

Kinetic study of the microwave-induced thermal degradation of cv. Arbequina olive oils flavored with lemon verbena essential oil

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Abstract

The effect of typical domestic microwave heating (0–15 min, at 800 W) on the thermal degradation of unflavored and flavored olive oils' minor bioactive compounds and related antioxidant activity was studied. Olive oils from cv. Arbequina were flavored with lemon verbena essential oil (0%, 0.2% and 0.4%, w/w) leading to a linear increase of total phenols (112–160 mg gallic acid kg⁻¹ oil, *R*-Pearson = +0.9870), total carotenoids (2.19–2.56 mg lutein kg⁻¹ oil, *R*-Pearson = +0.9611), and, to a less extent, of chlorophyll (2.32–3.19 mg pheophytin kg⁻¹ oil, *R*-Pearson = +0.8238). However, no such linear trend was observed for the oxidative stability (6.5–7.8 h) or the radical scavenging activity (inhibition rates: 40%–43%). The contents of total phenols, total carotenoids, and chlorophyll decreased with the rise of the microwave heating time, following their thermal degradation, a second-order kinetic model (0.8784 ≤ *R*-Pearson ≤ 0.9926). The essential oil addition did not influence the estimated second-order rate reaction constants of total phenols (0.00070–0.00072 kg oil min⁻¹ mg⁻¹ gallic acid) and total carotenoids (0.14–0.17 kg oil min⁻¹ mg⁻¹ lutein), with a broader variation observed for chlorophyll (0.014–0.022 kg oil min⁻¹ mg⁻¹ pheophytin). Globally, total carotenoids degraded faster than total phenols and chlorophyll (half-life of 2.3–3.4, 8.8–12.8, and 14.5–30.8 min, respectively). Moreover, except for chlorophyll, the half-life of total phenols and carotenoids linearly decreased with the essential oil addition (*R*-Pearson: –0.9999 and –0.9421, respectively), showing that flavoring did not have a protective effect against degradation when subjected to a microwave heating.

KEYWORDS

bioactive compounds, degradation rate reaction constant, essential oil flavoring, half-life period, olive oil

INTRODUCTION

Olive oil (*Olea europaea* L.) is highly appreciated and a key element of the Mediterranean diet, being its intake associated to several health benefits mainly due to the rich composition of monounsaturated fatty acids and minor constituents like phenolic compounds and carotenoids (Emma et al., 2021; Pocovi-Gerardino et al., 2021). Olive oil is usually consumed as a raw product, in salads, or in foods after being adsorbed, when used as a cooking oil (Chiou & Kalogeropoulos, 2017). As described in the literature, olive oil is resistant to oxidative deterioration

when cooked (e.g., used as a frying oil), which is attributed to the high monounsaturated and low polyunsaturated fatty acid contents together with its composition in minor bioactive constituents that protect olive oil from extensive oxidative deterioration at high temperatures (Chiou & Kalogeropoulos, 2017). However, several studies pointed out the negative impact of different heating processes (e.g., frying, baking, boiling, oven/conventional, and microwave heating) on the olive oil's chemical profile and composition (volatiles and phenolic compounds) as well as on the positive and appreciated sensory sensations (Jiménez et al., 2017; Prata et al., 2018). Still, olive

oil is commonly used in both “healthy” (defined as habit of cooking meals with fresh and natural seasoning foods) and “convenience” (defined as less frequency and time to cook, using microwave and ready-to-heat meals) cooking patterns by worldwide consumers (Martins et al., 2021). Aiming to reduce the natural (due to storage) or accelerated (due to cooking) thermal degradation of olive oils as well as to meet sensory preferences of olive oil worldwide consumers, flavoring with aromatic herbs, spices, or essential oils (EO) has emerged as a commercial trend (Baiano et al., 2009; Caponio et al., 2016; Khemakhem et al., 2015). Several studies reported the kinetic modeling of bioactive compounds' degradation rates during thermal processing and/or storage, namely, for example, of saffron flowers and related bio-residues (Moratalla-López et al., 2019), common beans (Mba et al., 2019), *Piper betle* extracts (Ali et al., 2018), Brussels sprouts (Nakilcioglu-Taş & Otleş, 2017), sea buckthorn (Ursache et al., 2017), plum extracts (Turturică et al., 2016), fresh fruit juices (Marszałek et al., 2015; Sapei & Hwa, 2014), and apple peel by-products (Henríquez et al., 2014). Regarding olive oils, to the authors' best knowledge, only one study evaluated the thermal degradation kinetics of individual carotenoids (e.g., lutein, β -carotene and β -cryptoxanthin) of a blend virgin olive oil (cvs. Hojiblanca and Manzanilla) subjected to different time-temperature conditions (varying from 42 h at 120°C to 744 h at 60°C) (Aparicio-Ruiz et al., 2011).

Thus, this study aims to evaluate the thermal degradation of cv. Arbequina oils, a worldwide widespread olive cultivar, during typical microwave heating process simulating a usual domestic use as well as the possible protective effect of oils' flavoring with lemon verbena EO (*Aloysia citrodora*) (0%, 0.2% and 0.4%, w/w). This EO is recognized as one of the most valuable aromatic and medicinal plants with known positive health effects (Bahramsoltani et al., 2018; Combrinck et al., 2019). Recently, it was observed that flavoring cv. Arbequina oils with lemon verbena EO increased the antioxidant and total reducing capacities but decreased the oxidative stability (OS) (Cherif et al., 2021), and it was found that the oils' oxidation at high temperatures (110–150°C) had a nonspontaneous, endothermic, and endergonic nature. In this sense, several parameters were evaluated namely the free acidity (FA), peroxide value (PV), extinction coefficients at 232 and 270 nm, K_{232} and K_{270} , as well as the radical scavenging antioxidant activity, total phenols content (TPC), total carotenoids content (TCC), and chlorophyll content. Besides, the degradation rates of the minor constituents (TPC, TCC, and chlorophyll) were also evaluated during the microwave heating at five usual domestic time periods (0, 1, 3, 5, 10, and 15 min). For that, a kinetic approach was used, aiming, for the first time, to establish the half-life times of these compounds of unflavored and flavored cv. Arbequina oils subjected to common microwave heating (cooking) patterns.

