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Cross-supply-chain by-product valorization: optimization of Sorrento lemon peel aromatization of refined olive and grape seed oils via Ultrasound-Assisted Extraction

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ABSTRACT

The growing challenge of food waste highlights the need for strategies that reintroduce discarded materials into the food chain with added value. Sorrento lemon peels and grape seed oil are by-products of the citrus and wine industries that are often underutilized despite their potential, while the refined olive oils are obtained from low-quality virgin oils. In this study, these three materials were jointly valorized through ultrasound-assisted extraction to produce lemon-flavored oils with improved functional and sensory properties. A Design of Experiments approach was applied, and experimental data were fitted to second-order polynomial models to optimize the aromatization process based on peroxide value. Oil quality was further evaluated through free acidity, UV spectrophotometric indices, total phenolic content (TPC), antioxidant activity (DPPH), and volatile profiling (HS-SPME-GC–MS). Optimal conditions were identified as 3 min, 0.15 g/g matrix-to-oil ratio, 15 °C for olive oil, and 16.5 min, 0.25 g/g, 15 °C for grape seed oil. Under these conditions, the flavored olive oil showed the highest citral percentage and a threefold increase in TPC (from 54.24 ± 8.04 to 179.06±6.87 mg GAE/kg), while the flavored grape seed oil exhibited a fourfold increase compared to controls (from 42.41±8.29 to 168.22 ±1.10 mg GAE/kg) . Moreover, off-flavors present in the base oils were not detected. This approach provides a sustainable and scalable method to convert multiple low-value by-products into higher-value flavored oils.

1. Introduction

Nowadays, food waste managing, valorize and reuse is an essential topic, considering that in 2022 around 132 kg/year/capita were generated in the European Union and that amount is continuously increasing, concerning fruit and vegetables for around 50 % of the total (Eurostat, 2024).

The percentage of the discarded matrices even increases for PGI (Protected Geography Indication) or PDO (Protected Designation of Origin) products. For instance, PGI Sorrento lemons, cultivated using traditional systems and a precise planting density in the Italian Sorrento Peninsula and Capri, must undergo to high aspect or weight standards (>85 g each), leading to a high amount of under-sized units explicitly exploited in transformations. (Commission Regulation (EU) No 14/2011, 2011).

Another example comes from the wine production, from which about 3 million tons of grape seeds are discarded every year in Europe. On this line, grape seed oil (GSO) is a valid and sustainable way to reuse this matrix. The production process involves drying, grinding and subsequent solvent extraction or mechanical pressing steps (Dimić et al., 2020). However, since crude GSO often has high content of free fatty acids, impurities, and off-flavors, a refining step is necessary before consumption. This process is a sequence of steps including degumming with water or acids (e.g., phosphoric acid); neutralization with alkalis (e.g., sodium hydroxide) to eliminate free fatty acids; bleaching through adsorbents (e.g., activated clay, charcoal) to strip pigments and trace impurities; and deodorization using high heat (180–270 °C) and steam under vacuum to remove volatile compounds causing

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off-flavors—resulting in safe, stable, and palatable cooking oils. Unfortunately, the refining process cannot overlook a significant molecular impact including the reduction of bioactive compounds such as polyphenols, phytosterols or tocopherols, naturally high-concentrated in GSO.

Similarly, low-quality olive oil with sensory defects or high acidity (i.e., lampante olive oil) can be reintroduced to the market as refined product (OO) (Regulation (EU) 2022/2104). On this line, refining practice represents a useful form of recovery, as it transforms a product that would otherwise be discarded into an edible oil suitable for consumption. Especially during particularly unfavorable periods, attacks by the olive flies or plant disease outbreaks threaten olive oil quality—specifically increasing free acidity, impairing sensory characteristics (e.g., aroma, flavor), and elevating levels of oxidative compounds—such that in some cases, approximately 41 % of the collected oils cannot even be classified as extra virgin olive oil (EVOO) (Abacıgil et al., 2023; Cecchi et al., 2016).

Obviously, GSO and OO lack the pungency and flavor of EVOO, which is commonly associated with beneficial qualities. On this line, aromatization with lemon or orange resulted a good strategy to improve the aromatic and nutritional profile and the oxidative stability in OO (Ayu et al., 2024) and in GSO, since they are often less consumed, due to their low bioactive content, which significantly decrease during the refining process. Several studies reported that the interest toward GSO is growing (Cavallo et al., 2019; Laqui-Estaña et al., 2024; Menghini et al., 2022). The flavoring of these edible oils is mainly due to essential oil contained in the flavedo of fruit peel (flavedo), since the citrus essential oil composition is influenced by numerous factors, ranging from the harvest time, the cultivar, the isolation methods (Bozova et al., 2025; Gölükcü et al., 2024) and environmental conditions (Luro et al., 2020). Moreover, from a technological and industrial perspective, refined oils represents a suitable and controlled lipid matrix for investigating the sole contribution of added plant materials and processing conditions to flavour development, without interference from intrinsic olive oil aromas.

According to the green economy principles, this paper aims in the valorization of low-value oils (GSO and OO) using a food waste, as the lemon peel discarded from transformation of not-PGI-suitable Sorrento lemons, representing a by-products supply chain-crossing. For this purpose, the possibility to use a green Ultrasound Assisted Extraction (UAE) method instead of the time-consuming infusions or maceration processes in oil aromatization, was explored. A detailed experimental design was prepared and followed for the optimization of all the process parameters, including power, frequency, matrix/solvent ratio, temperature and exposure time. Consequently, all the obtained products were characterized in terms of stability through acidity and peroxides analysis, bioactive compounds content and aromatic profile using HS-SPME-GC–MS, and resulting data were used to choose the best parameters for UAE oil aromatization. To the best of authors knowledge, this represents the first optimization UAE approach to GSO and OO lemon peels aromatization, including quality parameters, beyond their enrichment of polyphenols or antioxidant activity. Thus, this could be a good starting point to increase scientific interest in the enhancement of food waste products through supply chain crossing and the use of green and sustainable techniques.

2. Materials and methods

2.1. Reagents and standards

The analytical standards of gallic acid (≥97.5 %, $C_7H_6O_5$), 2,2-diphenyl-1-picrylhydrazil (DPPH, $C_{18}H_{12}N_5O_6$), 6-hydroxy-2,5,7,8 tetramethylchromane-2-carboxylic acid (≥ 97 %, Trolox, $C_{14}H_{18}O_4$), rutin (≥ 94 %, $C_{27}H_{30}O_{16}$), chloroform (≥ 99.8 %, $CHCl_3$), potassium iodide (≥99.0 %, KI), sodium thiosulfate (≥99.0 %, $Na_2S_2O_3$), phenolphthalein (ACS reagent, $C_{20}H_{14}O_4$) and Folin-Ciocalteu phenol reagent were supplied by Sigma–Aldrich (Milan, Italy). Sodium carbonate anhydrous (≥99 %, Na_2CO_3), sodium nitrite (≥99,0 %, $NaNO_2$), aluminum chloride (≥99 %, $AlCl_3$), sodium hydroxide (≥98 %, NaOH), acetic acid (≥99 %, CH_3CO_2H), potassium hydroxide (90 %, KOH) and sodium chloride (≥99 %, NaCl) were purchased from Merck (Milan, Italy). Methanol, hexane, ethanol and ethyl ether were purchased from Carlo Erba Reagents Srl (Cornaredo, Milan, Italy).

2.2. Samples collection

The vegetable oils, GSO and OO were purchased from a local supermarket in Camerino (MC, Italy) in May 2025 and stored at room temperature. Both of them were produced in 2025, refined and obtained by solvent extraction, according to the label, and were sold in dark glass bottles for OO or GSO, respectively, protected from light, with a total volume of 1 L. For the analyses 3 bottles of GSO or OO have been sampled each time.

The peels (flavedos) from the cultivar of PGI Sorrento Oval Lemon (*Citrus limon* L.), were provided by New Flavours® company (Via dell'Artigianato 7, Monte Santa Tiberina, PG, Italy) in February 2025. They were cultivated according to the PGI production regulations and harvested in February 2025. They were dried in an ventilated dryer at 40 °C until constant weight and ground using a laboratory bench homogenizer prior to extraction. All samples were processed under identical grinding conditions to ensure experimental consistency and the resulting material was used immediately for extraction.

2.3. Ultrasound-Assisted extraction (UAE) optimization by experimental design

OO and GSO were flavored with lemon peels through UAE using an ultrasonic bath (Model Lbs 2, FALC, 10-liter capacity) operating at 40 kHz and a nominal power of 550 W. The reported power refers to the total power consumption of the ultrasonic bath, while the effective acoustic power delivered to the individual sample was not directly measured (Licht et al. (2021). All experiments (mentioned as Exp followed by a sequential number) were conducted under identical operating conditions (bath model, nominal power, temperature, extraction time, sample mass and geometry) to ensure experimental reproducibility. To optimize the extraction conditions, namely temperature (from 15 to 35 °C), extraction time (from 3 to 30 min) and matrix/solvent ratio (from 0.05 to 0.25), the Design of Experiment (DoE) tool of XLSTAT software (version 2023.1.4.1408) was used in Box-Behnken design mode. During the entire UAE, the extraction temperature was continuously monitored and ensured to be constant in the ultrasonic bath: any enhancement beyond the set temperature due to ultrasound irradiation was immediately counteracted by ice-addition to the bath (temperature deviations never overreached ±1 °C). Two different DoEs were prepared, one for GSO and one for OO samples. The monitored variable response was the peroxide value (PV) of the final flavored oil as described in *section 2.4*: it was selected as the only response variable, towards the first objective of an investigation and optimization of the primary oxidation phenomenon during the UAE. The extraction parameters ranges were chosen according to previous works about the optimization of ultrasonic extraction for oil and the reduction of degradative processes for many compounds, due to prolonged exposure to ultrasounds or higher temperature (Da Rosa et al., 2019; Heck et al., 2018; Soares et al., 2020). After preliminary tests, matrix/solvent ratios higher than 0.25 were excluded, since they resulted in excessive oil absorption by the plant material. A total of 26 experiments were recommended from DoE for each kind of oil. In each DoE experiment 50 g of oil were used, varying the amount of lemon peels, according to the suggested matrix/solvent ratio.

