

In-depth study of the chemical composition and volatile profile of oils extracted from blueberry and raspberry pomace

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ABSTRACT

This study presents a comprehensive chemical and volatilomic characterization of oils exhaustively extracted from blueberry (*Vaccinium corymbosum* L.) and raspberry (*Rubus idaeus* L.) pomace. Both oils are dominated by polyunsaturated fatty acids (PUFAs): linoleic acid (36.5 % in blueberry, 49.3 % in raspberry) and α -linolenic acid (32.7 % and 28.2 %, respectively), with favorable ω -6/ ω -3 ratios of 1.75 (blueberry) and 1.12 (raspberry). Key unsaponifiable bioactives were quantified: raspberry pomace oil (RPO) contained 3620 mg/kg total tocopherols and 4244 mg/kg phytosterols; blueberry pomace oil (BPO) was notable for 509 mg/kg squalene. Polar phenolics (21–32 mg/kg) included *p*-coumaric and ferulic acids, vanillin, and flavonoids such as naringenin, enhancing antioxidant potential. Headspace SPME–GC–MS quantified 41 volatile organic compounds; BPO's aroma was dominated by terpenes and aldehydes, whereas RPO showed a richer carboxylic acid profile. The use of an in-depth analytical approach enabled a thorough assessment of the oils' compositional potential, offering a valuable reference for future applications. These findings provide a foundation for the optimization and improvement of extraction processes aimed at producing sustainable and bioactive-rich ingredients, aligning with circular economy principles to reduce waste and develop high-value products with health and sensory benefits.

1. Introduction

Over recent years, berry oils have gained popularity as niche value-added products in the cosmetic and nutraceutical fields. Berry oils encompass a variety of oils extracted from different types of berry fruits, like raspberry and blueberry. These fruits, due to their limited storage capacity and evolving consumer demands, are typically processed industrially into juice and purees (Howard et al., 2012). The by-products generated by these transformations are significant and can be upgraded in various production processes following the circular economy system (Duan et al., 2022; Klavins & Klavins, 2022; Struck et al., 2016). Utilizing process residues in the food or cosmetic industry offers

opportunities to reduce production costs and establish a new resource suitable for human use (Elimam et al., 2022; Jaouhari et al., 2023).

Typically, after juice extraction, berry pomace contains skins and seeds rich in sugars, insoluble and soluble dietary fiber, lipids and bioactive compounds (Tagliani et al., 2019). Recent studies have reported the nutritional and functional values of prebiotic oligosaccharides and phenolic compounds extracted from agrifood residues, which can be used as functional ingredient (Disca et al., 2024; Jaouhari et al., 2024).

The lipid fraction, which constitutes roughly 10 % of the pomace, is rich in unsaponifiable lipophilic compounds, including tocopherols, phytosterols, squalene, and (poly)phenols, that contribute significantly

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to human health and exhibit potent benefits in dermatological and cosmetic treatments (Mazzocchi et al., 2021; Michalak et al., 2021). Most of these molecules are naturally found in the oil contained in the seeds, with a smaller fraction derived from the fruit's skin and stems, which dissolve in the lipid fraction after extraction (Baydar et al., 2007). These components have been extensively studied and have potential *in-vivo* effects, representing excellent candidates for the development of cosmetics (Miazek et al., 2022; Vergallo, 2020). Natural lipophilic antioxidants, including tocopherols, squalene, and (poly)phenols are mainly present in seeds, fruit skins, and green parts of higher plants. Tocopherols represent the most active fat-soluble antioxidants, which act by chain-breaking electron donor mechanism to free radicals, delaying the autocatalytic lipid peroxidation processes (Barouh et al., 2022). Interestingly, α -tocopherol, which represents the most active isoform, followed by β -tocopherol, γ -tocopherol, and δ -tocopherol, is known to slow down the process of skin's photoaging induced by UV radiation exposure (Fernández-García, 2014). Phytosterols are triterpenes similar to cholesterol but with a different side-chain configuration. Over 100 types of phytosterols have been identified in fruits and vegetables, with the most abundant and active being 4-desmethyl sterols (β -sitosterol, stigmasterol, and campesterol). These compounds exhibit higher bioactivity and efficacy when esterified with fatty acids (Fernandes & Cabral, 2007). The activity of this molecular class is interesting at cosmetic level. According to Kobayashi et al. (2022), phytosterols in rice bran oil improve barrier function and aid in repairing the dermis structure by increasing hyaluronic acid and type III collagen.

Another group of molecules of crucial importance for the chemical characterization is represented by volatile organic compounds (VOCs) (Feng et al., 2024; Zheng et al., 2024). In vegetable oils, the volatile profile underpins sensory attributes such as aroma and flavor. It also reveals oxidation and other deterioration phenomena, and can act as a marker of processing conditions and product authentication (Cecchi et al., 2023; Choe & Min, 2006; Morales et al., 2005). For blueberries and raspberries, studies on lipid volatile composition have focused specifically on seed oil rather than on pomace (Bederska-Łojewska et al., 2021; Ferrão et al., 2022; Mildner-Szkudlarz et al., 2021; Pico et al., 2022).

To the authors' best knowledge, no in-depth investigation of the molecules linked to bioactive and sensory properties of the lipidic fraction extracted from blueberry and raspberry pomace has been reported so far. Most existing studies focus on the general characterization of blueberry and raspberry oils obtained solely from seeds. In an industrial context and considering a circular economy process, extracting the lipidic fraction from the whole milled pomace offers dual benefits: reducing costs by eliminating the need to separate seeds and recovering fat-soluble or partially fat-soluble compounds present in the skin.