MATERIALS AND METHODS

Olive oil samples and EO

The cv. Arbequina extra virgin olive oils were produced from olives grown in an olive grove with high-density production mode located in Trás-os-Montes region. The olives were collected in 2019, and the oil was extracted in an olive mill (Olimontes) located in Macedo de Cavaleiros (Portugal), using a two-phase centrifugation system. The olive oils were kept in their original amber glass bottles (approximately 500 ml), during 4 months, in a dark environment, at room temperature (18–25°C), and were filtrated before analysis. Then lemon verbena EO was added in different amounts allowing to obtain flavored oils with 0.2% and 0.4% (w/w) of EO. In total, two independent closed amber glass bottles (with approximately 100 ml of oil) were also stored in dark at 18–25°C during 2 weeks before being analyzed, for each flavoring concentration, plus other two bottles without no EO addition (0.0%, w/w), used as the control samples. Two weeks after being flavored, the oil from each independent bottle was analyzed in duplicate after being subjected to five microwave heating time-periods (0, 1, 3, 5, 10, and 15 min, which corresponded to approximately, 20, 41, 115, 160, 205, and 235°C), at a preset power of 800 W, using a microwave Amstrad (model WP810, London, UK), totalizing 60 assays for each evaluated parameter (i.e., 3 EO concentrations \times 2 independent bottles \times 5 heating time-periods \times 2 replicates = 60 assays). Four quality parameters (FA, PV, and extinction coefficients at 232 and 270 nm), the antioxidant activity, the OS at 120°C, TPC, TCC, and chlorophyll content were assessed. The EO was purchased in a local specialty store devoted to the commercialization of EOs for the food industry. According to the label information, the EO had a lemony profile and sweet fresh aroma. All flavored oils could be easily recognized by nontrained consumers due to the perception of a lemony profile and sweet fresh taste/aroma (sensory attributes in accordance with the EO label). Lemon verbena EO was chosen due to its well-documented richness in polyphenols (Ishkeh et al., 2019; Rocha et al., 2019). The volatile composition of the lemon verbena EO was previously reported by Cherif et al. (2021) and was in line with the literature data, and no sign of deterioration was visible.

Quality parameters and OS of unflavored and flavored olive oils

The physicochemical quality parameters, the FA (in % oleic acid), PV (in mEq O₂/kg), and specific coefficients of extinction at 232 nm and 268 nm (K_{232} , and K_{268}) were evaluated according to the European Union

Regulation (Commission Delegated Regulation [EU] 2015/1830, 2015). The OS (in h) was determined under accelerated oxidation conditions (120°C) using the Rancimat method (Rancimat 743, Metrohm CH, Herisau, Switzerland) as previously described (Veloso et al., 2020). Briefly, 3 g of olive oil was heated at $120.0 \pm 1.6^\circ\text{C}$ under a flow rate of 20 L/h of air (filtered, cleaned, and dried), releasing volatile compounds that are collected in water, water conductivity (mS/cm) being continuously measured and plotted against time. The time (in hours) taken to reach the conductivity inflection curve point is determined, representing the OS of the oil.

Chlorophyll and carotenoids contents of unflavored and flavored olive oils

Chlorophyll and TCC were spectrophotometrically determined as previously described (Minguez-Mosquera et al., 1991). For the procedure, olive oil (7.5 g) was vortexed with cyclohexane (final volume of 25 ml) for 30 s. After homogenization, the mixture was spectrophotometrically analyzed, at 470 and 670 nm for carotenoids and chlorophyll assessment, respectively. Chlorophyll content was expressed as mg of pheophytin equivalent per kg of oil and carotenoids contents as mg of lutein equivalent per kg of oil. The specific extinction coefficients considered for the calculation were 613 for pheophytin, as a major component of the chlorophyll fraction, and 2000 for lutein, as a major component of the carotenoid fraction. The pigments' contents were then calculated as follows (Equations (1) and (2)):

$$\text{Chlorophyll} \left(\frac{\text{mg pheophytin eq.}}{\text{kg oil}} \right) = \frac{\text{Abs}_{670} \times 10^6}{613 \times 100 \times d} \quad (1)$$

$$\text{Carotenoids} \left(\frac{\text{mg lutein eq.}}{\text{kg oil}} \right) = \frac{\text{Abs}_{470} \times 10^6}{2000 \times 100 \times d} \quad (2)$$

where *Abs* is the absorbance at 670 or 470 nm and *d* is the cuvette path length (equal to 1 cm).

TPC and antioxidant activity of unflavored and flavored olive oils

TPC and the radical scavenging activity were assessed using olive oils' extracts obtained by micro methanol–water extraction (Pizarro et al., 2013). Briefly, in a 2 -ml Eppendorf tube, 1 ml of methanol–water solution (80% MeOH-H₂O, v / v) was added to 0.5 g of olive oil. The mixture was vortexed for 1 min and then centrifuged during 5 min at 13,200 rpm (Minispin 5452 Eppendorf centrifuge). The supernatant (extract) was recovered into a

3-ml volumetric flask. The procedure was repeated twice, after the addition of 1 ml of the 80% MeOH-H₂O solution in the same Eppendorf centrifuge. The three extracts were collected and placed in the same 3-ml volumetric flask, which was then filled with ultrapure water. Each olive oil sample was extracted in triplicate.