2.4. Oil quality parameters: peroxide value, free acidity and specific coefficients analyses

The determination of peroxide value (PV), free acidity (expressed as % of oleic acid), and specific coefficients (K232, K270, and ΔK) were conducted according to the protocols of EC Reg. no. 2568/1991 and the subsequent amendments reported by the European Commission (Official Journal of the European Communities, 2016). Free fatty acid content and extinction coefficients were monitored as a quality indicator and was not included as a response variable in the modelling approach, as it is mainly influenced by the initial oil quality and was not expected to be directly affected by the UAE operating conditions investigated. They were performed in triplicate for all the above-cited samples from the 52 experiments of the 2 DoEs. Thus, the analysis of free acidity (FFA) was conducted through titration with a 0.05 mol/L KOH ethanolic solution. A 1 % ethanolic solution of phenolphthalein was used as indicator and 5 g of oil was dissolved in an ether/ethanol solution (1/1), which had been neutralized with KOH immediately before use. The measurements were performed through iodometric titration with sodium thiosulfate, in acetic acid and chloroform and using starch solution as an indicator. The number of peroxides (the primary products of oxidation), was expressed in milliequivalents of active oxygen per kg of oil (meq O_2/kg oil), while the specific extinction coefficients were evaluated spectrophotometrically in triplicate, according to the aforementioned Regulation.

2.5. Total phenolic content and antioxidant activity of flavored oils

For total phenolic content (TPC) analysis, a procedure previously reported by Ricciutelli et al. (2017) was involved. Briefly, 2.5 g of sample was diluted using the same volume of hexane, then extracted with 2.5 mL of MeOH/H_2O 80:20 *v/v* mixture and centrifuged. A total of 2.5 mL of Folin-Ciocalteu reagent (FC) and 2.5 mL of 7.5 % Na_2CO_3 *w/v* aqueous solution were added to the supernatant, and the solution is brought to 50 mL with distilled water. After 4 h in the dark, the absorbance was measured at 765 nm with Cary 8454 UV–visible spectrophotometer (Agilent Technologies, Woburn, MA, USA). The calibration curve was prepared with gallic acid and results are expressed as mg of gallic acid equivalents (GAE)/kg of oil. The procedure was repeated in triplicate for each of the 52 experiments of the 2 DoEs.

Antioxidant activity was measured through the DPPH assay, following the same procedure reported by Alessandroni et al., 2024 and Corsetti et al., 2025. Firstly, 0.8 mL of sample was mixed with 2.4 mL of methanol and centrifuged, then 4.5 mL of DPPH (2,2-diphenyl-1-picrylhydrazyl radical) 0.1 mM ethanolic solution was added to 0.5 mL of supernatant. After 30 min in the dark at room temperature, the absorbance was read at 517 nm. Trolox was used as the reference antioxidant compound for the calibration curve and the results were reported as the μmol concentration of Trolox equivalents/g oil. The procedure was repeated in triplicate for each of the 52 experiments of the 2 DoEs, for OO and GSO.

2.6. Analysis of volatile organic compounds using HS-SPME-GC-MS

The analysis of the volatile organic compounds (VOCs) was conducted in triplicate for each sample, according to the method reported by Fiorini et al., 2018, with slight modifications. Briefly, 1 g of each oil was placed in a vial. A solid-phase microextraction (SPME) fiber with a 50/30 μm coating of divinylbenzene/carbon-wide range/polydimethylsiloxane (DVB/C-WR/PDMS, 1 cm length stationary phase) from Supelco (Bellefonte, PA, USA) was involved. It was conditioned at 40 °C for 10 min. then exposed to the headspace of the sample for 30 min. The injector temperature was 260 °C.

A gas chromatography instrument coupled with mass spectrometer (GC–MS) was equipped with a PAL3-Auto Sampler System (Agilent 7890B GC Hardware with an Agilent 5977 Series MSD, Agilent, Santa Clara, CA, USA) and was used to acquire data that were analysed by MassHunter GC/MSD Data Acquisition software. The capillary column was a 60 m DB-WAX with an internal diameter of 250 μm and a film thickness 0.25 μm. The He flow rate was 1 mL/min. A spitless injection was applied. The initial oven temperature was 40 °C held for 4 min, then the temperature increased to 120 °C at 2.5 °C/min and then raised again to 250 °C at 15 °C/min, holding it for 3.33 min. The ionization source was an electron impact ionization (EI) and data were collected in SCAN mode; the transfer line was set at 250 °C and the temperature of the ionization source and the mass analyser were set at 230 and 150 °C, respectively. Compounds identification was both performed by spectra comparison with NIST library (US National Institute of Standards and Technology) and by retention indices (RI) calculation and comparison with literature values. The results are expressed as relative abundances of each volatile compound (% peak area of the compound/ total area of the peaks). The amount of volatile compounds retained in the oils was not modelled as a DoE response variable, since VOCs analysis was performed in scan mode without internal standards or calibration curves, allowing only a qualitative comparison among samples.

2.7. Statistical analysis

XLSTAT software (version 2023.1.4.1408) was involved in the 3-variables Design of Experiment in Box-Behnken designs mode preparation and analysis with the PV as dependent variable. The experimental data were analysed by analysis of variance (ANOVA) to evaluate the significance and adequacy of the fitted models. Model significance was assessed using F-values and corresponding p-values, while lack-of-fit tests were performed to verify the goodness of fit. Regression coefficients (β_n) were estimated for all model terms in order to describe the contribution of each factor to the response variables. Statistical analyses were performed at a significance level of $p < 0.05$. All data are reported as triplicate average values and standard deviations.

3. Results and discussion

3.1. Optimization of UAE parameters via response surface modeling

The UAE parameters optimization, including time, temperature and matrix/solvent ratio, was carried out according to the Box-Behnken DoEs. The Box–Behnken design was chosen as it allows the evaluation of potential interactions between parameters with fewer experimental runs, while still providing reliable estimates of first- and second-order coefficients. Unlike central composite designs, which may include extreme combinations and require up to five levels per factor, the Box–Behnken approach relies on only three levels per factor, making it more efficient and time-saving (Alessandroni et al., 2024).

The experimental data were fitted to two different second-order polynomial regression model, whose adequacy was confirmed by ANOVA (OO data results: $F = 49.68$, $p < 0.0001$, $R^2 = 0.97$, adjusted $R^2 = 0.95$; GSO data results: $F = 27.13$, $p < 0.0001$, $R^2 = 0.94$, adjusted $R^2 = 0.90$). The lack-of-fit tests were not significant, demonstrating an adequate agreement between experimental and predicted values. These results indicate a good agreement between experimental and predicted values, confirming the reliability of the model.

The final regression equation describing the relationship between PV and the coded process variables is expressed in Eq. 1 and 2 for OO and GSO respectively.

$$Y = 5.85 + 0.33t + 0.27R + 1.46T - 0.23t^2 + 0.44Rt + 0.66Tt - 0.40R^2 + 0.36RT + 0.05T^2 \tag{1}$$

$$Y = 6.3 + 0.07t - 0.06R + 0.57T + 0.08\ T^2 + 0.03Rt + 0.04\ Tt + 0.14R^2 + 0.09\ RT - 0.84T^2 \tag{2}$$

where Y is the PV value, and T, R, and t are the coded variables for temperature, matrix/solvent ratio, and time, respectively. Regression coefficients (βₙ) highlighted the relative contribution of the individual terms to the model responses, supporting the robustness and reliability of the proposed models.

In both matrices, OO and GSO, the peroxide value (PV) of the final product was monitored as response. The PV monitoring resulted of particular interest as, according to previously published evidences, UAE may cause the degradation of some compounds, especially in high-lipid matrices, like vegetable oils (Pingret et al., 2013; Ayu et al., 2024). For instance, it was reported that the PV increased from 5.38 meq O_2/kg oil for untreated oil to 6.33 meq O_2/kg oil for sonicated sunflower oil (Chemat et al., 2004). A similar behavior was observed in other studies, together with a slightly increase in the percentage of conjugated dienes, especially in presence of oxygen during ultrasonic treatment (Sicaire et al., 2016; Soares et al., 2020). In addition, some conditions can lead to the formation of compound conferring unpleasant taste, affecting organoleptic characteristics of the final product (Soares et al., 2020).

Results of OO and GSO aromatization optimization are reported in surface plots in Fig. 1 and 2, respectively. In OO aromatization, temperature emerged to have higher impact in peroxide formation than matrix/solvent ratio (Fig. 1A). In fact, at lower temperatures, regardless of the matrix/solvent ratio, the PV is low, while it increases working at higher temperatures reaching the highest value (8.25±0.25 meq O_2/kg) at 35 °C. A similar tendency was confirmed also for other vegetable matrices (Jing et al., 2015; Vo et al., 2023). Temperature is the most influencing parameter also in comparison with extraction time (Fig. 1B): in this case, prolonged extraction at low temperature seems to provide the lower PV (3.95±0.05 meq O_2/kg). As widely reported, in UAE, increasing both temperature and time, the peroxides formation in vegetable oils is favored (Pingret et al., 2013). Matrix/solvent ratios emerged to be influencing in long lasting aromatization with proportionally increasing PV (Fig. 1C). A similar tendence was also noticed by Kishimoto et al. (2022) in the aromatization of EVOOs with lemon flavedos through maceration. In fact, higher amount of solid (grinded dry lemon peels) may be related to a lower specific heat capacity, compared to a lower solid/liquid ratio experiment, in which probably a faster heating with an enhanced oxidation occurs (Çengel & Yunus, 2011).