The objective of this study was to perform a comprehensive assessment of these upcycled oils through advanced chromatographic techniques. To achieve this, a full extraction was deliberately applied to recover the widest possible range of bioactive constituents, thereby enabling a detailed compositional benchmark. This approach provides not only a thorough chemical and volatilomic profile of the oils but also a valuable reference for future optimization of greener and industry-aligned extraction processes. A key strength of this work lies in its multi-technique analytical strategy—combining GC-FID, HPLC-DAD, HS-SPME-GC-MS, and HPLC-ESI-MS—to deliver the first in-depth profiling of oils extracted from whole blueberry and raspberry pomace.

2. Materials and methods

2.1. Raw materials and sample extraction

Blueberry (*Vaccinium corymbosum* L.) and red raspberry (*Rubus idaeus* L.) pomace, from juice pressing processes, were kindly supplied by the "Polo AGRIFOOD - MIAC Scpa" (Cuneo, Italy). The raw materials

were freeze-dried and the lipidic fraction was extracted using dichloromethane for 6 h at 60 °C with a Soxhlet apparatus (Büchi Universal Extraction System B-811, Switzerland). After extraction, the solvent was entirely evaporated with a rotary evaporator (Büchi Rotavapor® R-210, Switzerland) at 40 °C. The resulting oil was then stored at −20 °C until further analysis.

2.2. Reagents and solvents

Methanol, acetonitrile (all HPLC grade), and formic acid (50 %, LC-MS grade) were purchased from Sigma-Aldrich (Milan, Italy). Ultrapure water (18.2 M Ω cm at 25 °C) was obtained by ELGA PURELAB Ultra system (M-medical, Milan, Italy).

The molecules used as internal standards such as 4-methyl-2-pentanol (≥ 98.0 %) and α -thujone (> 96.0 %), and all the compounds used as reference standards for analysis of volatile organic compounds (VOCs) were from Sigma-Aldrich (Steinheim, Germany): their purity is reported in previous manuscripts (Cecchi, Migliorini, et al., 2019; Cecchi et al., 2024). Solutions of internal standards were prepared in refined olive oil free from interfering VOCs. A solution of linear alkanes (C7–C30) in hexane was also purchased from Sigma Aldrich (Steinheim, Germany).

All the other reference compounds (polyphenols, tocopherols and phytosterols) were of analytical grade and purchased from Merck KGaA (Darmstadt, Germany).

2.3. Gas chromatographic determination of fatty acids profile

Fatty acids profile was obtained by transesterification of triacylglycerols, as previously reported by Locatelli et al. (2011). Fatty acid methyl esters (FAMES) obtained during extraction process were analyzed on a gas chromatograph Shimadzu GC-17A (Shimadzu Italia, Milan, Italy), equipped with a flame ionization detector (FID) and a DB-23 J&W column (30 m; i.d. 0.25 mm; film thickness 0.25 μ m, J&W Scientific, Folsom, CA, USA). FAME identification was achieved by comparing retention times with those of the Supelco 37 Component FAME Mix, and quantification was based on the sum of peak areas, reported as internal relative percentages.

2.4. Analysis of tocopherols, squalene and phytosterols by HPLC-DAD

A previously developed and validated method was employed for the analysis of tocopherols, pigments and squalene (Martakos et al., 2020). In brief, Briefly, the lipidic fractions were diluted in isopropanol, filtered, and injected into a HPLC system (Shimadzu 2030AD, Japan). Methanol and acetonitrile were used as mobile phases A and B, respectively, in a gradient elution. The separation was performed using a Waters (USA) Spherisorb ODS2 C18 column, with detection carried out by a photodiode array (PDA) detector at specific wavelengths for each target compound (Martakos et al., 2020). Phytosterols extraction was based on a previously established protocol (Zhang et al., 2019), involving alkaline hydrolysis, liquid-liquid extraction with n-hexane, and purification. The analysis was carried out using an Agilent 1260 High Performance Liquid Chromatography (HPLC) system equipped with a Diode Array Detector (DAD). Separation was achieved using a Waters Spherisorb ODS-2 column (250 $\times$ 4.6 mm, 5 μ m; Waters) under isocratic elution with a mobile phase of methanol and acetonitrile (55:45). The total run time was 25 min, with the column maintained at 25 °C. An injection volume of 20 μ L was used for each sample, and detection of the four phytosterols—campesterol, stigmasterol, brassicasterol, and β -sitosterol—was conducted at a wavelength of 205 nm. Calibration parameters can be found in [Supplementary Table 1](#).

2.5. Polar phenolic compounds extraction

The procedure was adapted from the solid phase extraction (SPE)

method described by Carrà et al. (2025). Approximately 0.5 g of the lipid sample was accurately weighed and dissolved in 6 mL of hexane, achieving a homogeneous solution through vortexing. The SPE cartridge (Discovery DSC-DIOL 500 mg, 3 mL; SUPELCO) was conditioned sequentially with 6 mL of methanol followed by 6 mL of hexane to prepare it for the sample loading.

The hexane-dissolved lipid sample was then loaded onto the conditioned SPE cartridge. To remove the non-polar lipid fraction, the cartridge was washed with 6 mL of hexane, which was discarded, and subsequently with 4 mL of a hexane/ethyl acetate mixture (90:10) to ensure the removal of residual lipids.