For TPC assessment, 1.5 μl of ultrapure water, 0.1 μl of the phenolic extract, and 0.100 μl of Folin–Ciocalteu reagent were vortexed for 3 s and allowed to react for 3 min. After that, 0.300 μl of 20% sodium carbonate (w/v) solution was added, vortexed for 3 s, and further allowed to react for 60 min in the dark and at room temperature (20–22°C). This procedure was carried out in triplicate, for each olive oil extract obtained according to the micro methanol–water extraction. All samples were spectrophotometrically evaluated (UV–VIS/UV-1280 Shimadzu spectrophotometer) at 765 nm. TPC quantification (in mg of gallic acid equivalents [GAE] per kg of oil) was carried out using a calibration curve between the absorbance and the GA concentration in methanol solution ($R^2 \geq 0.9999$).

The antioxidant capacity of olive oil samples was investigated and the radical scavenging activity (2,2-diphenyl-1-picrylhydrazyl) (DPPH) was assessed spectrophotometrically at 517 nm (UV–VIS/UV-1280 Shimadzu spectrophotometer), at 20°C, using the extract solutions obtained according to the micro methanol–water extraction. The DPPH control solution was prepared by mixing 0.1 ml of methanol and 3.9 ml of DPPH (0.06 mM), and the absorbance is being read after 30 min in the dark. Similarly, for the olive oil samples, 0.1 ml of methanolic extract previously obtained was added to 3.9 ml of DPPH (0.06 mM), and the absorbance is being read after 30 min in the dark. The DPPH radical scavenging was expressed as the percentage of reduction of DPPH activity.

Kinetic modeling of the thermal degradation reactions

The observed changes on the contents of phenols, carotenoids, and chlorophyll (expressed in mg/kg) were used to calculate the thermal degradation reaction rates due to the microwave heating of unflavored and flavored olive oils. Three reaction orders were considered (zero-, first- and second-order kinetic models, Equation (3)), being selected the kinetic model that allowed the best mathematical fit of the experimental data for the three oil types studied:

$$-\frac{dC_i}{dt} = k \times C_i^n \quad (3)$$

where C_i is the content (in mg/kg of oil) of the *i* compound under analysis (TPC, TCC, or chlorophyll); *t* is the microwave heating time-period (in min); *k* is the

rate constant; and n is the reaction order (equal to 0, 1, or 2 for zero-, first-, and second-order kinetic models, respectively).

The integration of the previous equation is straightforwardly assuming that k is constant during the entire microwave heating time-period. Indeed, a pseudo-constant k can be assumed, although the olive oil temperature increases during the heating time-period, as previously stated. Since a commercial home device was used to mimic the usual domestic microwave use, it is not possible to fix a constant temperature during the entire heating time-period, only being possible to fix the heating power, which turns out into an increasing heating temperature during the process. However, and as described in the literature, the microwave absorption is closely related to the moisture content of the samples under analysis and so, since the same sample weight (m) was used in each assay and heating was conducted at a predefined and fixed microwave power (P), the following equation can be used to describe the dependency of k with m , P , and the energy of activation (E_a), based on the Arrhenius equation (Dadali et al., 2007; Demirhan & Özbek, 2015; Kumar et al., 2019):

$$k = k_0 e^{\left(-\frac{E_a}{Pm}\right)} \quad (4)$$

where k_0 is the pre-exponential constant.

Therefore, since m and P were the same in all assays, k can be assumed as a constant allowing the usual integration of the empirical kinetic models (Equations (1)–(3)). This approach is in line with different researchers when studying the impact of the domestic microwave heating on the degradation kinetics of bioactive compounds of several food matrices (Dadali et al., 2007, 2009; Demirhan & Özbek, 2009; Demirhan & Özbek, 2015; Igoumenidis et al., 2011; Jaiswal & Abu-Ghannam, 2013; Aamir & Boonsupthip, 2017; Javidipour et al., 2017; Nakilcioglu-Taş & Otleş, 2017; İltir et al., 2018; Kumar et al., 2019; Yarahmadi et al., 2020).

Thus, depending on the considered reaction order, for each parameter (TPC, TCC, or chlorophyll), three relationships (Equations (5)–(7)) can be established between the content and the heating time, after integrating Equation (3),

$$\text{zero – order kinetic model } (n = 0) : C_0 - C = k \times t \quad (5)$$

$$\text{first – order kinetic model } (n = 1) : \ln\left(\frac{C_0}{C}\right) = k \times t \quad (6)$$

$$\text{second – order kinetic model } (n = 2) : \frac{1}{C} - \frac{1}{C_0} = k \times t \quad (7)$$

where C_0 is the content (in mg/kg) of TPC, TCC, or chlorophyll for the unheated olive oils (microwave

heating time equal to 0 min for each flavored or not olive oil).

