birth of secondary ones, as demonstrated by the kinetics of hydroperoxide oxidation, which exhibits an exponential curve (Cuvelier & Maillard, 2013). Thus, based on the current results, thyme and oregano concentrations of 5 and 10% can be recommended for the stabilization of vegetable oils which can certainly be linked to the phenolic compounds, which prolonge the shelf life of foods and protect lipids against autoxidation by acting as antioxidants that quench free radicals (Kozłowska et al., 2015). In fact, the ability of these two plants to inhibit the formation of primary oxidation products has already been found by Kozłowska and Gruczyńska (2018) (Kozłowska & Gruczyńska, 2018).

The two plants in question are proven for their ability to neutralize superoxide radicals reactive molecules that cause oxidative damage to cells. Their effectiveness in countering these radicals suggests that they may help to protect cells against oxidative stress and related health problems, which may also be behind the current findings (Oniga et al., 2018; Stoilova et al., 2008). Evaluating this K232 parameter, Kehili et al. (2019) reported the same behaviour when tomato peels were added to refined olive oil using the same enrichment method (maceration) (Kehili et al., 2019).

3.3.4. Secondary oxidation: p-anisidine value and K270

To monitor the generation of primary oxidation products, *p*-anisidine and K270 are used (Gharby & Charrouf, 2022).

p-anisidine value provides data on the kinetics involved in the generation of secondary oxidation by-products, specifically carbonyl compounds (aldehydes and ketones), which have an adverse impact on the flavor and aroma of oils. Generally, a lower *p*-AV indicates higher oil quality (Zahran & Najafi, 2020). Although the *p*-anisidine value is determined more empirically, it is thought to have a strong correlation with the amount of secondary oxidation products, such as aldehydes, which are more persistent than hydroperoxides (Miguel et al., 2014). The *p*-AVs as recorded are depicted in Fig. 4 (C). For all samples, *p*-anisidine levels typically rose during the accelerated ageing at 60 °C. Once enriched (inittial 0) and over 120 days of oxidation monitoring, the enriched oils showed lower *p*-AVs than the control ($p < 0.05$). The *p*-anisidine recorded values after two months are 5.12, 2.34 and 2.25 for control, EOTL (5%) and EOTL (10%) respectively. This behaviour of lowering the intensity of secondary oxidation lasted throughout the four months of storage except that after 90 and 120 days, EOOL (10%) showed a greater increase than control, although EOTL at both concentrations continued to show *p*-AVs less than unenriched oil. Whereas the lowest values were recorded by EOTLs, with amounts that did not exceed 5.41 for EOTL 5% and 2.97 for EOTL 10%. Among the two added plants, the addition of TL had a more relevant effect in slowing down the production of secondary oxidation products than that of OL. This may be linked to the preservative action of the bioactive phenolic compounds of thyme which was found to be significantly higher than that of the used oregano. Such results may be explained by the power of phenolic compounds in the preservation of lipids, which has already been demonstrated by Miguel et al. (2014) on fried olive and sunflower oils reporting a reduce in *p*-AVs in enriched oils with *Thymbra capitata* essential oil (Miguel et al., 2014). Moreover, using maceration as the enrichment technique, the enriched soybean oil samples showed the least intense *p*-AVs than the control (Nid Ahmed, Abourat, et al., 2024; Nid Ahmed, Gagour, et al., 2024).

Beside the *p*-AV, the ultraviolet absorption measured at $\lambda = 270$ nm serves as a crucial marker for the presence of secondary oxidation products in oils and fats. It also functions as an indicator of antioxidant effectiveness in preserving and stabilizing the oil (Bijla et al., 2024).