The (poly)phenolic compounds were finally eluted from the cartridge using 10 mL of methanol, and the solvent was then evaporated to dryness using a rotary evaporator under vacuum at 40 °C. The dried (poly)phenolic extract was reconstituted in 1 mL of a methanol/water mixture (50:50) and subsequently filtered through a 0.45 µm filter to ensure purity.

2.5.1. Quantification by HPLC-ESI-MS

Liquid extracts were injected in a Luna C18 column (150 mm × 2 mm; 3 µm particle diameter; Phenomenex) and phenolics compounds were quantified in an Agilent 1260 series HPLC (Palo Alto, CA, USA) with AB SCIEX Triple Quad 3500 detector (Foster City, CA, USA), equipped with an electrospray source of ionization (ESI) as recently reported by Jaouhari et al. (2024). For the separation two mobile phases were used: 0.1 % formic acid in water (solvent A) and acetonitrile with 0.1 % formic acid (solvent B). A positive/negative ionization source with turbo VTM (ion spray voltage of 4500 V), using nitrogen as nebulizer and collision gas, was employed at a source temperature of 400 °C. Multiple reaction monitoring (MRM) was used to obtain the data using Analyst 1.6.2 software (AB Sciex, Foster City, CA). Authentic phenolic compound standards were injected separately for identification and quantification. The calibration curves and fragmentation patterns employed in this study are documented in [Supplementary Table 2](#).

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

Volatile organic compounds (VOCs) in blueberry and red raspberry oil samples were analyzed according to previous HS-SPME-GC-MS methods (Cecchi et al., 2023, 2024), with slight modifications. Briefly, 3.27 g of oil sample and 0.1 g of internal standards mix solution were weighted into a screw cap vial with a capacity of 20 mL. The VOCs were pre-concentrated through adsorption onto a DVB/CAR/PDMS 50/30 µm SPME fiber (Agilent, Palo Alto, CA, USA): after sample equilibration (5 min, 90 °C), the fiber was exposed for 65 min under orbital shaking (400 rpm) in the vial headspace. The adsorbed VOCs were then desorbed for 1.7 min at 260 °C in the injection port of a 6890 N GC system equipped with a 5975 MS detector (all from Agilent, Palo Alto, CA, USA). After each analysis, a fiber backout was carried out (20 min, 260 °C). A HP-Innowax capillary column was employed for VOCs separation (50 m × 0.2 mm i.d., 0.4 µm film thickness), which was performed by applying the following oven temperature program: the initial temperature was maintained at 40 °C for 2 min, then raised to 156 °C at 4 °C/min and to 260 °C at 10 °C/min, with a final stay at 260 °C for 6 min, for a total analysis time of 47.4 min. The carrier gas was helium at 1.2 mL/min, the ion source temperature was 230 °C, while the transfer line temperature was 250 °C. The Mass Detector worked in scan mode in the mass range of 29–350 Th (Thomson), with an IE energy of 70 eV.

For VOCs identification, the comparison of mass spectra (mass spectral database NIST08), retention times and retention indices (van Den Dool & Dec. Kratz, 1963) of chromatographic peaks of samples with those of available authentic standards allowed for the identification of these target VOCs.

As for quantitative analysis, suitable quantifier and qualifier ions were selected for each molecule to be analyzed in order to achieve a complete separation also for partial co-eluting peaks. Semi-quantitative

Table 1

Fatty acid composition of blueberry pomace oil (BPO) and raspberry pomace oil (RPO), expressed as relative percentages of fatty acid methyl esters (FAMES) determined by GC-FID.

Compound name	Formula	BPO	RPO
Palmitic acid	C16:0	4.54 ± 0.02 ^d	2.53 ± 0.01 ^d
Palmitoleic acid	C16:1	0.10 ± 0.00 ^{fg}	0.09 ± 0.00 ^e
Heptadecenoic acid	C17:1	0.02 ± 0.00 ^g	0.06 ± 0.00 ^e
Stearic acid	C18:0	1.11 ± 0.01 ^e	1.13 ± 0.18 ^{de}
Oleic acid	C18:1n9cis	22.71 ± 0.04 ^c	12.22 ± 0.12 ^c
Elaidic acid	C18:1n9trans	nd	0.06 ± 0.00 ^e
Linoleic acid	C18:2n6cis	36.51 ± 0.05 ^a	49.29 ± 1.02 ^a
α-Linolenic acid	C18:3n3	32.73 ± 0.07 ^b	28.18 ± 0.70 ^b
Eicosenoic acid	C20:1	0.16 ± 0.00 ^f	0.14 ± 0.01 ^e
Eicosadienoic acid	C20:2	0.08 ± 0.01 ^{fg}	0.26 ± 0.04 ^e
Arachidonic acid	C20:4n6	0.03 ± 0.00 ^g	nd
Nervonic acid	C24:1	nd	0.02 ± 0.00 ^e
PUFA		69.35 ± 0.09	77.73 ± 1.68
MUFA		22.99 ± 0.04	12.59 ± 0.11
SFA		7.66 ± 0.10	9.69 ± 1.78
n6/n3		1.75	1.12

Results are expressed as mean ± standard deviation (n = 3). Values in the same column annotated with different superscript letters are significantly different according to ANOVA followed by Tukey's HSD test (p < 0.05).

nd: not detectable; PUFA: polyunsaturated fatty acids; MUFA: monounsaturated fatty acids; SFA: saturated fatty acids; n6: omega-6 fatty acids; n3: omega-3 fatty acids.

data were obtained after area normalization with the internal standard (i.e., α-thujone for terpenes, 4-methyl-2-pentanol for all other molecules), according to the following formula:

$$\left[\text{VOC} \left(\frac{\text{mg}}{\text{kg}} \right) \right] = \frac{A_{\text{VOC}}}{A_{\text{ISTD}}} \times \frac{m_{\text{ISTD}}}{m_{\text{sample}}} \quad (1)$$

where A_{VOC} was the area of the quantifier peak of the VOC, A_{ISTD} was the area of the quantifier peak of the internal standard, m_{ISTD} was the amount of internal standard and m_{sample} was the sample amount into the vial. A response factor of 1 was assumed for all quantitated VOCs, and a calibration curve was constructed for each compound. [Supplementary Table 3](#) provides a comprehensive list of the calibration parameters applied.