To the best of the authors' knowledge, while the aromatization of various seed oils has been optimized in previous studies, this is the first work specifically focusing on grape seed oil (GSO) (Drosaki et al., 2025; Goula et al., 2017; Piasecka et al., 2023). The GSO flavoring optimization surface plots are reported in Fig. 2. As shown in Fig. 2A, at higher temperatures (23–35 °C), extreme matrix/solvent ratios (0.05–0.12 and 0.22–0.25) were associated with increased PV. In Fig. 2B, higher (18–30 min) extraction times led to higher PV, particularly in the 24–33 °C range. In line with the previous findings, Fig. 2C highlights that the highest PV occurred when both extraction time and matrix/solvent ratio were set at their lower or upper extremes. In contrast, intermediate levels of these parameters appeared to provide an optimal amount of lemon peel for antioxidant extraction, thereby better protecting the oil against oxidative damage (Blasi et al., 2020; Weremfo et al., 2023). It is also important to note that GSO exhibited higher PV than olive oil even before UAE flavoring, due to its fatty acid profile: with 85–90 % unsaturated fatty acids, GSO is inherently more susceptible to oxidation, even under minimal or short ultrasonic treatment (Laqui-Estaña et al., 2024; Lutterodt et al., 2011). Therefore, intermediate extraction times (16.5 min) and average or high matrix/solvent ratios (0.20–0.25) and 15 °C processing appear more suitable to mitigate oxidative stress in GSO aromatization.3.2. Impact of UAE conditions on oxidative stability and legal quality indices

DoE suggested experiments are reported in Tables 1 and 2 for OO and GSO respectively. For each flavored oil, PV was evaluated as response of the design model and free fatty acids (expressed by oleic acid %) as further information about oxidation and potential oxidation capacity of each product.

In olive oil, peroxide values (PV) and free fatty acids (FFA) varied significantly across treatments, as indicated by the ANOVA results (Table 1).

The control sample showed the lowest PV (3.25 ± 0.35 meqO_2/kg), while Exp5 (30 min, 0.15 g/g, 35 °C) recorded the highest PV (8.25 ± 0.35 meqO_2/kg), significantly higher than all other trials. Intermediate conditions, such as Exp3, Exp7, Exp11 or Exp1, displayed statistically lower PV (3.95–4.35 meqO_2/kg), closer to the control. Regarding FFA, Exp9 (0.44 ± 0.06 %) was the highest, whereas the control and treatments such as Exp1 and Exp11 (0.11–0.17 %) showed the lowest values. Importantly, all FFA levels remained well below the regulatory threshold of 1 % established by Delegated Regulation (EU) 2016/2095 and confirmed by Regulation (EU) 2022/2104, indicating that UAE did not compromise compliance with quality standards.

In grape seed oil, the key finding is that there were no statistical differences found between the PV values of the unflavored control and the lemon-flavored samples, except for 3 of the latter with statistically higher levels (Exp6, Exp10, Exp13). Indeed, PV values varied from 4.85 ± 0.21 (Exp11) to 6.65 ± 0.21 meq O_2/kg (Exp6) among all treatments, with the control (5.63 ± 1.10 meq O_2/kg) fitting quite comfortably within this range, even if significant differences emerged between different experiments (Table 2).

For FFA, the lowest and statistically similar values were recorded in Control samples (0.29 ± 0.01 %) and other seven flavored samples, while the maximum value in Exp2 (0.66 ± 0.05 %), significatively

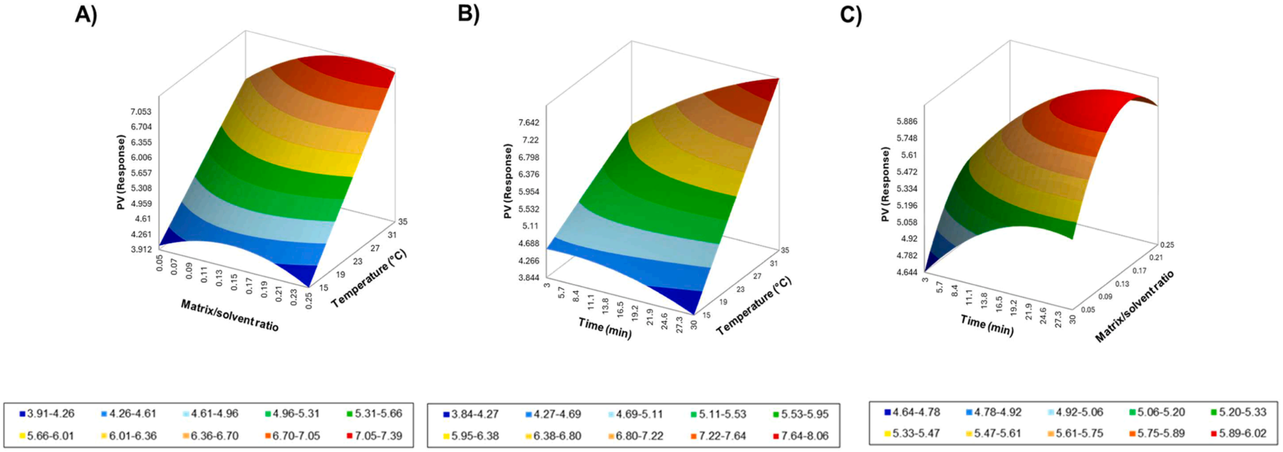

Fig. 1. Olive oil (OO) lemon peel UAE aromatization optimization surface plots based on PV responses. A) matrix/solvent ratio - temperature, B) time – temperature, C) time – matrix/solvent ratio.

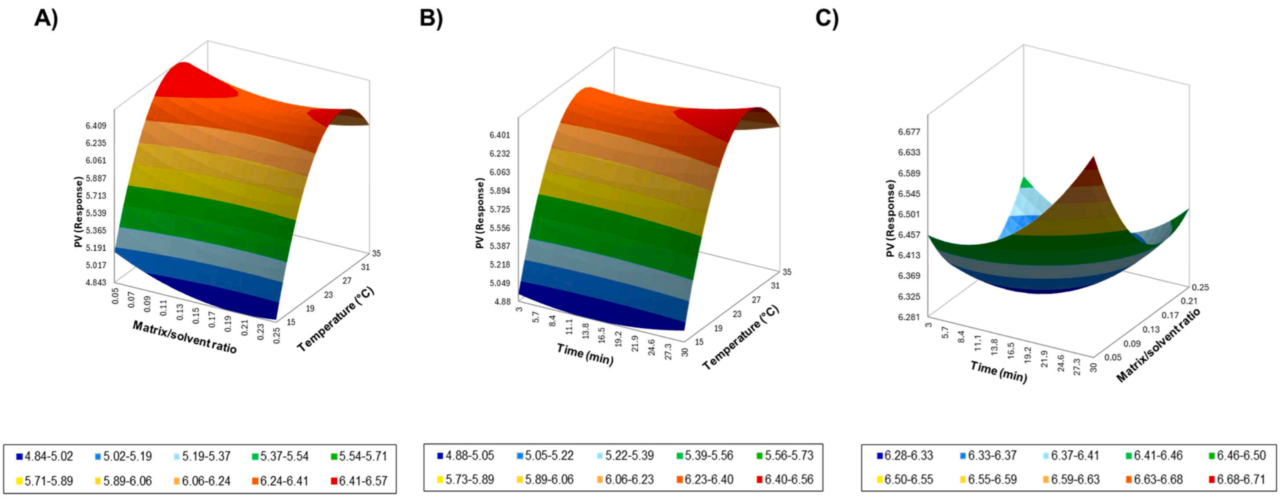

Fig. 2. Grape seeds oil (GSO) lemon peel UAE aromatization optimization surface plots based on PV responses. A) matrix/solvent ratio – temperature, B) time – temperature, C) time – matrix/solvent ratio.

Table 1
PV and FFA evaluated for each experiment of the OO Box-Benkhen design for the UAE of lemon peels in olive oil. The different letters indicate statistical differences evaluated through one-way ANOVA among each experiment (Exp), including the control sample (unflavored olive oil).

Experiments	Observations	Time (min)	Matrix/solvent ratio	Temperature (°C)	PV (meqO_2/kg)	FFA (% oleic acid)
Control (unflavored OO)	/	/	/	/	3.25 ± 0.35 f	0.13 ± 0.02 gh
Exp1	Obs1	3.00	0.15	15.00	4.35 ± 0.35 de	0.11 ± 0.02 h
	Obs2	3.00	0.15	15.00		
Exp2	Obs3	16.50	0.05	35.00	6.20 ± 0.28 b	0.17 ± 0.01 fg
	Obs4	16.50	0.05	35.00		
Exp3	Obs5	16.50	0.05	15.00	4.00 ± 0.00 e	0.17 ± 0.02 fg
	Obs6	16.50	0.05	15.00		
Exp4	Obs7	16.50	0.15	25.00	5.85 ± 0.21 b	0.26 ± 0.02 de
	Obs8	16.50	0.15	25.00		
Exp5	Obs9	30.00	0.15	35.00	8.25 ± 0.35 a	0.26 ± 0.02 de
	Obs10	30.00	0.15	35.00		
Exp6	Obs11	30.00	0.05	25.00	4.70 ± 0.28 d	0.14 ± 0.03 gh
	Obs12	30.00	0.05	25.00		
Exp7	Obs13	30.00	0.15	15.00	3.95 ± 0.07 e	0.24 ± 0.03 e
	Obs14	30.00	0.15	15.00		
Exp8	Obs15	3.00	0.15	35.00	5.90 ± 0.14 b	0.21 ± 0.02 ef
	Obs16	3.00	0.15	35.00		
Exp9	Obs17	16.50	0.25	35.00	6.00 ± 0.00 b	0.44 ± 0.06 a
	Obs18	16.50	0.25	35.00		
Exp10	Obs19	30.00	0.25	25.00	6.00 ± 0.28 b	0.33 ± 0.02 bc
	Obs20	30.00	0.25	25.00		
Exp11	Obs21	16.50	0.25	15.00	4.00 ± 0.00 e	0.13 ± 0.01 gh
	Obs22	16.50	0.25	15.00		
Exp12	Obs23	3.00	0.25	25.00	5.25 ± 0.28 c	0.30 ± 0.04 cd
	Obs24	3.00	0.25	25.00		
Exp13	Obs25	3.00	0.05	25.00	5.25 ± 0.35 c	0.36 ± 0.02 b
	Obs26	3.00	0.05	25.00		

different from all other treatments. As with olive oil, all FFA levels were consistently below the 1 % legal limit, confirming that, despite variability among treatments, the oils preserved their compliance with EU quality requirements. Overall, these findings highlight the strong influence of UAE parameters on oxidative stability without exceeding regulatory thresholds. Thus, Exp11 should be the best choice in terms of lowest PV and statistically similar FFA to the Control.