The linear regression in each case (i.e., $C_0 - C$ vs. t ; $\ln(C_0/C)$ vs. t ; or, $1/C - 1/C_0$ vs. t , for the zero-, first-, or second-order kinetic model, respectively) would allow to estimate k , as the slope value of each regression, as well as the respective standard error (SE). It should be remarked that the units of k depend on the model used, being mg/kg min⁻¹; min⁻¹; and, kg min⁻¹ mg⁻¹ for the zero-, first-, and second-order rate constants. Finally, half-life ($t_{1/2}$, in min) of each compound, for unflavored and flavored olive oils, were estimated as the microwave heating time needed to decrease each content by 50% from its initial value ($C = 0.5 \times C_0$, in mg/kg), based on the previous kinetic models (Equations (5)–(7)), depending on the best-fitting model to the experimental data:

$$\text{zero – order kinetic model } (n = 0) : t_{1/2} = \frac{0.5 \times C_0}{k} \quad (8)$$

$$\text{first – order kinetic model } (n = 1) : t_{1/2} = \frac{\ln(2)}{k} \quad (9)$$

$$\text{second – order kinetic model } (n = 2) : t_{1/2} = \frac{1}{k \times C_0} \quad (10)$$

Statistical analysis

Two-way analysis of variance (ANOVA) with interaction was applied to evaluate the statistical significance of olive oil flavoring, with lemon verbena EO (0%, 0.2%, and 0.4%, w/w) and microwave heating times (0, 1, 3, 5, 10, and 15 min) on the cv. Arbequina oils' FA, peroxide value, extinction coefficients, OS, antioxidant activity, total phenols, total carotenoids, and chlorophyll contents. Depending on the statistical significance of the interaction effect and on the type of interaction found (additive or nonadditive/disordinal effect) post hoc multiple range tests or the estimated marginal mean plots were further used to discuss at which level the factors influenced the dependent variables under study (Field, 2009). Furthermore, a significant statistical interaction effect was assumed as additive if no crossing lines were observed on the estimated marginal mean plots representing the changes observed for both effects considered, for each of the parameters under study. The significance level was set at 5%. The Pearson correlation coefficient (R -Pearson) was calculated to infer about the existence of correlation trends of the oils' characteristics and the EO addition. Principal component analysis (PCA) was used, as an unsupervised multivariate pattern recognition technique, to further

evaluate the type of oil-microwave heating times' differentiation capability based on the observed changes of the evaluated parameters. Linear regressions were also used to calculate the reaction rate constants of the thermal degradation of TPC, TCC, and chlorophyll content, which were performed after the linearization of the zero-, first-, and second-order kinetic models. The statistical analysis was performed using the subselect and MASS packages of the open-source statistical program R (version 3.6.2) (Cadima et al., 2004; Venables & Ripley, 2002).

RESULTS AND DISCUSSION

Impact of olive oil flavoring and microwave heating on the physicochemical and antioxidant characteristics of cv. Arbequina olive oils

In this study, the possible protective impact of flavoring cv. Arbequina olive oil was evaluated with different amounts of lemon verbena EO (0.2% and 0.4%, w/w), on the oils' quality indices (FA, PV, K_{232} , and K_{268}), OS, antioxidant activity (DPPH), as well as on the TPC, TCC, and chlorophyll, against the thermal degradation during microwave heating (0, 1, 3, 5, 10, and 15 min), by comparing with the observed behavior of unflavored oil (0% EO) under the same heating conditions. Table 1 shows the results according to each flavoring level and microwave heating time. Since two factors (flavoring and microwave heating) are being considered simultaneously, with possible interaction effects, a two-way ANOVA was performed. This analysis allowed, for each parameter studied, to determine the statistical significance of each main factor ("flavoring level" and "microwave heating") as well to verify if the interaction between the two main factors was significant or not. The results are shown in Table 2.

In which concerns the physicochemical quality parameters, the studied factors did not have a significant effect on the FA (p -value ≥ 0.3700 ; Table 2) and also no significant interaction effect was found (p -value = 0.3780; Table 2), demonstrating that cv. Arbequina oils' flavoring did not influence the FA, which was not affected by the different microwave heating times evaluated. These outcomes are in agreement with the previous findings of the research team (Cherif et al., 2021) and in line with the findings of Malheiro et al. (2009), Cerretani et al. (2009), and Yan et al. (2020). Recently, Kishimoto (2019) reported that the FA of olive oils was not affected by the microwave heating time, except when an intense microwave heating was applied (i.e., heating at 500 W, reaching a temperature of 207°C), which led to an FA increase over the heating procedure. On the other hand, Sevim et al. (2018) observed a slight

increase of the FA of olive oils during microwave heating. In contrast, the microwave heating time had significant impact (p -value < 0.0001 ; Table 2) on all the other parameters (PV, K_{232} , K_{268} , OS, DPPH, TPC, TCC, and chlorophyll content) and the EO flavoring level only showed a significant effect on PV, K_{232} , K_{268} , TPC, and TCC (p -value ≤ 0.0014 ; Table 2), pointing out that the thermal degradation was mainly responsible for the observed changes and to a less extent, the flavoring level. The flavoring level did not had a clear effect on the OS, DPPH scavenging activity, TCC and chlorophyll amount, being higher values observed for heating times lower than 5 min. Lastly, higher TPC could be found for higher flavoring levels as well as after lower heating time-periods. These findings should be considered with precaution since, for the abovementioned five parameters, nonadditive significant interaction effects were found (p -value ≤ 0.0117 ; Table 2). For the PV, the results showed a slight decreasing trend with the increase of the flavoring level allowing to admit that the addition of the EO could have a protective effect against the olive oils' oxidative degradation and rancid formation, minimizing the reaction with free oxygen. This trend was contrary to that previously observed by Cherif et al. (2021) for cv. Arbequina also flavored with lemon verbena EO or in other studies (Baiano et al., 2009; Caponio et al., 2016), but similar to the trends reported by other researchers (Bobiano et al., 2019; Tarchoune et al., 2019) for different flavoring agents (e.g., fruits, leaves, dry or fresh spices, EO, or aromatic herbs). No obvious effect could be found regarding the microwave heating impact on the PV of the studied unflavored and flavored olive oils. On the other hand, an increasing trend was found by Malheiro et al. (2009) for unflavored Portuguese olive oils and by Sevim et al. (2018) for Turkish olive oils, subjected to similar microwave heating time-periods (1–15 min and 3–15 min, respectively). Oppositely, Cerretani et al. (2009) reported the decreasing of the PV of Italian olive oils with the increasing microwave heating time (from 1.5 to 15 min), similarly to the findings of Yan et al. (2020). Finally, higher extinction coefficients (K_{232} and K_{268}) were observed for higher flavoring levels as well as for greater heating times, pointing out a possible pro-oxidant behavior enhanced by the thermal degradation due to the rise of both factors, with a rise of primary and secondary oxidation products. In the literature, contradictory effects have been reported regarding the oils' flavoring effect, being described both increasing (Baiano et al., 2009; Caponio et al., 2016; Sacchi et al., 2017) and decreasing (Bobiano et al., 2019; Tarchoune et al., 2019) trends of the evaluated quality parameters with the increase of the flavoring levels. It should be noticed that a similar increasing tendency of the extinction coefficients during the microwave heating