A further group of VOCs was tentatively identified calculating the retention indices of peaks of interest (van Den Dool & Dec. Kratz, 1963) after analysis of a mixture of linear alkanes C7-C30 in the same conditions of samples: tentative identification was based on the comparison of the mass spectra of the peak with that present in the standard NIST08 library database (minimum matching factor, 80 %), and of the calculated retention index with that of the NIST Standard Reference Database (van Den Dool & Dec. Kratz, 1963) as showed in [Supplementary Table 4](#).

Analyses were performed in triplicate; results were therefore expressed as mean ± standard deviation.

2.7. Statistical analysis

Statistical analyses and data visualizations were conducted using R software version 4.2.1 (Boston, USA) and GraphPad Prism version 8 (San Diego, USA). Data are reported as mean values with corresponding standard deviations (SD). Differences were assessed using analysis of variance (ANOVA), followed by Tukey's honest significant difference test, with the significance level set at 0.05.

3. Results and discussions

3.1. Characterization of fatty acid fraction

The fatty acid composition of blueberry (BPO) and raspberry pomace oil (RPO), analyzed by GC-FID, is presented in [Table 1](#). As expected, the

Table 2

Phytosterols, tocopherols, squalene and lutein results (mg/kg) in blueberry (BPO) and raspberry pomace oils (RPO) by RP-HPLC-DAD.

Compound name	BPO	RPO
Phytosterols		
Brassicasterol	507.9 ± 37.0	551.9 ± 0.7
Stigmasterol+Campesterol ¹	65.21 ± 8.59	237.0 ± 8.9
β-Sitosterol	2458 ± 113	3455 ± 49
Total	3031 ± 141	4244 ± 28
Tocopherols		
α-Tocopherol	639.5 ± 7.2	3620 ± 42
(β+γ)-Tocopherol ¹	441.7 ± 3.8	1629 ± 19
Total	1081 ± 11	5248 ± 61
Squalene	509.0 ± 19.0	nd
Lutein	11.11 ± 0.07	18.73 ± 0.15

Results are expressed as mean ± standard deviation (n = 3).

¹ Stigmasterol+Campesterol and (β + γ)-tocopherols co-elute; thus, they are quantified as a sum. nd: not detectable.

unsaturated lipid fraction was predominant, with samples containing over than 90 % of unsaturated fatty acids. Polyunsaturated fatty acids (PUFAs) were notably prevalent, comprising 69.35 % and 77.73 % of the total fatty acids in BPO and RPO, respectively. The major fatty acids, identified and quantified in terms of internal relative percentage, were linoleic acid, α-linolenic acid and oleic acid for both oils. In BPO, these fatty acids represent 36.51 %, 32.73 % and 22.71 %, respectively. The total amount of the other minor fatty acid is 8.05 % and the most representative are palmitic acid (4.54 %) and stearic acid (1.11 %). Concerning RPO, linoleic acid accounted for 49.29 % followed by α-linolenic acid (28.18 %) and oleic acid (12.22 %), with minor components such as palmitic acid (2.53 %) and stearic acid (1.13 %) also present. The results of a previous study conducted by Radocaj et al., (2014) have demonstrated similar fatty acid composition in red raspberry lipids extracted from the dried pomace. Overall, the analyses evidenced a significative presence of linoleic (LA) and α-linolenic acid (ALA), which represents the ω-6 and ω-3 essential fatty acid for humans, respectively, indicating that BPO and RPO are excellent dietary sources of these molecules. Essential fatty acids must be obtained through the diet as the human body lacks the enzymes to synthesize them. ALA serves as a biological precursor of long chain PUFAs, especially eicosapentaenoic (EPA, C20:5n-3) and docosahexaenoic (DHA, C22:6n-3) acid, through the action of desaturases and elongases. Several studies have demonstrated the cardio- and hepato-protection effects associated with ω-3 PUFAs and long-chain PUFAs metabolism. Since ω-3 PUFAs are precursors of anti-inflammatory molecules, maintaining a favorable ω-6/ω-3 ratio (ideally between 1 and 2) is highly beneficial for human health. Based on our analysis, BPO and RPO have ratios of 1.75 and 1.12, respectively. These values are consistent with those reported for various berry seed oils, including blueberry and raspberry, which range from 0.80 to 1.90 as documented by Bederska-Łojewska et al., (2021) and Luo et al., (2021). This favorable ratio underscores the potential health benefits of BPO and RPO, aligning them with other berry seed oils known for their optimum ω-6/ω-3 ratios compared to other niche oils, such as hempseed oil (~3), fenugreek oil (~2) and white mustard oil (~2) (Dubois et al., 2007).