Comparing our results with previous studies, Khemakhem et al. (2015) investigated the aromatization of virgin olive oils (VOO) using Citrus limetta and Citrus sinensis peels through maceration at 60 °C for 40 days. In our study, all flavored oils exhibited lower free fatty acid (FFA) values, which is noteworthy given that the control olive oil in our experiments already had a relatively low initial FFA. Similarly, both Khemakhem et al. (2015) and Kishimoto et al. (2022) observed minimal statistical differences in peroxide value (PV) and FFA between unflavored VOOs or extra virgin olive oils (EVOOs) and those flavored with lemon peels via maceration.

In contrast, Sacchi et al. (2017) reported a markedly different outcome: flavored VOOs had FFAs approximately double those of unflavored oils. This increase was attributed to their aromatization method, which involved adding fresh lemons to fresh olives prior to milling, rather than post-extraction maceration.

It is worth noting that while, in olive oil, the control showed the lowest PV and one of the lowest FFA values, the situation was different in grape seed oil (GSO), highlighting that important differences were already present in the starting matrices. For GSO, there is no dedicated European regulation comparable to that for olive oil. A refined GSO is regulated by industrial or commercial standards (e.g., ISO, DIN) and the Codex Standard for Named Vegetable Oils (CXS 210–1999, Codex Alimentarius Commission, 1999) defines not binding legislative thresholds:

Table 2
PV and FFA evaluated for each experiment of the GSO Box-Benkhen design for the UAE of lemon peels in olive oil. The different letters indicate statistical differences, evaluated through one-way ANOVA among each experiment (Exp), including the control sample (unflavored grape seed oil).

Experiments	Observations	Time (min)	M/S ratio	Temperature (°C)	PV (meqO_2/kg)	FFA (% oleic acid)
Control (unflavored GSO)	/	/	/	/	5.63 ± 1.10 bc	0.29 ± 0.01 ef
Exp1	Obs1	3.00	0.15	15.00	5.00 ± 0.00 c	0.23 ± 0.02 f
	Obs2	3.00	0.15	15.00		
Exp2	Obs3	16.50	0.05	35.00	6.15 ± 0.21 ab	0.66 ± 0.05 a
	Obs4	16.50	0.05	35.00		
Exp3	Obs5	16.50	0.05	15.00	5.15 ± 0.35 c	0.27 ± 0.04 f
	Obs6	16.50	0.05	15.00		
Exp4	Obs7	16.50	0.15	25.00	6.30 ± 0.28 ab	0.21 ± 0.03 f
	Obs8	16.50	0.15	25.00		
Exp5	Obs9	30.00	0.15	35.00	6.15 ± 0.21 ab	0.49 ± 0.09 bc
	Obs10	30.00	0.15	35.00		
Exp6	Obs11	30.00	0.05	25.00	6.65 ± 0.21 a	0.25 ± 0.01 f
	Obs12	30.00	0.05	25.00		
Exp7	Obs13	30.00	0.15	15.00	4.95 ± 0.35 c	0.28 ± 0.04 f
	Obs14	30.00	0.15	15.00		
Exp8	Obs15	3.00	0.15	35.00	6.00 ± 0.00 ab	0.22 ± 0.02 f
	Obs16	3.00	0.15	35.00		
Exp9	Obs17	16.50	0.25	35.00	6.15 ± 0.21 ab	0.37 ± 0.01 de
	Obs18	16.50	0.25	35.00		
Exp10	Obs19	30.00	0.25	25.00	6.60 ± 0.14 a	0.41 ± 0.02 cd
	Obs20	30.00	0.25	25.00		
Exp11	Obs21	16.50	0.25	15.00	4.85 ± 0.21 c	0.21 ± 0.02 f
	Obs22	16.50	0.25	15.00		
Exp12	Obs23	3.00	0.25	25.00	6.30 ± 0.28 ab	0.50 ± 0.05 b
	Obs24	3.00	0.25	25.00		
Exp13	Obs25	3.00	0.05	25.00	6.50 ± 0.00 a	0.38 ± 0.07 d
	Obs26	3.00	0.05	25.00		

in this case the limits of 0.6 % FFA and a 10 mEq O_2/kg PV are suggested (Khakbaz Heshmati et al., 2022; Laqui-Estaña et al., 2024; Martín-Torres et al., 2022). In the present study, all flavored GSOs and the related control showed PV and FFA values below these limits. Furthermore, FFA levels were lower than those reported by Shaukat et al. (2022), while PV values were comparable to those reported by Laqui-Estaña et al. (2024) for cold-pressed GSO and generally lower than those described by Khakbaz Heshmati et al. (2022). The slight increases observed here were consistent with literature evidence not only for PV but also for conjugated dienes, which may rise in the absence of protective measures such as argon flushing (Sicaire et al., 2016; Soares et al., 2020). Finally, during process optimization, particular attention was given to the extraction and quantification of antioxidant compounds, since these can interfere with lipase activity, and lipases themselves are more prone to denaturation at longer UAE times (Finimundy et al., 2020; Böger et al., 2018; Junior et al., 2020).

To further evaluate their quality, specific extinction coefficients (K232, K270, and ΔK) were measured on the flavored oils showing the lowest PV values, corresponding to the blue areas in the Box–Behnken surface plots. These parameters are determined by UV absorption: compounds formed during primary oxidation absorb at 232 nm, while secondary oxidation products absorb at 270 nm. ΔK represents the deviation of the spectral curve, calculated from absorbance at 266, 270, and 274 nm, and is mainly used to detect oil adulteration. In all cases, higher values of K232, K270, or ΔK indicate more advanced oxidation and, consequently, lower oil quality. However, as reported by Soares et al. (2020), aromatization can also affect these indices because compounds such as phenolics, organic acids, natural enzymes, and pigments are transferred from the plant matrix into the oil, potentially interfering with spectrophotometric readings.

The K232, K270, and ΔK values of the best flavored oil samples are reported in Table S1 and Table S2 for OO and GSO samples, respectively. For the OO control, K270 and ΔK remained below the Codex Alimentarius (2017) and Regulation (EU) 2022/2104 thresholds of 1.15 and 0.15, respectively, for this oil category (refined olive oil blended with virgin oils). A specific regulatory limit for K232 is not defined. No significant differences were observed through ANOVA statistical analysis between the flavored OO or GSO samples and their respective controls, except for OO Exp6. As expected, GSO and its control showed higher extinction values than OO, reflecting intrinsic differences between seed oils and olive oils oxidation. Overall, considering PV, FFA, K232, K270, and ΔK, the best-performing flavored samples were Exp1, Exp3, and Exp11 for OO, and Exp11 for GSO.

3.2. *Volatile aroma profile*

In the present study, HS-SPME-GC–MS analysis was intentionally performed in scan mode to obtain a qualitative comparative overview of the volatile profile and to compare compositional trends among aromatized oils with the unflavored ones. Results are reported in Table 3 and Table 4, for olive and grape seed oils samples, respectively.

Firstly, as for the unflavored oils, many of their VOCs are quite in line with those reported in the literature for OO and GSO (Fiorini et al., 2018; Sevindik et al., 2022). As also indicated by Sevindik et al. (2022), the lipid oxidation-indicator of hexanal is one of the most abundant compounds in grape seed oils, with even a 6 times-higher percentage, compared to OO in this case. Also (E)-2-heptenal was described by the authors in several GSO samples, while its Z-isomer may have been generated by the high temperatures of the refining process applied to the oil, especially during deodorization (An et al., 2022). These compounds need to be mentioned, since they have also low olfactory thresholds, so that they may contribute to the overall sensorial characteristics, but hardly in a positive way. Actually, in an olive oil, also a balanced hexanal/(E)-2-hexenal ratio is a quality index: if it's too high it could give unpleasant notes such as "oxidized grease" or "cardboard". Instead, the (E)-2-hexenal is generally associated with better, greener-fruity and fresher oil, as well as the alcohols of (E)-3-hexen-1-ol or (E)-2-hexen-1-ol, which are formed in the LOX pathway and were detected in OO also in this case (Fiorini et al., 2018).

On the other side, ethanol, acetic acid and butanoic acid are particularly known to be associated with the negative odors of winey, vinegary or rancid. Even if in this case the unflavored olive oil shows ethanol percentages in line with the average relative abundances reported by Fiorini et al. (2018) for commercial OOs, the acetic acid is

Table 3

Volatile organic compounds (VOCs) of the unflavored sample and of each experiment (Exp) of the Box-Benkhen design evaluated for the lemon-flavored olive oils. They are divided in chemical class and their odor descriptions are reported.