TABLE 1 Free acidity (FA, % oleic acid), peroxide value (PV, mEq O₂/kg), extinction coefficients at 232 and 268 nm (K₂₃₂ and K₂₆₈), oxidative stability (OS, h), radical scavenging activity (2,2-diphenyl-1-picrylhydrazyl [DPPH] inhibition rate, %), total phenolic content (TPC, mg GAE/kg), total carotenoids content (TCC, mg pheophytin equivalents/kg), and chlorophyll content (mg lutein equivalents/kg) of cv. Arbequina olive oils, unflavored or flavored with lemon verbena essential oil (0, 0.2 and 0.4%, w/w) during microwave heating (0, 1, 3, 5, 10, and 15 min), at 800 W of power

Essential oil added to olive oil (w/w)	Microwave heating time (min)	FA (% oleic acid)	PV (mEq O ₂ /kg)	K ₂₃₂	K ₂₆₈	OS (h)	DPPH (%)	TPC (mg GAE/kg)	TCC (mg pheophytin eq/kg)	Chlorophyll (mg lutein eq/kg)
0.0%	0	0.27 ± 0.03	14.54 ± 0.45	2.25 ± 0.04	0.080 ± 0.003	6.5 ± 0.3	40 ± 3	112 ± 2	2.19 ± 0.02	2.32 ± 0.19
	1	0.25 ± 0.03	13.09 ± 0.81	2.40 ± 0.10	0.094 ± 0.015	7.7 ± 0.7	42 ± 3	107 ± 13	2.58 ± 0.18	3.35 ± 0.22
	3	0.25 ± 0.03	13.27 ± 0.03	2.50 ± 0.30	0.066 ± 0.013	6.8 ± 0.6	36 ± 4	109 ± 20	2.47 ± 0.22	3.30 ± 0.36
	5	0.23 ± 0.00	15.40 ± 0.50	2.65 ± 0.15	0.119 ± 0.009	6.1 ± 0.4	34 ± 3	81 ± 4	2.00 ± 0.12	2.55 ± 0.22
	10	0.25 ± 0.03	12.88 ± 0.48	3.13 ± 0.20	0.551 ± 0.032	5.6 ± 0.3	23 ± 1	71 ± 3	0.75 ± 0.09	2.83 ± 0.18
	15	0.25 ± 0.03	12.68 ± 0.43	3.92 ± 0.11	0.492 ± 0.077	4.5 ± 0.2	16 ± 2	52 ± 3	0.46 ± 0.06	1.92 ± 0.14
0.2%	0	0.25 ± 0.03	11.00 ± 0.39	3.09 ± 0.10	0.104 ± 0.013	7.8 ± 0.5	43 ± 4	129 ± 4	2.47 ± 0.07	3.19 ± 0.19
	1	0.28 ± 0.00	10.58 ± 0.43	3.55 ± 0.24	0.115 ± 0.008	7.9 ± 0.3	44 ± 1	141 ± 10	2.68 ± 0.07	3.49 ± 0.28
	3	0.25 ± 0.03	10.78 ± 0.03	3.46 ± 0.15	0.087 ± 0.008	7.6 ± 0.2	44 ± 2	134 ± 11	2.68 ± 0.26	3.65 ± 0.28
	5	0.25 ± 0.03	12.88 ± 0.50	3.44 ± 0.17	0.093 ± 0.009	7.1 ± 0.8	27 ± 2	103 ± 9	2.34 ± 0.05	3.07 ± 0.22
	10	0.25 ± 0.03	12.46 ± 0.04	3.10 ± 0.18	0.534 ± 0.018	4.6 ± 0.2	18 ± 1	80 ± 3	0.62 ± 0.02	2.24 ± 0.36
	15	0.23 ± 0.00	12.46 ± 0.02	4.76 ± 0.35	0.555 ± 0.022	3.5 ± 0.9	18 ± 1	59 ± 2	0.40 ± 0.02	1.77 ± 0.12
0.4%	0	0.25 ± 0.03	10.97 ± 0.41	4.76 ± 0.25	0.146 ± 0.017	7.6 ± 0.2	42 ± 3	160 ± 14	2.56 ± 0.11	3.11 ± 0.08
	1	0.24 ± 0.00	10.15 ± 0.40	4.74 ± 0.28	0.152 ± 0.008	8.0 ± 0.8	45 ± 3	147 ± 12	2.62 ± 0.12	3.50 ± 0.38
	3	0.27 ± 0.03	10.37 ± 0.47	4.87 ± 0.37	0.107 ± 0.003	7.6 ± 0.2	47 ± 2	131 ± 3	2.46 ± 0.06	3.18 ± 0.13
	5	0.25 ± 0.03	12.23 ± 0.40	4.22 ± 0.10	0.162 ± 0.010	6.5 ± 0.2	39 ± 2	112 ± 11	2.16 ± 0.09	3.07 ± 0.16
	10	0.27 ± 0.03	9.76 ± 0.40	3.71 ± 0.12	0.490 ± 0.031	4.8 ± 0.1	13 ± 4	86 ± 4	0.48 ± 0.01	2.19 ± 0.15
	15	0.24 ± 0.03	12.05 ± 0.47	4.82 ± 0.25	0.632 ± 0.031	3.5 ± 0.1	11 ± 1	59 ± 3	0.41 ± 0.05	2.04 ± 0.19