Beyond dietary implications, the fatty acid profiles of BPO and RPO suggest useful skincare functions. LA is a major component of epidermal lipids, and its incorporation into ceramides is essential for maintaining the skin's barrier function (X. Wang et al., 2024). Topical applications of LA-rich oils has been shown to repair and strengthen the skin barrier, increase hydration and reduce transepidermal water. Additionally, LA exhibits anti-inflammatory and photoprotective activities in the skin, helping to protect against UV-induced damage and irritation.

Similarly, ALA, an ω-3 PUFA present in both oils, has anti-inflammatory effects that can modulate skin inflammation and support epidermal lipid metabolism (Balić et al., 2020). The relatively balanced ω-6/ω-3 ratio in BPO and RPO (1.75 and 1.12, respectively) may thus

help create an anti-inflammatory environment in skin formulations.

3.2. Lipid and polar bioactive compounds analysis

3.2.1. Tocopherols, phytosterols, lutein and squalene

The analysis of BPO and RPO reveals a robust presence of tocopherols, phytosterols, and carotenoids as reported in Table 2. RPO exhibited a high α-tocopherol concentration of 3620 mg/kg, contrasting with earlier studies that reported γ-tocopherol as the dominant form in raspberry seed oil (Oomah et al., 2000). This discrepancy may arise from differences in cultivar genetics, agronomic conditions, or extraction methods, particularly since our process targeted lipophilic compounds from the whole pomace. High levels of α-tocopherol and γ-tocopherol contribute to the rich antioxidant properties of RPO (Ispiryan et al., 2021). By comparison, BPO displayed lower α-tocopherol levels (639.5 mg/kg), which remain the principal component of the total tocopherols (Klavins et al., 2016; Van Hoed et al., 2009). Considering the total tocopherol content, RPO contains approximately five times higher levels (5248 mg/kg) than BPO (1081 mg/kg). When taken together, the concentrations measured in both oils are markedly higher than those generally reported for raspberry and blueberry seed oils (620–2112 mg/kg and 101–105 mg/kg, respectively (Milala et al., 2018; Parry et al., 2005; Van Hoed et al., 2009; Van Hoed et al., 2011)) which are the only available reference data, as no studies to date have focused on oils obtained from berry pomace. This difference, in total tocopherols as well as α-tocopherol content, can reasonably be attributed to the contribution of the fruit skin, which is present in the pomace matrix but absent when only seeds are processed for oil extraction.

In line with this hypothesis, a recent study by Ribalta-Pizarro et al., (2023) evaluated tocopherol distribution in grape berries (skin, pulp, and seeds) and showed that the skin is particularly enriched in α-tocopherol. The authors associated this with the higher density and developmental state of plastids in the skin, which retain photosynthetic activity and form large plastoglobules during ripening, structures known to act as reservoirs for tocopherols. In contrast, the pulp contains fewer chloroplasts, and seeds mainly harbor less differentiated plastids. These tissue-specific differences in plastid abundance and morphology help to explain why oils extracted from whole pomace, which includes skin, may display higher tocopherol levels than oils obtained exclusively from seeds.

Phytosterol content was also high, with RPO containing 4244 mg/kg and BPO 3031 mg/kg. These levels are considerably above those in oils like canola, which average around 900–1000 mg/kg, thereby underscoring the potential of these berry oils as a cholesterol-lowering agent. These values align with findings from Van Hoed et al. (2009), who reported high phytosterol concentrations in various berry seed oils, especially raspberry, which is particularly rich in β-sitosterol. The analysis identified also a significant presence of brassicasterol in both oils. Although commonly found in Brassicaceae plants, brassicasterol has also been reported in oils of other botanical origins, and its presence in our samples may reflect natural biosynthetic variability or compound concentration during pomace drying and oil extraction.

Squalene was detected in BPO at 509 mg/kg but was undetectable in RPO. This result is consistent with findings on cold-pressed berry seed oils, where squalene content varies significantly across berry species (Cheikhoussef et al., 2020). For example, cranberry seed oil exhibits squalene levels around 6715 mg/kg, while raspberry and blackberry seed oils contain much lower levels, at approximately 84 mg/kg and 170 mg/kg, respectively. Squalene is recognized for its emollient properties, which make BPO an excellent candidate for cosmetic formulations, where it can act as a moisturizing and antioxidant agent, similar to squalene's role in olive oil-based products.

Moreover, carotenoid analysis revealed that lutein, the primary carotenoid found in these oils, was present in BPO at 11.11 mg/kg and in RPO at 18.73 mg/kg. These levels align with carotenoid concentrations reported in similar berry seed oils in the literature. Parry et al. reported

Table 3

(Poly)phenolic profile of blueberry (BPO) and raspberry pomace oil (RPO) samples (mg/kg) obtained by HPLC-ESI-MS.