	ODOUR DESCRIPTION	RI	RI lit	OO control	Exp 1	Exp 2	Exp 3	Exp 4	Exp 5	Exp 6	Exp 7	Exp 8	Exp 9	Exp 10	Exp 11	Exp 12	Exp 13
ALKANES																	
decane	gasoline-like odor	996	1000	n.d.	1.04 ±0.17	9.15 ±1.55	1.82 ±0.50	0.67 ±0.02	3.02 ±0.17	0.89 ±0.03	1.79 ±0.33	n.d.	n.d.	0.73 ±0.10	1.84 ±0.09	0.62 ±0.03	2.60 ±0.26
dodecane	gasoline-like to odorless	1198	1200	n.d.	1.29 ±0.19	8.89 ±0.92	2.26 ±0.65	1.14 ±0.04	3.41 ±0.07	1.41 ±0.09	1.40 ±0.35	n.d.	n.d.	13.86 ±0.28	1.97 ±0.34	1.19 ±0.12	2.52 ±0.28
tridecane		1297	1300	n.d.	n.d.	0.95 ±0.23	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.12 ±0.003	0.20 ±0.03	0.10 ±0.01	0.20 ±0.05
tetradecane		1397	1400	n.d.	1.90 ±0.65	2.49 ±0.09	0.75 ±0.22	0.30 ±0.05	1.29 ±0.10	n.d.	0.82 ±0.23	n.d.	n.d.	0.45 ±0.06	0.77 ±0.01	0.32 ±0.01	0.88 ±0.10
SUM				n.d.	4.23 ±1.01	21.48 ±2.79	4.83 ±1.37	2.11 ±0.11	7.72 ±0.34	2.3 ± 0.12	4.01 ±0.91	n.d.	n.d.	15.16 ±0.44	4.78 ±0.47	2.23 ±0.17	6.2 ± 0.69
ALDEHYDES																	
decanal	citrus, waxy, fatty, floral, orange, green, aldehydic, fresh, sweet, rose	1492	1498	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.04 ±0.001	0.03 ±0.01	0.03 ±0.01	n.d.
hexanal	green, fruity, vegetable	1078	1079	6.77 ±1.35	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	21.14 ±2.07	3.76 ±0.08	n.d.	n.d.	n.d.	n.d.
2-hexenal, (E)-	green-leafy, oily aroma with fruity-green banana and apple pips nuances	1208	1218	26.72 ±6.67	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	69.14 ±1.63	7.58 ±1.95	n.d.	n.d.	n.d.	n.d.
pentanal	fermented, bready, fruity, nutty, berry	974	979	1.34 ±0.12	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
nonanal	strong fruity or floral odor	1386	1396	n.d.	0.14 ±0.02	n.d.	0.15 ±0.02	0.16 ±0.001	0.10 ±0.0005	0.37 ±0.01	0.19 ±0.02	n.d.	n.d.	0.18 ±0.01	0.20 ±0.01	0.18 ±0.02	0.15 ±0.003
SUM				34.83 ±8.14	0.14 ±0.02	n.d.	0.15 ±0.02	0.16 ±0.001	0.10 ±0.0005	0.37 ±0.01	0.19 ±0.02	90.28 ±3.70	11.34 ±2.03	0.22 ±0.01	0.23 ±0.02	0.21 ±0.03	0.15 ±0.03
ALCOHOLS																	
1-octanol	waxy type odor	1562	1561	n.d.	n.d.	n.d.	n.d.	n.d.	0.04 ±0.004	n.d.	0.05 ±0.01	n.d.	1.63 ±0.24	n.d.	n.d.	n.d.	n.d.
ethanol	mild order, like wine or whiskey	934	926	27.16 ±5.43	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3-hexen-1-ol, (E)-		1387	1384	3.66 ±0.96	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2-hexen-1-ol, (E)-	fruity	1409	1410	1.72 ±0.51	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
1-hexanol	sweet, green, herbaceous, woody	1350	1359	2.54 ±0.25	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	9.10 ±1.44	n.d.	n.d.	n.d.	n.d.	n.d.
SUM				35.08 ±7.15	n.d.	n.d.	n.d.	n.d.	0.04 ±0.004	n.d.	0.05 ±0.01	9.10 ±1.44	1.63 ±0.24	n.d.	n.d.	n.d.	n.d.
KETONES																	
cyclohexanone	minty	1279	1281	n.d.	n.d.	0.54 ±0.14	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
OXYGENATED MONOTERPENES																	
citronellal	fresh, citrus, floreal	1471	1481	n.d.	0.11 ±0.02	n.d.	n.d.	0.12 ±0.001	0.06 ±0.006	n.d.	n.d.	n.d.	n.d.	0.11 ±0.03	0.15 ±0.002	0.14 ±0.02	0.07 ±0.01
terpinen-4-ol	pepper woody earth musty sweet	1598	1600	n.d.	0.15 ±0.02	n.d.	5.98 ±0.21	0.19 ±0.004	0.23 ±0.03	0.21 ±0.001	0.27 ±0.01	n.d.	3.64 ±0.17	0.23 ±0.01	0.25 ±0.05	0.19 ±0.01	0.20 ±0.01
a-terpineol	delicate aroma of lilac flowers, sweet with an ozone tint	1694	1697	n.d.	0.37 ±0.07	0.79 ±0.14	0.29 ±0.01	0.53 ±0.02	0.51 ±0.05	0.91 ±0.27	0.60 ±0.04	n.d.	27.23 ±3.63	0.62 ±0.03	0.63 ±0.01	0.57 ±0.08	0.40 ±0.03
citral	strong, lemon-like odor	1678	1686	n.d.	18.1 ± 0.88	0.51 ±0.08	1.68 ±0.68	0.62 ±0.001	0.35 ±0.04	0.38 ±0.09	0.70 ±0.08	n.d.	n.d.	0.79 ±0.03	0.70 ±0.02	0.72 ±0.15	0.45 ±0.03

(*continued on next page*)

Table 3 (*continued*)

	ODOUR DESCRIPTION	RI	RI lit	OO control	Exp 1	Exp 2	Exp 3	Exp 4	Exp 5	Exp 6	Exp 7	Exp 8	Exp 9	Exp 10	Exp 11	Exp 12	Exp 13
linalool	floral type odor	1550	1549	n.d.	0.30 ±0.06	0.48 ±0.05	0.26 ±0.04	0.38 ±0.002	0.33 ±0.02	0.56 ±0.03	0.14 ±0.06	n.d.	6.44 ±0.54	0.42 ±0.01	0.46 ±0.01	0.39 ±0.04	0.29 ±0.02
SUM				n.d.	19.03 ±1.05	1.79 ±0.27	8.21 ±0.94	1.84 ±0.03	1.48 ±0.15	2.06 ±0.39	1.71 ±0.19	n.d.	37.31 ±4.34	2.17 ±0.11	2.19 ±0.09	2.01 ±0.30	1.41 ±0.10
MONOTERPENE HYDROCARBONS																	
p-cymene	mild pleasant odor	1259	1266	n.d.	2.74 ±0.77	2.12 ±0.05	2.24 ±0.12	2.91 ±0.40	1.51 ±0.15	1.60 ±0.42	3.77 ±0.36	n.d.	n.d.	2.86 ±0.02	2.99 ±0.49	2.91 ±0.24	3.42 ±0.13
α-pinene	herbal type odor	1014	1020	n.d.	0.90 ±0.26	n.d.	0.63 ±0.04	1.55 ±0.16	0.31 ±0.11	n.d.	1.22 ±0.01	n.d.	n.d.	1.88 ±0.07	1.46 ±0.18	1.79 ±0.04	0.80 ±0.06
α-terpinene	citrusy, woody, terpy with camphoraceous and thymol notes	1273	1280	n.d.	0.46 ±0.05	n.d.	0.25 ±0.07	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.98 ±0.07	0.73 ±0.003	0.79 ±0.09	0.35 ±0.40
d-limonene	citrus orange fresh sweet	1191	1196	n.d.	49.5 ± 1.09	49.27 ±3.85	47.55 ±10.24	61.95 ±0.35	74.59 ±23.19	63.95 ±5.47	59.80 ±0.91	n.d.	10.89 ±1.70	57.60 ±0.38	60.81 ±4.69	58.11 ±0.25	60.90 ±1.34
β–pinene	woody-green pine-like smell	1103	1108	n.d.	6.24 ±1.83	2.62 ±0.13	1.84 ±0.24	9.41 ±0.50	2.90 ±0.06	3.68 ±0.03	8.27 ±0.06	n.d.	n.d.	10.56 ±0.13	4.78 ±1.09	10.44 ±0.06	6.02 ±0.21
β-myrcene	spicy, earthy and musky	1158	1160	n.d.	0.32 ±0.04	0.15 ±0.01	0.27 ±0.03	2.25 ±0.08	0.91 ±0.04	1.28 ±0.04	2.47 ±0.52	n.d.	n.d.	3.29 ±0.10	2.40 ±0.02	3.01 ±0.21	2.19 ±0.09
y-terpinene	terpy, citrus, lime-like, oily, green with a tropical fruity nuance	1236	1243	n.d.	10.3 ± 1.72	9.36 ±0.50	26.46 ±2.30	13.71 ±0.18	6.63 ±0.20	10.59 ±1.61	12.41 ±0.34	n.d.	n.d.	n.d.	13.67 ±1.27	13.58 ±0.29	10.90 ±0.27
SUM				n.d.	70.46 ±5.76	63.52 ±4.54	79.24 ±13.04	91.78 ±1.67	86.85 ±23.75	81.1 ± 7.57	87.94 ±2.2	n.d.	10.89 ±1.70	77.17 ±0.77	86.84 ±7.74	90.63 ±1.18	84.58 ±2.5
SESQUITERPENE HYDROCARBONS																	
trans-a-bergamotene	woody warm tea	1584	1583	n.d.	0.30 ±0.07	0.95 ±0.10	0.26 ±0.05	0.44 ±0.01	0.32 ±0.02	0.66 ±0.05	0.49 ±0.05	n.d.	11.62 ±0.89	0.57 ±0.006	0.55 ±0.01	0.53 ±0.07	0.30 ±0.03
caryophyllene	spicy, woody and terpenic	1594	1585	n.d.	0.24 ±0.05	0.37 ±0.08	1.45 ±0.49	0.34 ±0.02	0.26 ±0.03	0.72 ±0.01	0.37 ±0.02	n.d.	17.11 ±2.57	0.43 ±0.02	0.44 ±0.001	0.41 ±0.04	0.23 ±0.01
SUM				n.d.	0.54 ±0.12	1.32 ±0.18	1.71 ±0.54	0.78 ±0.03	0.58 ±0.05	1.38 ±0.06	0.83 ±0.07	n.d.	28.73 ±3.3	1 ± 0.026	0.99 ±0.01	0.94 ±0.11	0.53 ±0.04
ACIDS																	
acetic acid	strong vinegar-like odor	1447	1447	12.97 ±2.97	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
propanoic acid	pungent, disagreeable, rancid odor	1544	1535	0.84 ±0.12	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
SUM				13.81 ±3.09	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Table 4

Volatile organic compounds (VOCs) of the unflavored sample and of each experiment (Exp) of the Box-Benkhen design evaluated for the lemon-flavored grapeseed oils. They are divided in chemical class and their odor descriptions are reported.