TABLE 2 Two-way analysis of variance for physicochemical parameters (free acidity, FA in % oleic acid); peroxide value, PV in mEq O₂/kg; extinction coefficients at 232 and 268 nm, K₂₃₂ and K₂₆₈, respectively), oxidative stability (OS, h), radical scavenging activity (2,2-diphenyl-1-picrylhydrazyl [DPPH] inhibition rate, %), total phenolic content (TPC, mg GAE/kg), total carotenoids content (TCC, mg pheophytin equivalents/kg), and chlorophyll content (mg lutein equivalents/kg) of cv. Arbequina olive oils unflavored (0%) and flavored with lemon verbena essential oil (0.2% and 0.4%, w/w) and subjected to different microwave heating time-periods (0, 1, 3, 5, 10, and 15 min), at 800 W of power. Statistical significance (*p*-values) at a 5% level, for each main factor evaluated ("flavoring level" and "microwave heating") and type of effect (additive or nonadditive/disordinal effect)

Factor	Statistical significance (<i>p</i> -value) ^a								
	FA	PV	K ₂₃₂	K ₂₆₈	OS	DPPH	TPC	TCC	Chlorophyll
Flavoring level	0.9470	<0.0001	<0.0001	<0.0001	0.4310	0.4040	<0.0001	0.0012	0.0171
Microwave heating time	0.3700	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
Interaction effect (type of interaction)	0.3780	<0.0001 (nonadditive)	<0.0001 (nonadditive)	<0.0001 (nonadditive)	<0.0001 (nonadditive)	<0.0001 (nonadditive)	0.0002 (nonadditive)	<0.0001 (nonadditive)	<0.0001 (nonadditive)

^aValues in bold and italic mean a significant effect at 5% significance level.

has been previously reported in the literature for different unflavored olive oils (Kishimoto, 2019; Malheiro et al., 2009; Sevim et al., 2018).

Concerning the OS, DPPH, TPC, TCC, and chlorophyll content, from Table 1, it could be inferred that, for unheated samples (0 min), except for TPC and TCC, the flavoring level did not promote a clear increase of those parameters on flavored (0.2% and 0.4% EO) compared to the unflavored oils (0% EO). Flavoring resulted in a linear increase of the TPC (112–160 mg GAE/kg oil, *R*-Pearson = +0.9870) and of the TCC (2.19–2.56 mg lutein equivalents/kg oil, *R*-Pearson = +0.9611), but not so evident for chlorophyll content (2.32–3.19 mg pheophytin equivalents/kg oil, *R*-Pearson = +0.8238). Previously, Cherif et al. (2021) reported a decrease of the OS and an increase of both TPC and DPPH with the increase of the lemon verbena EO flavoring level in cv. Arbequina oils. In the literature, both increasing and decreasing trends have been reported, depending on several factors such as the type and level of flavoring agent, the type and quality of the olive oil, and the storage conditions (Baiano et al., 2009; Bobiano et al., 2019; Farhoosh & Hoseini-Yazdi, 2014; Khemakhem et al., 2015; Taoudiat et al., 2018; Tarchoune et al., 2019; Veloso et al., 2020). Concerning the microwave heating time-period, it had an obvious negative impact on the OS, DPPH, TPC, TCC, and chlorophyll content, leading to a marked decrease of their values, mainly for heating times between 5 and 15 min. Moreover, in general, the EO addition did not have a clear protective effect against the thermal degradation of the oils' quality, and similar levels were found in both unflavored and flavored oils after 10–15 min of microwave heating. The OS decrease for higher heating time-periods was expected since it would correspond to oils heated at higher temperatures, being known that the OS of olive oils decrease when the temperature rises, for both unflavored and flavored olive oils (Cherif et al., 2021; Farhoosh & Hoseini-Yazdi, 2014; Heidarpour & Farhoosh, 2018; Veloso et al., 2020). Also, Prata et al. (2018) reported a drastic reduction of the TPC of unflavored Portuguese commercial olive oils during microwave heating (1.5–10 min, corresponding to 72–205°C) compared to unheated olive oils (20°C). Similar findings were reported by Cerretani et al. (2009) and Kishimoto (2019). Oueslati et al. (2020) also verified the oxidative and hydrolytic degradation of the different phenolic subclasses due to microwave heating (2–15 min, corresponding to 139–268°C) of cv. Chétoui virgin olive oils, compared to that of unheated oils, as well as a slight decrease of the DPPH inhibition rate.