Individual phenolic compounds	BPO	RPO
Phenolic acids		
Ferulic acid	1.35 ± 0.02 ^{fg}	4.12 ± 0.06 ^b
<i>p</i> -Coumaric acid	12.81 ± 0.02 ^a	3.22 ± 0.03 ^c
Protocatechuic acid	2.88 ± 0.05 ^c	0.11 ± 0.00 ^e
Syringic acid	1.75 ± 0.02 ^e	0.17 ± 0.01 ^e
4-Hydroxybenzoic acid	0.25 ± 0.01 ^h	0.40 ± 0.01 ^{de}
Vanillic acid	1.51 ± 0.03 ^{ef}	0.86 ± 0.03 ^d
Salicylic acid	1.10 ± 0.00 ^g	0.33 ± 0.02 ^e
Flavonoids		
Naringenin	3.94 ± 0.08 ^b	0.34 ± 0.00 ^{de}
Quercetin	2.14 ± 0.18 ^d	0.02 ± 0.00 ^e
Luteolin	0.06 ± 0.00 ^h	nd
Catechin	nd	0.02 ± 0.00 ^e
Epicatechin	0.01 ± 0.00 ^h	0.08 ± 0.01 ^e
Phenolic aldehydes		
Ethylvanillin	0.01 ± 0.00 ^h	0.02 ± 0.00 ^e
Vanillin	3.63 ± 0.07 ^b	11.93 ± 0.38 ^a
Syringaldehyde	0.26 ± 0.00 ^h	0.14 ± 0.01 ^e
Total	31.70 ± 0.12	21.76 ± 0.45

Results are expressed as mean ± standard deviation (n = 3). Values in the same column annotated with different superscript letters are significantly different according to ANOVA followed by Tukey's HSD test ($p < 0.05$).

nd: not detectable.

similar levels of total carotenoids (beta-carotene, zeaxanthin, cryptoxanthin and lutein) in blueberry and raspberry seeds oils although the lutein findings were significantly lower (Parry et al., 2005). While these levels are lower than those in red palm oil, which typically contains over 200 mg/kg of carotenoids, the presence of lutein adds nutritional value, as it is known to protect against age-related macular degeneration. Phytosterol, tocopherol and carotenoid profiles of BPO and RPO highlight their functional potential in cosmetic applications. The elevated levels of tocopherols and phytosterols in RPO indicate its potential to provide significant antioxidant benefits. Meanwhile, BPO's moderate squalene content supports its use in skincare formulations, where moisturizing and protective properties are desirable. These findings reinforce the value of pomace oils as sustainable, high-value ingredients, as they retain much of the bioactive content of their seed oil counterparts, derived from fruit processing by-products.

3.2.2. (Poly)phenols

Polar extracts of BPO and RPO were analyzed by HPLC-ESI-MS and 15 compounds were identified as showed in Table 3. These compounds include phenolic acids (ferulic, *p*-coumaric, protocatechuic, syringic, 4-hydroxybenzoic, vanillic and salicylic acids), flavonoids (naringenin, quercetin, luteolin, catechin and epicatechin) and phenolic aldehydes (ethylvanillin, vanillin and syringaldehyde). The total content of phenolic compounds ranged from 21.76 mg/kg for RPO to 31.70 mg/kg for BPO. These concentrations are considerably lower compared to those in olive oil, which is the most studied oil rich in polar antioxidants, with values ranged from about 25 mg/kg for low-quality olive oil commercial category up to 1000 mg/kg for high-quality extra virgin olive oil commercial category (Pedan et al., 2019), but greater compared to cold-pressed grape seed oils from different grape varieties (i.e., from 0.83 to 15.16 mg/kg) (Cecchi, Innocenti, et al., 2019). Phenolics in edible oil are correlated with organoleptic properties such as bitterness and pungency, and more important with oxidative stability. In fact, these polar compounds are able to prevent lipid oxidation acting as free radical and carbonyl scavengers (Cai et al., 2021).

Phenolic acids represent the major class of (poly)phenols present in BPO (21.65 mg/kg), followed by flavonoids (6.15 mg/kg) and phenolic aldehydes (3.90 mg/kg). Detailing the phenolic profile in BPO, the pattern was dominated by *p*-coumaric acid, which contributed for nearly 40 % (12.81 mg/kg) of the total (poly)phenols. This finding diverges

Table 4

Content of the volatile organic compounds (expressed in mg/kg) quantified in blueberry (BPO) and raspberry pomace oil (RPO) samples by HS-SPME-GC-MS analysis.

Compound name	BPO	RPO	RI _{CAL} A	RI _{REF} B	quantifi/ quali ions
Saturated aldehydes					
Pentanal	nf	0.125 ± 0.002	989	980	44/58
Hexanal	1.564 ± 0.189	0.242 ± 0.002	1086	1088	44/56
Heptanal	0.145 ± 0.023	nf	1191	1197	70/55
Octanal	0.202 ± 0.025	0.024 ± 0.003	1295	1295	43/84
Nonanal	1.331 ± 0.136	0.112 ± 0.006	1401	1401	56/98
Decanal	0.252 ± 0.030	0.059 ± 0.014	1509	1511	43/112
2-Methyl-butanol	0.005 ± 0.005	nf	921	912	41/58
3-Methyl-butanol	0.004 ± 0.005	nf	926	916	44/43
Sum of saturated aldehydes	3.503 ± 0.237	0.562 ± 0.016			
Unsaturated aldehydes					
(<i>E</i>)-2-Heptenal	0.203 ± 0.008	0.030 ± 0.002	1334	1334	41/83
(<i>E</i>)-2-Nonenal	0.021 ± 0.006	nf	1549	1548	43/55
(<i>E</i>)-2-Decenal	0.013 ± 0.005	nf	1667	1650	110/70
(<i>E,E</i>)-2,4-Hexadienal	0.010 ± 0.005	0.006 ± 0.001	1419	1411	81/96
(<i>E,E</i>)-2,4-Heptadienal	0.047 ± 0.006	0.038 ± 0.001	1508	1508	81/110
(<i>E,E</i>)-2,4-Nonadienal	nf	0.010 ± 0.001	1712	1705	81/138
(<i>E,E</i>)-2,4-Decadienal	0.018 ± 0.005	0.016 ± 0.002	1826	1815	81/152
Sum of unsaturated aldehydes	0.312 ± 0.015	0.100 ± 0.003			
Aromatic/heterocyclic aldehydes					
Benzaldehyde	0.181 ± 0.006	0.105 ± 0.001	1533	1534	105/106
Sum of aromatic/heterocyclic aldehydes	0.181 ± 0.006	0.105 ± 0.001			
Ketones					
2-Butanone	nf	0.026 ± 0.006	901	900	43/72
2-Heptanone	nf	0.021 ± 0.001	1187	1185	43/58
2-Nonanone	0.013 ± 0.005	nf	1396	1398	58/43
Sum of ketones	0.013 ± 0.005	0.047 ± 0.006			
Alcohols					
Methanol	0.668 ± 0.011	nf	900	910	31/32
Ethanol	0.034 ± 0.004	nf	942	933	31/46–45
1-Hexanol	0.261 ± 0.010	0.051 ± 0.001	1351	1356	56/69
1-Octanol	0.115 ± 0.032	nf	1557	1561	56/55
1-Nonanol	0.063 ± 0.005	nf	1669	1663	56/31
1-Penten-3-ol	nf	0.027 ± 0.001	1150	1154	57/29
3-Methyl-1-butanol	0.639 ± 0.040	nf	1206	1210	55/31