	ODOR DESCRIPTION	RI	RI lit	GSO control	Exp 1	Exp 2	Exp 3	Exp 4	Exp 5	Exp 6	Exp 7	Exp 8	Exp 9	Exp 10	Exp 11	Exp 12	Exp 13
ALKANES																	
decane	gasoline-like odor	996	1000	n.d.	n.d.	0.88 ±0.14	n.d.	0.93 ±0.06	1.09 ±0.15	1.4 ± 0.21	n.d.	1.00 ±0.16	0.62 ±0.07	1.78 ±0.33	0.82 ±0.09	0.78 ±0.14	1.58 ±0.06
dodecane	gasoline-like to odorless	1197	1200	n.d.	n.d.	2.21 ±0.04	n.d.	1.59 ±0.25	2.04 ±0.35	6.40 ±1.24	n.d.	1.62 ±0.24	0.29 ±0.04	2.66 ±0.42	1.75 ±0.06	1.80 ±0.29	2.07 ±0.31
tridecane		1297	1300	n.d.	n.d.	0.2 ± 0.01	n.d.	n.d.	0.17 ±0.03	0.16 ±0.01	n.d.	0.10 ±0.01	0.10 ±0.01	0.41 ±0.07	0.28 ±0.05	0.13 ±0.01	n.d.
tetradecane		1398	1397	n.d.	n.d.	0.89 ±0.04	n.d.	0.54 ±0.08	0.95 ±0.08	0.94 ±0.14	n.d.	0.54 ±0.05	0.39 ±0.06	1.20 ±0.24	0.47 ±0.07	0.61 ±0.06	0.62 ±0.06
SUM				n.d.	n.d.	4.18 ±0.23	n.d.	3 ± 0.39	4.25 ±0.61	8.9 ± 1.6	n.d.	3.26 ±0.46	1.4 ± 0.18	6.05 ±1.06	3.32 ±0.27	3.32 ±0.50	4.27 ±0.43
ALDEHYDES																	
hexanal	green, grassy, sharp, waxy, unripened fruit, leafy	1078	1079	41.00 ±8.44	41.22 ±3.47	0.16 ±0.03	34.46 ±4.09	0.49 ±0.09	0.05 ±0.01	0.17 ±0.03	26.94 ±4.05	0.06 ±0.00	0.06 ±0.01	0.06 ±0.01	0.52 ±0.08	n.d.	n.d.
2-heptenal, (Z)-	green, ripe cucumber, rancid, cooked fat	1314	1318	2.10 ±0.35	30.13 ±3.11	n.d.	25.76 ±2.25	0.33 ±0.05	0.07 ±0.01	0.16 ±0.02	21.66 ±0.11	0.07 ±0.00	0.05 ±0.01	n.d.	n.d.	0.05 ±0.01	0.10 ±0.04
nonanal	strong fruity or floral odor	1386	1396	n.d.	n.d.	0.10 ±0.01	n.d.	0.09 ±0.01	0.12 ±0.02	0.08 ±0.01	n.d.	0.10 ±0.01	0.09 ±0.02	0.12 ±0.00	0.06 ±0.01	0.13 ±0.02	0.04 ±0.01
pentanal	fermented, bready, fruity, nutty, berry	966	971	5.94 ±0.95	4.71 ±0.49	n.d.	9.75 ±1.44	n.d.	n.d.	n.d.	3.84 ±0.41	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
SUM				49.04 ±9.74	76.06 ±7.07	0.26 ±0.04	69.97 ±7.78	0.91 ±0.15	0.24 ±0.04	0.41 ±0.06	52.44 ±4.57	0.23 ±0.01	0.20 ±0.04	0.18 ±0.01	0.58 ±0.09	0.18 ±0.03	0.14 ±0.05
OXYGENATED MONOTERPENES																	
citronellal	fresh, citrus, floreal	1471	1481	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.10 ±0.01	n.d.
linalool	floral type odor	1550	1549	n.d.	n.d.	0.36 ±0.01	n.d.	0.41 ±0.04	0.43 ±0.05	0.28 ±0.00	n.d.	0.40 ±0.02	0.32 ±0.05	0.41 ±0.03	0.56 ±0.08	0.40 ±0.01	0.22 ±0.01
6-octen-1-ol, 3,7-dimethyl-, acetate		1659	1660	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.15 ±0.01	0.15 ±0.03	0.17 ±0.01	0.07 ±0.00
terpinen-4-ol	pepper woody earth musty sweet	1598	1600	n.d.	n.d.	0.23 ±0.03	n.d.	0.25 ±0.03	0.31 ±0.05	0.22 ±0.02	n.d.	0.23 ±0.01	0.17 ±0.03	0.33 ±0.06	0.33 ±0.06	0.26 ±0.05	0.17 ±0.01
citral	strong, lemon-like, odor	1677	1686	n.d.	n.d.	0.58 ±0.03	n.d.	0.39 ±0.00	0.69 ±0.10	0.45 ±0.03	n.d.	0.66 ±0.09	0.61 ±0.09	0.73 ±0.05	0.39 ±0.06	0.75 ±0.00	0.32 ±0.00
a-terpineol	delicate aroma of lilac flowers, sweet with an ozone tint	1693	1697	n.d.	9.23 ±1.52	0.53 ±0.01	13.18 ±1.65	0.84 ±0.09	0.60 ±0.05	0.44 ±0.01	19.44 ±2.87	0.57 ±0.02	0.50 ±0.06	0.65 ±0.03	1.35 ±0.22	0.62 ±0.04	0.38 ±0.01
SUM				n.d.	9.23 ±1.52	1.7 ± 0.08	13.18 ±1.65	1.89 ±0.16	2.03 ±0.25	1.39 ±0.06	19.44 ±2.87	1.86 ±0.14	1.6 ± 0.23	2.27 ±0.18	2.78 ±0.45	2.3 ± 0.12	1.16 ±0.02
MONOTERPENE HYDROCARBONS																	
α-pinene	herbal type odor	1014	1020	n.d.	n.d.	0.60 ±0.05	n.d.	0.86 ±0.06	1.11 ±0.08	0.84 ±0.02	n.d.	1.01 ±0.10	0.87 ±0.14	1.60 ±0.23	0.75 ±0.04	1.74 ±0.09	0.93 ±0.01
β-pinene	woody-green pine-like smell	1103	1108	n.d.	n.d.	5.08 ±0.02	n.d.	6.31 ±0.31	7.55 ±0.19	6.45 ±0.13	n.d.	7.10 ±0.01	5.59 ±0.93	10.23 ±0.41	6.28 ±0.13	10.33 ±0.15	7.16 ±0.02
β-myrcene	spicy, earthy and musky	1158	1160	n.d.	n.d.	1.20 ±0.12	n.d.	2.01 ±0.34	2.32 ±0.31	2.08 ±0.13	n.d.	2.51 ±0.08	1.96 ±0.30	2.68 ±0.33	2.10 ±0.17	2.93 ±0.22	2.16 ±0.20
d-limonene	citrus orange fresh sweet	1190	1196	n.d.	n.d.	68.69 ±1.85	n.d.	67.83 ±0.08	62.98 ±3.28	62.40 ±3.37	n.d.	64.73 ±1.00	73.67 ±13.91	55.19 ±3.72	66.27 ±1.62	57.86 ±0.87	66.70 ±0.73
y-terpinene	terpy, citrus, lime-Iike, oily, green with a tropical fruity nuance	1236	1243	n.d.	n.d.	11.60 ±0.39	n.d.	10.85 ±0.35	12.20 ±0.54	10.77 ±0.71	n.d.	12.01 ±0.40	8.91 ±1.52	11.61 ±2.10	10.30 ±0.34	12.67 ±0.58	10.70 ±0.26
sabinene	woody, spicy, citrus and terpy	1116	1108	n.d.	n.d.	0.54 ±0.01	n.d.	0.61 ±0.06	0.89 ±0.04	0.71 ±0.03	n.d.	0.86 ±0.00	0.76 ±0.10	1.30 ±0.18	0.56 ±0.04	1.38 ±0.10	0.74 ±0.01

(*continued on next page*)

Table 4 (*continued*)