An increase in K270 over the 4 months period indicates that thermooxidation is triggering the generation of secondary oxidation products (Fig. 4 (C)). At the start of the accelerated storage period (D0), the addition of OL and TL increased values of the conjugated trienes (K270). However, during the time spent storing, control oils marked an increase in the concentration of secondary oxidation products compared to the EOTLs. However, from day 0, enrichment of the AO with *Origanum vulgare* L. resulted in a significantly higher elevation of K270 values compared to the control. When, *Thymus vulgaris* L. provided very significant protection, since EOTLs showed lower K270 values than the untreated samples, offering a strongest oil protection effect especially with 10% amount confirming the results previously found in *p*-AV. At the end of storage (120 days) EOTL 5% presented 0.36 value of K270 while control reached 0.47. Enriching AO with TL and OL can reduce the production of oxidation by-products through various antioxidant mechanisms. The antioxidant compounds present in thyme and oregano neutralize free radicals, such as peroxyl radicals (ROO•), by providing electrons or protons to stabilise these reactive molecules, preventing them from interacting with unsaturated lipids. By capturing free radicals, these antioxidants interrupt the oxidation chain reactions that lead to the formation of peroxides and, subsequently, secondary products such as ketones and aldehydes. In addition, rosmarinic acid and other antioxidants present in these herbs also act by scavenging reactive oxygen species (ROS), such as peroxides, reducing their concentration in the oil and limiting their ability to cause further oxidation reactions (secondary oxidation), by inhibiting the formation of peroxides and neutralizing free radicals as soon as they are formed (Brewer, 2011). Many studies have confirmed that the natural antioxidants can avoid the thermal degradation of vegetable oils by inhibiting the formation of conjugated triple bonds and improving their hydrolytic stability, such as the study of Bijla et al. (2024) who have found that coffee grounds could be used as an effective natural antioxidant to replace the synthetic

antioxidants in industrial processes (Bijla et al., 2024).

3.3.5. Total oxidation (TOTOX)

The total oxidation (TOTOX) value theoretically predicts the oil's oxidative stability by quantifying both primary and secondary oxidation products. Due to its incorporation of both oxidation indexes (PV and *p*-AV), the TOTOX rate provides a comprehensive evaluation, making it an effective measure for assessing the overall oxidation and freshness of an oil (Nid Ahmed, Abourat, et al., 2024; Nid Ahmed, Gagour, et al., 2024). Throughout the processing period, all evaluated samples showed a gradual increase (Fig. 4 (D)). The TOTOX value of the control oil at the initial state (0 D) was 14.37, reaching a maximum value of 45.33. In contrast, the enriched oils exhibited TOTOX values of 43.45, 38.93, 11.73, and 9.63 for EOOL 5%, EOOL 10%, EOTL 5% and EOTL 10% respectively. EOOL's TOTOX index revealed a modest increase than the sample control at the end of the third month (90 days) ($p < 0.05$), but this dropped right away after 120 days. Also, the addition of TL was more effective in controlling total oxidation compared to OL. Overall, the combination of flavonoids, tannins, saponins, and phenolic acids in thyme and oregano provides a multifaceted approach to protect AO from oxidation. These compounds work synergistically to neutralize free radicals, chelate metal ions, and form non-reactive complexes, thereby enhancing the stability and longevity of the oil. These findings coordinate with those of Abdo et al. (2023) who reported that using pomegranate/orange/beetroot leaf extracts to enrich soybean oil during deep-frying reduced the TOTOX value by 55% compared to the raw oil (Abdo et al., 2023). Besides, Nid Ahmed, Abourat, et al. (2024) and Nid Ahmed, Gagour, et al. (2024) observed a comparable behaviour in the TOTOX index when they enriched sunflower oil (*Helianthus annuus* L.) with saffron (*Crocus sativus* L.) stigmas through maceration (Nid Ahmed, Abourat, et al., 2024; Nid Ahmed, Gagour, et al., 2024).