Lastly, the impact of the flavoring and microwave heating on the physicochemical and antioxidant characteristics of the studied cv. Arbequina oils is evidenced by their unsupervised PCA satisfactory differentiation (Figure 1). The PCA biplot clearly pointed

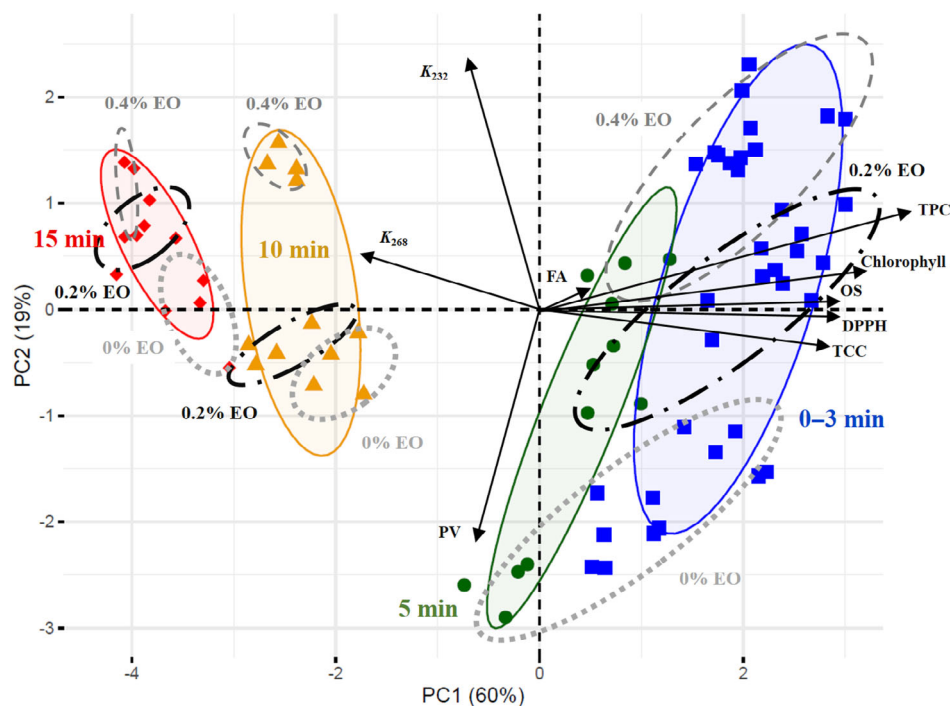


FIGURE 1 Principal component analysis biplot based on the cv. Arbequina olive oils' quality data, composition, and antioxidant activity (free acidity, % oleic acid; peroxide value, mEq O₂/kg; K_{232} ; K_{268} ; oxidative stability, h; 2,2-diphenyl-1-picrylhydrazyl, %; total phenols content, mg GAE/kg; total carotenoids content, mg pheophytin equivalents/kg; and, chlorophyll content, mg lutein equivalents/kg): Unsupervised classification according to the flavoring level with lemon verbena essential oils (EO) (.....: 0% EO; —: 0.2% EO; and, - - - -: 0.4% EO) and microwave heating time (■: 0–3 min; ●: 5 min; ▲: 10 min; and, ●: 15 min)

out that the microwave heating time had a greater impact on the changes of the parameters evaluated compared to the oils' flavoring, allowing a clear unsupervised grouping of the oil samples according to the time-periods (first principal component function, i.e., x-axis, with the higher heating time-periods on the negative region and the lower heating time-periods on the positive region). Specifically, oils heated for 0–3 min and 5 min could be clearly split from those heated during 10 or 15 min, for which the quality degradation was more drastic (higher PV, K_{232} , and K_{268} and quite lower TPC, OS, DPPH, TCC, and chlorophyll content). On the other hand, the flavoring level grouping split is mainly linked to the second principal component function (y-axis) being the lower flavoring level (0% EO) located in the negative part of the axis (higher PV values and lower extinction coefficients) and the higher level (0.4%) placed on the positive region (higher extinction coefficients and lower PV), independently of the heating time-period.

Kinetic modeling of TPC, TCC, and chlorophyll thermal degradation on unflavored and flavored cv. Arbequina oils during microwave heating

Chemical degradation reactions occurring in foods during processing or storage can be modeled using kinetic models of different reaction orders. Zero-, first-, and second-order models have been applied to describe the thermal degradation of bioactive compounds (e.g., phytosterols, phenolic compounds, carotenoids,

vitamin C, chlorophyll, vitamin C) in different food matrices (e.g., apple peel by-products, beans, Brussels sprouts, fruit juices, betel extracts, plum extracts, dried flowers, and floral bio-residues of saffron, edible and nonedible vegetable oils, sea buckthorn) during storage or processing (e.g., different types of cooking processes) (Ali et al., 2018; Aparicio-Ruiz et al., 2011; Henríquez et al., 2014; Igoumenidis et al., 2011; Mba et al., 2019; Moratalla-López et al., 2019; Nakilcioglu-Taş & Otleş, 2017; Sapei & Hwa, 2014; Turturică et al., 2016; Ursache et al., 2017; Van Bree et al., 2012). A brief survey clearly pointed out that the first-order kinetic model is the most used one to describe the thermal impact at the bioactive composition level of the food matrices. In which concerns the olive oil, the number of studies is limited. Igoumenidis et al. (2011) studied the degradation of β -sitosterol and total phytosterols in virgin olive oils during repeated deep and pan-frying of prefried potatoes, reporting that the first-order model was superior compared to the zero-order and second-order models. Also, Aparicio-Ruiz et al. (2011) proposed a first-order kinetic mechanism to model the thermal degradation of lutein, β -carotene, and β -cryptoxanthin in virgin olive oils subjected to different heat treatments (from 42 h/120°C to 744 h/60°C).