	ODOR DESCRIPTION	RI	RI lit	GSO control	Exp 1	Exp 2	Exp 3	Exp 4	Exp 5	Exp 6	Exp 7	Exp 8	Exp 9	Exp 10	Exp 11	Exp 12	Exp 13
terpinolene	sweet, fresh, piney citrus with a woody old lemon peel nuance	1273	1280	n.d.	n.d.	0.56 ±0.08	n.d.	0.55 ±0.02	0.70 ±0.01	0.54 ±0.01	n.d.	0.77 ±0.14	0.33 ±0.06	0.52 ±0.10	0.46 ±0.08	0.67 ±0.03	0.41 ±0.07
α-tujene	woody, green, herbal	1020	1019	n.d.	n.d.	0.16 ±0.00	n.d.	0.18 ±0.02	0.24 ±0.04	0.21 ±0.02	n.d.	0.15 ±0.02	0.21 ±0.03	0.36 ±0.06	0.11 ±0.13	0.34 ±0.05	0.26 ±0.05
p-cymene	mild pleasant odor	1259	1266	n.d.	n.d.	2.74 ±0.37	n.d.	2.58 ±0.27	2.77 ±0.13	2.47 ±0.15	n.d.	3.06 ±0.12	2.37 ±0.40	3.20 ±0.27	2.35 ±0.02	3.40 ±0.03	3.16 ±0.14
SUM				n.d.	n.d.	91.17 ±2.89	n.d.	91.78 ±1.51	90.76 ±4.26	86.47 ±4.57	n.d.	92.2 ± 1.87	94.67 ±17.39	86.69 ±7.4	89.18 ±2.57	91.23 ±2.12	92.22 ±1.49
SESQUITERPENE HYDROCARBONS																	
trans-a-bergamotene	woody warm tea	1583	1583	n.d.	4.79 ±0.76	0.33 ±0.03	5.76 ±0.54	0.56 ±0.07	0.42 ±0.05	0.34 ±0.02	9.18 ±1.26	0.40 ±0.04	0.37 ±0.07	0.60 ±0.03	0.87 ±0.07	0.64 ±0.03	0.27 ±0.00
caryophyllene	spicy, woody and terpenic	1593	1585	n.d.	5.67 ±0.98	0.27 ±0.01	7.43 ±0.51	0.50 ±0.07	0.35 ±0.03	0.24 ±0.01	12.94 ±1.39	0.33 ±0.01	0.30 ±0.03	0.47 ±0.02	0.95 ±0.14	0.48 ±0.02	0.21 ±0.00
SUM				n.d.	10.46 ±1.74	0.6 ± 0.04	13.19 ±1.05	1.06 ±0.14	0.77 ±0.08	0.58 ±.0.03	22.12 ±2.65	0.73 ±0.05	0.67 ±0.11	1.07 ±0.05	1.82 ±0.21	1.12 ±0.05	0.48 ±0.00
ACIDS																	
acetic acid	strong vinegar-like odor.	1448	1447	7.25 ±1.89	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
propanoic acid	pungent, disagreeable, rancid odor.	1544	1535	1.02 ±0.17	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
butanoic acid	rotten fish, ammonia, rancid butter, cheesy, sweet, and rancid	1630	1624	0.63 ±0.01	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
propanoic acid-2-methyl	sour/cheesy odor	1583	1579	0.84 ±0.03	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
SUM				9.74 ±2.1	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
ALCOHOLS																	
ethanol	mild order, like wine or whiskey	934	926	18.01 ±3.47													

doubly abundant (almost 13 % here). Besides, even if GSO presents lower percentages of ethanol and acetic acid than OO, in the GSO butanoic acid and propanoic acid-2-methyl were also identified. They are also both perceived even at low concentrations, they are characterized by particularly unpleasant or rancid odors and they could also be markers of undesired rancidity or fermentations in oils and foods (Cecchi et al., 2019).However, all these just mentioned and unpleasant VOCs were undetected in the headspace of all the flavored OO and GSO samples analysed, after ultrasonic aromatization. Also the volatilization equilibrium may have been modified at the presence of those more abundant compounds extracted from lemon peels, which dominate the headspace. Thus, the endogenous VOCs of oil and the related odors could have remained trapped in the oily phase. This phenomenon of reduced or undetected vegetable-oils-associated VOCs was also noticed in oils flavored with different herbs, but in this case it is even more evident. That aspect may depend on the lower identification of minor compounds in samples due to the higher abundance of limonene, for instance: that may also be related to signal masking phenomena or competitive adsorption effects on the SPME fiber (Chahdoura et al., 2023; Perestrelo et al., 2017). The changes in relative abundancies, especially for the minor VOCs, does not reflect those in their absolute amounts, since this SPME-GC–MS analysis is discussed just in qualitative and comparative terms (Chahdoura et al., 2023; Perestrelo et al., 2017). Anyway, it was also described by Chahdoura et al. (2023) for the lemon-aromatization of olive oils through maceration or co-pressing, also in terms of the sensorial masking effect of some properties of the olive oil, including defects (Bobiano et al., 2019; Sacchi et al., 2017). Indeed, these added VOCs often have also lower odor thresholds.

In fact, in this case especially several monoterpene or sesquiterpene hydrocarbons and the oxygenated monoterpenes were introduced from lemon peels to the corresponding oil after aromatization, also thanks to polarity affinities and solubility in the oily solvent (Ayu et al., 2024). Specifically, limonene was the most abundant volatile compound in almost all the flavored OOs and GSOs (till the 74.59 % of abundance), also in accordance to its main distribution in the lemon flavedo highlighted by the scientific literature. This molecule is interestingly associated with anti-inflammatory, antioxidant properties or with a therapeutic potential in respiratory diseases or as anticancer (Piccialli et al., 2021; Santana et al., 2020; Zhou et al., 2021). It is followed by γ-terpinene in this case, with not so relevant different abundances between the samples (generally from 8.91 % to 26.46 %) (Ayu et al., 2024; Kishimoto et al., 2022). Thus, even if limonene and γ-terpinene have intermediate odor thresholds, their associated *citrus*, fresh, sweetish or green aromas may be impactful in these flavored oils.

Among other particularly impactful compounds for the lemon aroma with lower olfactory thresholds or higher abundances, α -terpinene, nonanal, decanal, citral, linalool, α and β-pinene must be mentioned (Ayu et al., 2024; Kishimoto et al., 2022; Sacchi et al., 2017). While, for instance, nonanal is tendentially a little more abundant in the flavored OOs and α-terpinene was just detected in some of the latter (their odors vary from a fruity, floral one to a citrusy, terpenic aroma), sabinene, terpinolene and α-thujene were just identified in flavored GSOs, conversely. The latter three may contribute to the odorous profile of these samples, perhaps differentiating them with their overall peppery, green, floral, fresh, earthy or resinous attributes, considering also their quite low olfactory thresholds (generally from 0.2 to 1 ppb in air) (Zhong et al., 2014).

Comparing all the different samples, the related extractive conditions of Exp8 and 9 OO or Exp1, 3 and 7 GSO seem not to be preferable also in terms of a volatile profile less rich in the mentioned lemon-characteristic VOCs. The flavored GSOs of Exp1, 3, 7 even show the highest oxidative markers of hexanal and 2-heptenal-(Z).

Interestingly, some of the samples associated with the highest PV and the highest temperature and time of extraction, such as Exp8, were also those with the lowest number of VOCs identified, reflecting what discussed above: probably the undefined peaks might include also artefacts generated during the cavitation effect of UAE (Chemat et al., 2004; Teke et al., 2023). Conversely, Exp 1 stands clearly aside among the other flavored OOs and it differentiates greatly from the Exp1 GSO, in spite of being obtained with the same extractive conditions: just the former has a really more abundant citral (18.1 %), which consists of the two isomers neral and geranial and it's generally associated with strong, lemon-like odor. That could be explained by the differences in the two vegetable oils: from their concentration of protective antioxidants or polar micro components to their diverse viscosity, density or their oil/air partition coefficients. They could all differently influence the diffusion of VOCs through them (Yara-Varón et al., 2017).

3.3. Characterization of phenolic and antioxidant profiles in low-PV samples

To further characterize the quality of the flavored oils, the samples showing the lowest peroxide values identified through the Box–Behnken design were selected for the evaluation of total phenolic content (TPC) and antioxidant activity (DPPH). This choice ensured that the assessment of bioactive enrichment was carried out on oils that exhibit the lowest oxidative degradation. The effect of the aromatization process on bioactive compounds such as phenolics, organic acids, natural enzymes, and pigments was previously highlighted by Soares et al. (2020), contributing to the studied and added healthy effects of flavored oils (Chahdoura et al., 2023). These substances can migrate from the plant matrix—in this case, lemon peels—into the oil during extraction. To evaluate this phenomenon, the total phenolic content (TPC) and antioxidant activity (DPPH assay) were measured in the flavored oils with the lowest PV values, corresponding to the blue areas of the Box–Behnken surface plots (from 3.84 to 4.78 and 4.83 to 6.30 meq O_2/kg, for OO and GSO respectively). The spectrophotometric results for olive oil (OO) and grape seed oil (GSO) are presented in Fig. 3A and 3B, respectively

In olive oil, the Total Phenolic Content (TPC, Fig. 3A) of the control sample was the lowest (54.24 ± 8.04 mg GAE/kg), while all the flavored oils showed significantly higher values. Exp1, Exp3, Exp7, and Exp11 reached the highest TPC (ranging from 158.85 ± 12.10 to 179.06 ± 6.87 mg GAE/kg), whereas Exp6 showed intermediate values (91.10 ± 15.50 mg GAE/kg). These results underline a strong capacity of the UAE processing to extract phenolic compounds from the lemon peels. The difference among Exp6 and the other flavored samples can be related to a lower matrix/solvent ratio (0.05) in this experiment.

Similarly, for antioxidant activity (DPPH, Fig. 3B), the control sample had the lowest activity (1.14 ± 0.18 μmol TE/g) and all flavored oils displayed significantly higher values, with no significant differences among them. Specifically, Exp3 and Exp11 emerged to have the highest antioxidant capacity (7.05 ± 0.11 and 7.05 ± 0.15 μmol TE/g, respectively), with statistically significant differences compared to Exp6 and Exp7 (6.72 ± 0.11 and 6.71 ± 0.03 μmol TE/g, respectively).