3.4. Chlorophylls content

Chlorophylls, the most widely prevalent natural pigment, is found in the leaves and various other parts of virtually all plants (Humphrey, 2004). The quality of vegetable oils is also affected by the content of chlorophylls, whose high antioxidant activity determines the oxidative stability of oils and prevents their degradation (Lapčíková et al., 2018). Fig. 5 illustrates the variations in chlorophyll content over the storage period for both the control and the enriched oils. Highly significant ($p <$ 0.05) variations in chlorophyll content were observed between the enriched samples and the control, the reported control oil content was 0.072 ppm, while EOOL 5% attained value of 6.105 ppm, which represents 98% improvement. In all oil samples, the chlorophyll content decreased during oxidation through accelerated ageing, suggesting that chlorophylls undergo decomposition during storage under elevated temperatures, which has already been proved by Liu et al. (2022) (Liu et al., 2022). Moreover, the chlorophyll levels attenuated more prominently in samples enriched with *Origanum vulgare* L. than those enriched with *Thymus vulgaris* L, these findings can be elucidated by considering the chlorophyll content of oregano, which was observed to be greater than that of thyme (Ansar et al., 2020). The maceration method used may also be the reason for this increase in chlorophylls in AO, since it enables a dissolution that is favored by prolonged contact and agitation, allowing the chlorophyll molecules to separate from the cellular structures of the herbs and mix with the oil. As well, this solubility is due to the chemical structure of chlorophyll, which comprises a long hydrophobic (non-polar) chain that interacts favorably with vegetable oils (Humphrey, 2004). Elsewhere, these findings offer a plausible explanation for the previously observed outcomes in the quality indices, aligning with the study conducted by Jaber et al. (2012). The mentioned study examined the oxidative stability of refined olive oil following the incorporation of chlorophylls extracted from Chemlali olive leaves. Notably, their results indicated that oils supplemented with the leaf pigment extract exhibited the lowest PVs, thereby showcasing superior stability (Jaber et al., 2012).

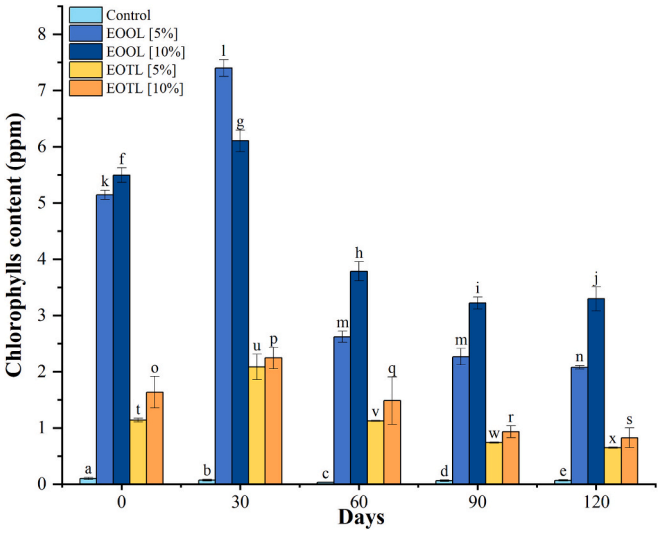

Fig. 5. Chlorophylls content (ppm) of control, enriched oil with oregano leaves (EOOL) and enriched oil with thyme leaves (EOTL) over 120 days of storage under accelerated ageing conditions (60 °C) at two concentrations (5 and 10%). Bars with different letters are significantly different at $p < 0.05$.