(continued on next page)

Table 4 (continued)

Compound name	BPO	RPO	RI _{CAL} A	RI _{REF} B	quanti/ quali ions
2-Heptanol	nf	0.148 ± 0.007	1313	1321	45/55
(Z)-3-Hexen-1-ol	nf	0.034 ± 0.001	1375	1363	67/41
Sum of alcohols	1.780 ± 0.054	0.136 ± 0.004			
Aromatic alcohols					
Phenylethyl alcohol	1.351 ± 0.096	0.136 ± 0.004	1919	1905	91/122
Sum of aromatic alcohols	1.351 ± 0.096	0.136 ± 0.004			
Carboxylic acids					
Acetic acid	5.279 ± 0.543	10.287 ± 0.560	1442	1427	43/60
Butanoic acid	nf	0.310 ± 0.009	1647	1637	60/73
Pentanoic acid	0.061 ± 0.008	0.036 ± 0.001	1770	1762	60/73
Hexanoic acid	0.417 ± 0.029	0.858 ± 0.019	1862	1852	60/73
Sum of carboxylic acids	5.757 ± 0.096	11.491 ± 0.560			
Esters					
Ethyl Acetate	0.106 ± 0.014	nf	890	898	43/61
(Z)-3-Hexenyl acetate	nf	0.059 ± 0.001	1310	1306	67/82
Sum of esters	0.106 ± 0.014	0.059 ± 0.001			
Terpenes					
α-Pinene	1.751 ± 0.095	0.996 ± 0.052	1023	1027	93/77
D-Limonene	1.777 ± 0.048	1.072 ± 0.034	1203	1210	68/136
δ-3-Carene	3.173 ± 0.062	nf	1148	1154	93/121
β-Myrcene	nf	0.276 ± 0.018	1159	1166	93/69
Sum of terpenes	6.701 ± 0.123	2.344 ± 0.065			
Phenols					
o-Guaiacol	0.024 ± 0.005	nf	1872	1867	109/124
Phenol	0.125 ± 0.010	nf	2007	2000	94/66
Sum of phenols	0.149 ± 0.011	0.000 ± 0.000			
Total	19.853 ± 0.616	15.104 ± 0.564			

Results are expressed as mean ± standard deviation (n = 3). nf: not found.

^A RI_{CAL}: non-isothermal Kovats retention indices from temperature-programming, using the definition of van Den Dool and Dec. Kratz (1963).

^B RI_{REF}: non-isothermal Kovats retention indices from temperature-programming from Chemistry WebBook.

from the results reported by Van Hoed et al., (2011), who detected and quantified only vanillin and homovanillic acid in blueberry seed oil. The different molecules observed in BPO may be attributed to the raw material employed during oil extraction. In fact, phenolics are widely distributed in the blueberry pomace, especially in the skin, and some of these compounds can be dissolved into the oil during extraction process, enriching and increasing the phenolic pattern and the antioxidant capacity of the final product. Regarding other major constituents, vanillin was the predominant phenolic aldehyde quantified in BPO, which accounted for 11 % (3.63 mg/kg) of the total phenolic content, while among flavonoids naringenin was the most abundant, representing 12 % (3.94 mg/kg) of the total phenolics. Contrarily to BPO, phenolic aldehydes (12.09 mg/kg) are contained in greater quantities in RPO followed by phenolic acids (9.21 mg/kg) and flavonoids (0.46 mg/kg). Also RPO is particularly rich in vanillin, which accounted for a concentration of 11.93 mg/kg. Other representative compounds identified

include ferulic and *p*-coumaric acid, at concentrations of 4.12 and 3.22 mg/kg, respectively. Comparing these findings with the previous cited study, it is possible to note that the authors identified only tyrosol in raspberry oil extracted from seeds, which contrarily was not detected in our sample (Van Hoed et al., 2011). As previously described, variability in the observed results could be due to the raw material employed as well as to the fruit variety and extraction method. Given the lack of information, these findings represent the vast landscape of BPO and RPO (poly)phenols that are yet to be detailed and discussed, by providing comprehensive data of these oils, contributing valuable insights into their potential health benefits and applications.