In this context, the thermal degradation of TPC, TCC, and chlorophyll occurred in each type of olive oil studied (unflavored and flavored oils) due to the microwave heating process at a constant microwave power of 800 W (from 1 to 15 min, which would roughly correspond to a temperature range varying from approximately 41–235°C) was further evaluated from a kinetic

TABLE 3 Rate constants ($k \pm$ standard error [SE]), correlation coefficients (R) and half-life values ($t_{1/2}$) estimated assuming a second-order kinetic mechanism for the thermal degradation due to microwave heating (0–15 min) at 800 W, for total phenols (TPC), total carotenoids (TCC), and chlorophyll contents, of cv. Arbequina olive oils without (0% essential oil [EO]) or with the addition (0.2% and 0.4%) of lemon verbena EO

EO added to olive oil (w/w)	TPC		
	Second-order rate reaction constant ($k \pm$ SE; kg min ⁻¹ mg ⁻¹ GAE) ^a	Correlation coefficient (R)	Half-life ($t_{1/2}$; min) ^b
0% EO	$(70 \pm 9) \times 10^{-5}$	0.9760	12.2
0.2% EO	$(72 \pm 5) \times 10^{-5}$	0.9920	10.8
0.4% EO	$(71 \pm 7) \times 10^{-5}$	0.9848	8.8
EO added to olive oil (w/w)	TCC		
	Second-order rate reaction constant ($k \pm$ SE; kg min ⁻¹ mg ⁻¹ lutein eq) ^a	Correlation coefficient (R)	Half-life ($t_{1/2}$; min) ^b
0% EO	0.14 ± 0.02	0.9775	3.4
0.2% EO	0.16 ± 0.02	0.9731	2.5
0.4% EO	0.17 ± 0.03	0.9541	2.3
EO added to olive oil (w/w)	Chlorophyll content		
	Second-order rate reaction constant ($k \pm$ SE; kg min ⁻¹ mg ⁻¹ pheophytin eq) ^a	Correlation coefficient (R)	Half-life ($t_{1/2}$; min) ^b
0% EO	0.014 ± 0.004	0.8784	30.8
0.2% EO	0.022 ± 0.002	0.9845	14.5
0.4% EO	0.016 ± 0.002	0.9769	20.2

^aRate reaction constants determined using Equation (3), assuming a second-order kinetic model to describe the thermal degradation of each bioactive compound (content decrease) in the studied olive oils, when subjected to increasing microwave heating time-periods (0–15 min).

^bHalf-life values calculated using Equation (10), assuming a second-order kinetic model to describe the thermal degradation of each bioactive compound (content decrease) in the studied olive oils, when subjected to increasing microwave heating time-periods (0–15 min).

point of view. Zero-order, first-order, and second-order kinetic models were considered. The results allowed verifying that, globally, the second-order model was the one that better fitted the thermal degradation process of the referred three parameters for all oils studied ($0.8784 \leq R\text{-Pearson} \leq 0.9926$). Table 3 shows the second-order rate constants (\pm SE), calculated as the slope of the linear regression of the inverse of the concentration versus the heating time (according to Equation (3), obtained after the linearization of kinetic model), the correlation coefficient, and the estimated half-life (determined using Equation (10)), for each compound and type of olive oil studied. The estimated second-order reaction rates for TPC (0.00070–0.00072 kg oil min⁻¹ mg⁻¹ GAE) and for TCC (0.14–0.17 kg oil min⁻¹ mg⁻¹ lutein equivalents), regarding the thermal degradation of the cv. Arbequina oils during microwave heating, were not influenced by the EO incorporation. In contrast, the level of EO added seemed to influence the degradation rate of chlorophyll (0.014–0.022 kg oil min⁻¹ mg⁻¹ pheophytin equivalents), although no direct trend could be established with the flavoring percentage. On the other hand, and as a possible shelf-life inference, it was observed that, for unflavored and flavored oils, the TCC suffered a faster degradation (half-life of 3.4 to 2.3 min, respectively), compared to TPC (half-life from 12.8 to 8.8 min, respectively) and chlorophyll (half-life between 30.8 and 14.5 min, respectively), being this latter the most thermal resistant. Besides,

with the exception of chlorophyll, the half-life of TPC and TCC linearly decreased with the rise of added EO level ($R\text{-Pearson}$ equal to -0.9999 and -0.9421 , respectively). This finding is of utmost relevance since, although lemon verbena EO was added to the cv. Arbequina olive oil aiming to enhance its resistance toward quality thermal degradation, the oils' flavoring did not have a protective effect against the degradation of these bioactive compounds when subjected to a microwave heating. In fact, on the contrary, this EO promoted the degradation of these bioactive compounds.

CONCLUSIONS

The study allowed concluding that flavoring cv. Arbequina olive oils with lemon verbena EO had a positive impact on the levels of minor bioactive compounds, increasing the total phenols and carotenoids contents, although this did not have a direct and clear effect on the oils' OS on the radical scavenging activity. On the other hand, the quality of the unflavored and flavored oils was clearly degraded after being subjected to microwave heating time-periods that mimic usual domestic cooking procedures and at a usual power of 800 W. Furthermore, based on the half-life determined based on the second-order rate reaction constants, chlorophyll was the minor constituent most thermally resistant followed by phenols and carotenoids. Indeed,

the kinetic evaluation pointed out that the olive oils' thermal degradation was not delayed by the EO addition, seeming that the flavoring procedure promoted a faster decrease of total contents of the minor bioactive constituents. In conclusion, from this study emerges the need to perform a preliminary thermal-kinetic evaluation when any olive oil flavoring process is envisaged, namely under typical domestic cooking application, to ensure the benefit of the flavoring practice. Finally, it should be remarked that, since olive oil was studied alone, the interactions between the oil and other food ingredients during the microwave heating of a specific meal were not taken into account, which must be addressed in a future work. Indeed, the type and amounts of bioactive compounds present in the other food ingredients may play a key role on the extent and speed of the thermal degradation of the olive oil.

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AUTHOR CONTRIBUTION

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CONFLICT OF INTEREST


The authors declare that they have no conflict of interest.

ETHICS STATEMENT

This article does not contain any studies with human participants or animals performed by any of the authors.

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