3.5. Fatty acids composition and iodine value (IV)

Fatty acids constitute crucial elements within the saponifiable fraction of vegetable oils. This group of compounds serves as a vital gauge for assessing the nutritional value of the oil, especially in terms of its appropriateness for cosmetic applications, with a particular emphasis on unsaturated fatty acids (Gharby et al., 2018). Consequently, we employed this analytical approach to examine how enrichment with OL and TL affects the composition of AO at the beginning (0 day) and after 120 days of the thermoxidation test. The obtained results are shown in Table 3. At the initial enrichment stage, no significant difference ($p <$ 0.05) was observed even at the highest concentration (10%) of OL and TL. With values of 14.21 ± 1.10, 16.16 ± 1.20, 15.28 ± 1.10, 14.44 ± 1.40 and 15.81 ± 1.08 g/100 g for control, EOOL (5%), EOOL (10%), EOTL (5%) and EOTL (10%). These results have remained unchanged despite four months of accelerated ageing (120 days). Overall, the levels of fatty acids (both saturated and unsaturated) stayed constant throughout the entire storage period. These findings align with prior research of Drinić et al. (2020) by adding pomegranate peel extract to pomegranate seed oil, indicating a limited impact of accelerated ageing conditions on the content of fatty acids (Drinić et al., 2020). Contrastingly, Jorge et al. (2015) observed a gradual decrease in fatty acids following the enrichment with oleoresin derived from extracts of *Thymus vulgaris* L. and *Origanum vulgare* L, the reductions were most prominent in linoleic fatty acid, demonstrating the lowest levels after enrichment (Jorge et al., 2015). Regarding the iodine value (IV), it's an index that gives an idea on the degree of unsaturation by determining the number of double bonds in fatty acids (El Bernoussi et al., 2020). As illustrated in Fig. 6, the iodine results showed no significant difference either before or after enrichment, which is unsurprising as the IV is computed based on FAs, and their composition has remained constant.

3.6. Correlation study

The correlation matrix presented reveals significant relationships between various chemical and physical properties of the samples studied (Fig. 7). The results showed a strong positive correlation between PV and other quality indices such as (*p*-AV, $r = 0.75$; K232, $r = 0.78$; K270, $r = 0.94$). In addition, polyphenols and antioxidant compounds,

Table 3
Fatty acids composition (g/100 g) of control, enriched oil with oregano leaves (EOOL) and enriched oil with thyme leaves (EOTL) with both amounts 5 and 10%. In each line, values followed by the same letter are not significantly different at $p < 0.05$.

Fatty acids	Control		EOOL (5%)		EOOL (10%)		EOTL (5%)		EOTL (10 %)	
	Initial	Final	Initial	Final	Initial	Final	Initial	Final	Initial	Final
C16:0	$14.21^{a} \pm 1.10$	$15.11^{a} \pm 1.00$	$16.16^{ab} \pm 1.20$	$14.74^{a} \pm 1.20$	$15.28^{a} \pm 1.10$	$15.12^{a} \pm 1.00$	$14.44^{a} \pm 1.08$	$14.75^{a} \pm 1.00$	$15.81^{a} \pm 1.40$	$15.12^{a} \pm 1.00$
C18:0	$5.31^{a} \pm 0.10$	$5.46^{a} \pm 0.00$	$5.03^{a} \pm 0.10$	$5.37^{a} \pm 0.10$	$5.09^{a} \pm 0.10$	$5.27^{a} \pm 0.10$	$5.31^{a} \pm 0.10$	$5.06^{a} \pm 0.10$	$5.37^{a} \pm 0.10$	$5.24^{a} \pm 0.10$
C18:1	$46.80^{a} \pm 2.10$	$46.00^{a} \pm 2.10$	$45.53^{ab} \pm 1.10$	$44.85^{abc} \pm 2.10$	$46.11^{a} \pm 2.10$	$46.00^{a} \pm 2.10$	$46.53^{a} \pm 2.10$	$45.86^{ab} \pm 1.10$	$45.82^{ab} \pm 2.10$	$46.23^{a} \pm 2.10$
C18:2	$33.44^{a} \pm 1.11$	$33.26^{a} \pm 1.00$	$32.65^{ab} \pm 2.10$	$34.24^{ab} \pm 1.80$	$33.02^{a} \pm 1.10$	$33.19^{ab} \pm 1.00$	$33.70^{a} \pm 1.90$	$34.09^{a} \pm 1.97$	$32.99^{a} \pm 1.25$	$33.14^{a} \pm 1.50$
Others	0.24	0.17	0.63	0.80	0.50	0.42	0.53	0.24	0.01	0.27
SFA*	19.52	20.77	21.19	20.11	20.37	20.39	19.75	20.11	21.18	20.36
USFA**	80.24	81.17	78.18	79.09	79.13	80.57	80.23	79.95	78.81	79.37

SFA*: saturated fatty acids; USFA**: unsaturated fatty acids.