It is important to note that most studies in the literature address the TPC of virgin or extra virgin olive oils (EVOOs), which are naturally rich in polyphenols, typically ranging from 100 up to over 500 mg GAE/kg (Negro et al., 2019). By contrast, refined olive oil generally contains only 1–2 mg GAE/kg of phenolics, and therefore a sharp reduction in TPC is expected when it is included in blends, as in the commercial refined olive oil used in this study (Liva et al., 2025). This difference must be considered when comparing the present results with those reported for flavored EVOOs. For example, Khemakhem et al. (2015) described unflavored EVOO with a TPC almost five times higher than the unflavored olive oil used here; consequently, the relative increase in TPC after lemon peel flavoring of these refined oils was nearly twice as high as that reported by those authors that used EVOO. In contrast, the TPC values presented by Kishimoto et al. (2022) for both control EVOO and flavored samples were more comparable to the levels found in the present UAE-flavored oils. Overall, obtained results confirm the capacity of citrus by-product enrichment to raise the phenolic content of refined

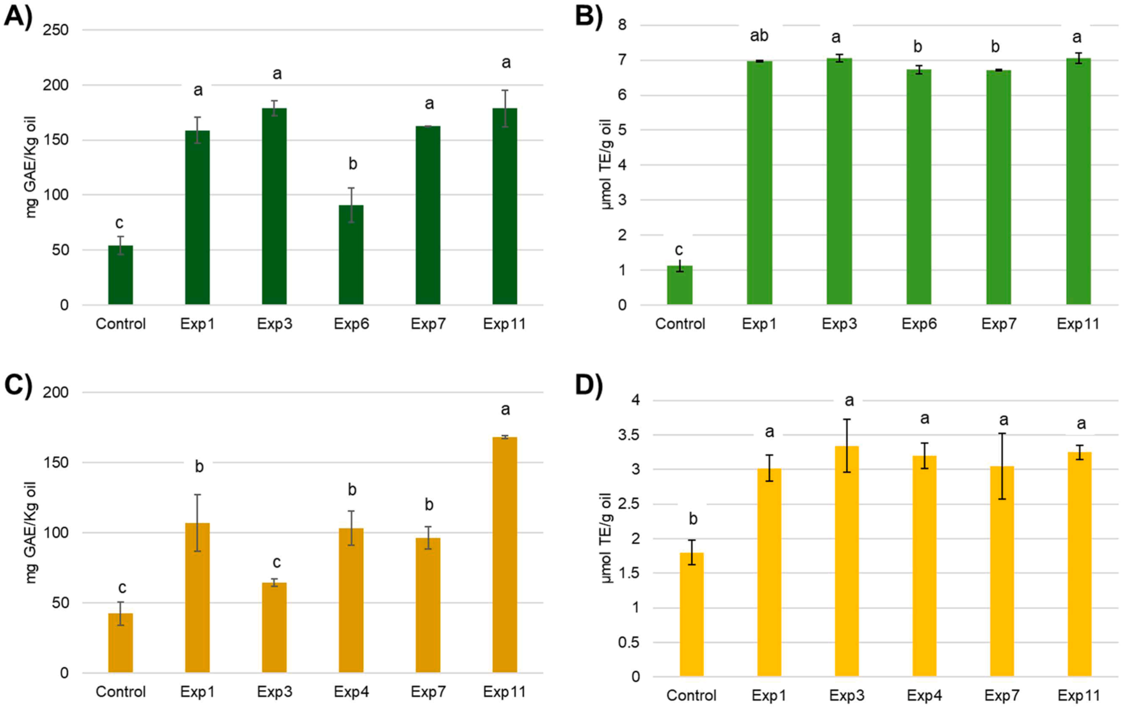

Fig. 3. TPC and DPPH of low-PV flavored oils according to Box-Benkhen experiments (Exp). A) Olive oils TPC values expressed in mg GAE/Kg oil; B) Olive oils DPPH results expressed in µmol TE/g oil; C) Grape seed soil TPC; D) Grape seeds oil DPPH. Different letters indicate statistically significant differences among samples.

oils, making their profile more similar to that of EVOO, as also observed by Sánchez de Medina et al. (2012).

In grape seed oil, the TPC results (Fig. 3C) followed the same trend as in OO samples, with the control and the Exp3 (0.05 matrix/solvent ratio) showing the lowest value (42.41±8.29 and 64.30±2.67 mg GAE/kg, respectively). Exp11 stood out with the highest TPC value (168.22±1.1 mg GAE/kg), significantly higher than all other treatments. Also in this case, the matrix/solvent ratio played a crucial role in the phenolic compound extraction, in fact Exp11 was performed with a matrix/solvent ratio of 0.25, while Exp1, Exp4 and Exp 7 with 0.15. The control sample showed the lowest antioxidant capacity in the DPPH test (Fig. 3D) with 1.80±0.18 µmol TE/g, while all flavored samples reached higher and statistically similar activities (ranging from 3.02±0.19 to 3.34±0.38 µmol TE/g).

Regarding the radical-scavenging activity (DPPH) of the flavored oils, it is important to note the particularly low value observed for the GSO control. This finding is consistent with the generally low antioxidant activity reported in the literature for grape seed oils, which varies according to cultivar and extraction method (Laqui-Estaña et al., 2024; Mohamed et al., 2016). In contrast, the OO control displayed a comparatively higher radical-scavenging activity, even exceeding some values previously reported for olive oils (Gerolim et al., 2025; Pellegrini et al., 2003).

Thus, particularly in flavored GSO, the increase in TPC after aromatization appeared to correlate more closely with the trends observed for PV and FFA than with the radical-scavenging activity (DPPH). This is consistent with the protective role of phenolic compounds during lipid oxidation, as discussed in Section 3.2. Indeed, PV and FFA showed a pattern more similar to TPC, highlighting the contribution of phenols to oxidative stability during extraction. By contrast, the overall antioxidant activity is influenced not only by phenolics but also by other compounds present in lemon peels, such as tocopherols, which are especially abundant in grape seed oil (Burčová et al., 2019; Rey et al., 2021).

In summary, aromatization significantly increased TPC and antioxidant activity in both oils compared to controls, with olive oil showing a more homogeneous response across experiments, while grape seed oil revealed clearer differentiation, in favor of Exp11. Table 5 displays a schematic overview of the overall findings from this research.

The present study is subject to some limitations, as the results are confined to the specific lipid matrices and plant materials examined and do not account for the storage stability or sensory properties of the flavored oils. Furthermore, spectrophotometric assays were used to

Table 5
Optimal UAE conditions and resulting improvements in Sorrento lemon peel–flavored oils.

Parameter	Refined olive oil (OO)	Grape seed Oil (GSO)
Optimal UAE conditions	3 min 15 °C matrix/oil ratio 0.15 g/g	16.5 min 15 °C matrix/oil ratio 0.25 g/g
Peroxide value (PV)	≈ 3.9–4.1 meq O_2/kg (comparable to control)*	≈ 5.0–5.3 meq O_2/kg (comparable to control)*
Free fatty acids (FFA)	0.11–0.17 % (well below legal limit of 1 %)	0.18–0.32 % (within industrial acceptable range)
K232 / K270 / ΔK	No significant differences from control	No significant differences from control
Increase in Total Phenolic Content (TPC)	+ 190–230 % compared to control	+ 250–300 % compared to control
Increase in Antioxidant Capacity (DPPH)	≈ + 5.5–6-fold compared to control	≈ + 1.7–2-fold compared to control
Key volatile organic compounds	Citral and limonene dominant; strong “lemon peel” aroma	Limonene and γ-terpinene, with presence of sabinene and terpinolene
Aroma effect	Strong masking of “flat / stale” notes	Masking of “rancid / acidic” notes
Main advantage of aromatization	Sensory recovery + functional enrichment of a neutral oil	Nutritional value enhancement in an oxidation-prone oil
Best-performing experiment (Exp)	Exp1	Exp11

*PV values consistent with: OO legal standards in Regulation (EU) 2022/2104; GSO quality specifications from Codex/ISO-based industrial guidelines.

provide an overall estimation of phenolic and flavonoid contents, while the identification of individual compounds was beyond the scope of this work. Likewise, VOC analysis by HS-SPME-GC–MS in scan mode enabled a mainly qualitative assessment of the volatile profile, suggesting that further targeted and quantitative investigations are warranted.

4. Conclusions

The results of this study highlight the possibility to use an optimized UAE method for the aromatization or enrichment of vegetable oils showing the case study of case of olive oil and grape seeds oils with Sorrento lemon peels. The optimal UAE conditions differ markedly between OO and GSO: olive oil requires a short and mild extraction, while grape seed oil benefits from a longer extraction time and higher matrix load, due to its inherently higher susceptibility to oxidation. Under optimized conditions, both oils show a substantial increase in phenolic content, enhanced antioxidant activity, and clear sensory improvement, including the masking of pre-existing off-flavors.

Overall, this work demonstrates a scalable and sustainable strategy for cross–supply chain valorization, where Sorrento lemon by-products and low-value refined vegetable oils are jointly upgraded into aromatic, antioxidant-enriched products, highlighting the relevance of UAE as an efficient green processing tool for the food industry. Future research should focus on untargeted metabolomic analyses to identify additional bioactive or aromatic compounds and quantificate the main ones, as well as sensory evaluations to assess consumer perception and determine how different oil matrices influence the expression of lemon flavor.

Author contributions: credit

S.C. Formal analysis, Methodology, Investigation, Writing – original draft; L.A. Conceptualization, Data curation, Validation, Visualization, Writing – review and editing; J.C., V.T., and M.F. Funding acquisition, Resources; G.S. Supervision, Project administration

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Declaration

The authors have no conflicts of interest to declare that are relevant to the content of this article.

Ethical statement - studies in humans and animals

No humans or animals were involved in the study

CRediT authorship contribution statement

Samanta Corsetti: Writing – original draft, Methodology, Investigation, Formal analysis. **Laura Alessandroni:** Writing – review & editing, Visualization, Validation, Data curation, Conceptualization. **Jean Christophe Coppin:** Resources, Funding acquisition. **Virginia Tonanni:** Resources, Funding acquisition. **Monia Floridi:** Resources, Funding acquisition. **Gianni Sagratini:** Supervision, Project administration.

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.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.afres.2026.101854.

Data availability

Data will be made available on request.

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