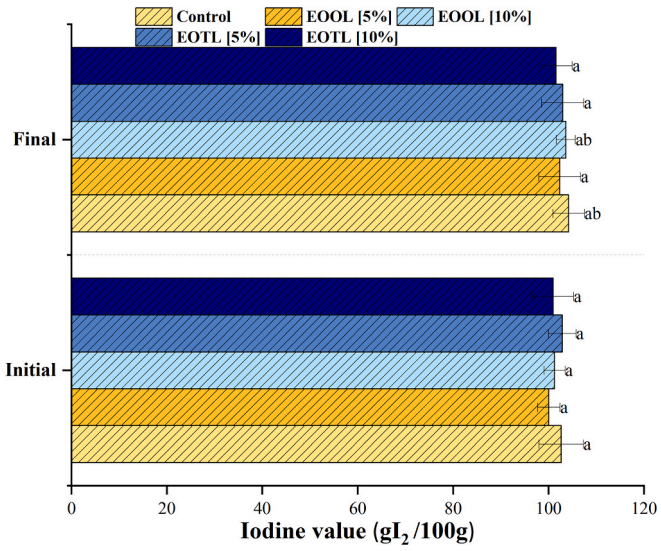

Fig. 6. Iodine value (g I_2/100g) of the control, enriched oil with oregano leaves (EOOL) and enriched oil with thyme leaves (EOTL) over 120 days of storage under accelerated ageing conditions (60 °C) at two concentrations (5 and 10%). Bars with different letters are significantly different at $p < 0.05$.

measured by SPC, TFC and DPPH•, are highly correlated between each other ($r \geq 0.78$). Chlorophylls show a notable positive correlation with DPPH• ($r = 0.64$), suggesting their influence on antioxidant activity. IV also show a positive correlation with oxidation parameters. Moreover, IP with polyphenols (SPC and TFC), DPPH• and chlorophylls are positively correlated ($r \geq 0.72$), this means these compounds help protect oils from deterioration, improving their quality and stability and extending their shelf life. On the other hand, SFA, USFA and IV are positively correlated with oxidation parameters. In contrast, the negative correlation between FFA and parameters such as PV ($r = -0.30$) indicates that there is an inverse relationship between the amount of free fatty acids and that of primary oxidation products. SPC, TFC and DPPH• are also negatively correlated with primary (PV and K232) and secondary (*p*-AV and K270) oxidation parameters, suggesting a crucial role for phenolics and antioxidants in reducing oxidation. Finally, negative correlations between SFA, USFA and IV with various parameters (SPC, TFC and DPPH•).

3.7. Sensoriel attributes

Established standards for edible oils, like the Codex Alimentarius Standard for Named Vegetable Oils (Codex Alimentarius, 1993) emphasize sensory evaluation as the primary parameter for gauging the quality of edible oils. Accordingly, sensory analyses were carried out after four months of accelerated thermooxidation. The parameters evaluated are: overall acceptance, rancidity, and color, aroma, and flavor of the added plant are presented in the form of radars for oils enriched with oregano (Fig. 8, (a)) and thyme (Fig. 8, (b)).

When evaluating the **overall acceptance** of the oils, the panels showed a preference for the 5% enrichment. In contrast to the 10% enrichment, the AO gold's appearance did not generally exhibit a distinct change. Sensory quality of vegetable oil can also deteriorate during storage, giving rise to a modification in odour known as **rancidity** (Matthäus et al., 2010). This attribute was stated in the control oils and in the EOTL 5%, while other oils enriched with either OL or TL did not manifest this rancid odour, which may be linked to the fact that the high quantities of antioxidant compounds in thyme hinder the production of volatile substances resulting from lipid oxidation, as already proven by (Qi et al., 2022). For the three other parameters relating to *Thymus vulgaris* L. and *Origanum vulgare* L., the green **color** of the added leaves was very intense in the oils enriched with 10%, with a score of 4/4 for both plants. The judges were able to recognize the added spice by the aroma and distinguish the concentrations used. In fact, the **aroma** created by OL was more accentuated than that of TL, with a score of 4/4 and 3/4 respectively. A concentration dependent effect of the **flavor** of thym and oregano was found in the fortified oils, meaning that the oils enriched with the highest concentation have the strongest flavor. Also, in comparison to thyme, oregano is much more flavorful with a subtle peppery aftertaste. The outcomes of these three parameters can be linked to the aromatic compounds found in thyme and oregano, such as thymol, carvacrol, and linalool. These compounds are typically lipophilic (hydrophobic), which means they dissolve readily in fatty or oily substances. Since AO is a lipid matrix rich in fatty acids, it creates an optimal environment for the dissolution of these compounds. One of the advantages of this method is that everyone can benefit from its virtues, as it can be easily applied at home, making the enrichment of AO by maceration an economically viable process due to its simplicity, low equipment costs and ability to offer customised, high-quality formulations, bearing in mind that the unit cost of the two plants used to make the product is low. So, by combining these advantages with a marketing strategy focused on the added value and unique properties of the product, this method is proving to be an effective and profitable solution for capturing consumer interest and maximising financial returns.

4. Conclusion

In conclusion, the study demonstrated that enrichment of AO with TL and OL effectively reduced the production of peroxides, ketones, and aldehydes, thereby maintaining the oil's quality and minimizing sensory alterations such as rancid odors, with a moderately superior anti-oxidant effect in TL. The maceration method employed, being environmentally friendly, offers significant economic advantages due to its low

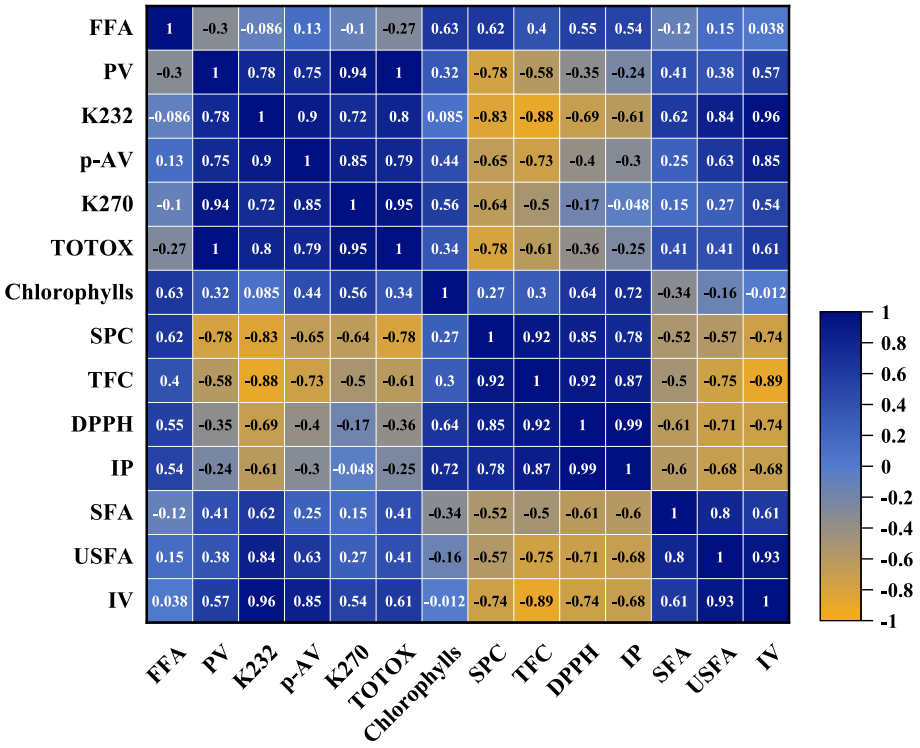

Fig. 7. Correlation coefficients among studied parameters.

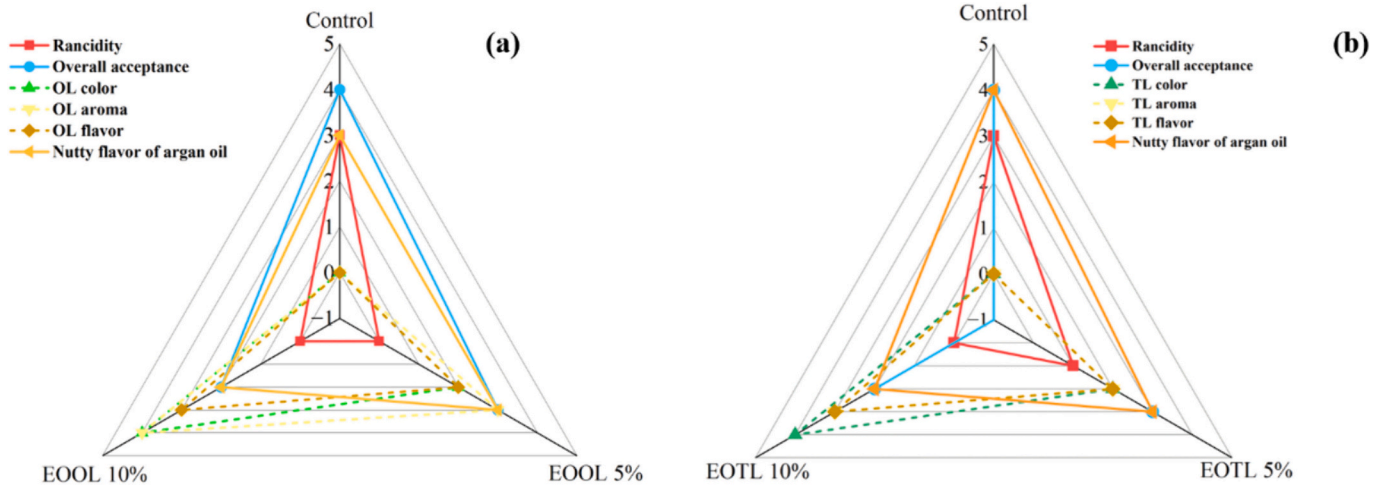

Fig. 8. Sensory attributes of control, enriched oil with oregano leaves (EOOL) **(a)** and enriched oil with thyme leaves (EOTL) **(b)** with both amounts 5 and 10%.

complexity relative to more sophisticated enrichment techniques. The necessary equipment is relatively simple and inexpensive, which helps lower production costs and allows for competitively priced products while preserving a healthy profit margin. This method is thus suitable for adoption by manufacturers, cooperatives, and for everyday culinary applications. The selection between thyme and oregano can be tailored to consumer preferences for taste, aroma, and flavor. Future research should explore additional biological activities and the nutraceutical value of AOs enriched using various aromatic and medicinal plants.

Sensory ethical statement

As the products involved in this study are safe for consumption, obtaining ethical permission to conduct a sensory study was deemed unnecessary. All participants were fully informed about the nature of the study and provided their consent before participation. This approach ensures that the study adheres to ethical standards while respecting the safety and rights of the participants.

CRediT authorship contribution statement

Samira Oubannin: Writing – original draft, Resources, Investigation, Formal analysis, Data curation. **Abderrahim Asbbane:** Writing – review & editing, Writing – original draft, Software, Resources. **Khang Wen Goh:** Writing – review & editing, Investigation, Funding acquisition. **Jyoti Singh:** Formal analysis, Funding acquisition, Investigation, Validation. **Iqbal Zafar:** Funding acquisition, Investigation, Visualization, Writing – review & editing. **Abdelhakim Bouyahya:** Writing – review & editing, Investigation, Data curation. **Said Gharby:** Writing – review & editing, Supervision, Project administration,

Conceptualization.

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.

Data availability

Data will be made available on request.

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