3.3. Volatile compounds

The identification of volatile aroma compounds in this study was fundamental, providing valuable insights for both the food and cosmetic industries, particularly in flavor formulation and quality control. Through HS-SPME-GC-MS analysis, a total of 41 volatile compounds were identified by injecting authentic standards, comparing mass spectra, and examining retention times and indices (Table 4). They include saturated aldehydes, unsaturated aldehydes, aromatic/heterocyclic aldehydes, ketones, alcohols, aromatic alcohols, carboxylic acids, esters, terpenes, volatile phenols, heterocyclic compounds and hydrocarbons.

The total content of volatile compounds in BPO and RPO were 19.853 mg/kg and 15.104 mg/kg, respectively. This indicates a particularly robust aromatic profile in the blueberry lipid, a result of significant interest due to the current lack of comparable data in the existing scientific literature. Regarding RPO, the data obtained were similar than those reported in the literature, which quantified a total volatile compounds ranging from 1.91 to 15.52 mg/kg, depending on the degree of seed roasting (Mildner-Szkudlarz et al., 2021).

Carboxylic acids emerged one of the most abundant class of volatile compounds in both BPO (5.757 mg/kg) and RPO (11.491 mg/kg) representing the 29 % and 76 % of the total volatile pattern, respectively. Acetic acid was particularly prominent in BPO and RPO, with concentrations of 5.279 and 10.287 mg/kg, respectively. The high levels of acetic acid among organic acids in RPO were confirmed by Mildner-Szkudlarz et al., (2021), who analyzed the volatile profile of raspberry oil extracted from roasted seeds.

Terpenes, which imply a range of aromas such as piney, citrusy, and floral (Ferrão et al., 2022; Yang et al., 2019), showed distinctive profiles in the two oils. The total terpene content was 6.701 mg/kg in BPO and 2.344 mg/kg in RPO. In BPO, δ-3-carene was the most abundant terpene with 3.173 mg/kg, while other notable molecules included D-limonene (1.777 mg/kg) and α-pinene (1.751 mg/kg). δ-3-carene is reported to be a key aroma compound of Mango (*Mangifera indica* L.) fruit and Bergamot (*Citrus × Bergamia*) essential oil (Marzocchi et al., 2019; Pino et al., 2005).

Saturated aldehydes, which usually represent oxidation products of oleic, linoleic and linolenic acids, were present in quite high amounts in BPO (total 3.503 mg/kg), with hexanal (1.564 mg/kg) and nonanal (1.331 mg/kg) being the predominant; on the contrary the content in RPO were lower (total 0.562 mg/kg), with hexanal (0.242 mg/kg) and pentanal (0.125 mg/kg) as the main ones. These compounds are mainly involved in the characteristics floral, green and grassy odors (Y. Wang et al., 2016). A similar behavior, but with values of one order of magnitude lower was highlighted for unsaturated aldehydes

The overall ketone content was very low in BPO (0.013 mg/kg) and RPO (0.047 mg/kg). Some ketones (tentatively quantified as presented in Supplementary Table 4), such as 2-methyl alkyl ketones—oxidation products of fatty acids (Zheng et al., 2024)—were present in very low amounts in both samples, whereas norisoprenoid ketones (e.g., damascenone, ionone, and their derivatives), which are degradation products of carotenoids known for their pleasant sensory notes and low odor threshold (Mendes-Pinto, 2009), clearly discriminated the samples,

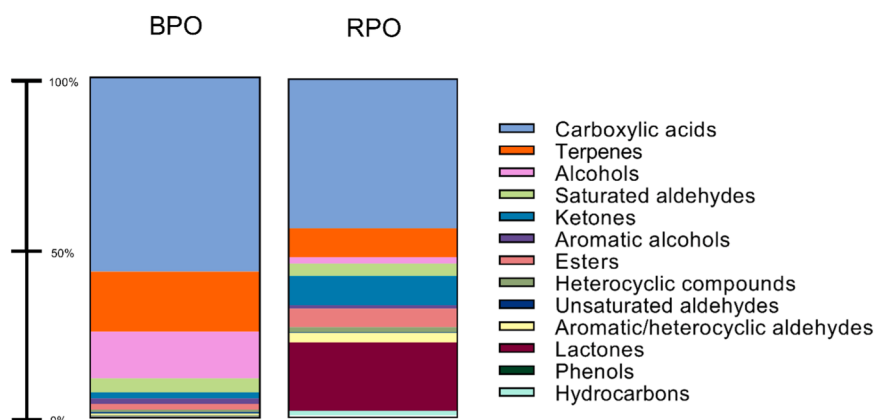


Fig. 1. Stacked bar charts representing the distribution of volatile organic compounds, categorized by chemical classes, in blueberry (BPO) and raspberry pomace oil (RPO) samples.

being present in significant amounts in RPO and entirely absent in BPO.

Other minor molecular classes contributing to the signature volatile odors of BPO and RPO included unsaturated aldehydes, aromatic aldehydes, aromatic alcohols, esters and phenols.

Overall, the comprehensive volatile profiles of blueberry and raspberry pomace oils revealed significant differences in their aroma and flavor compounds, as clearly shown in the bar charts reported in Fig. 1.

4. Conclusion

In this manuscript, an in-depth aromatic and chemical characteristics of raspberry pomace (RPO) and blueberry pomace (BPO) oils were investigated for the first time, emphasizing their relevance for cosmetic applications. The findings reveal high concentrations of bioactive compounds, including polyunsaturated fatty acids (PUFAs) with favorable ω -6/ ω -3 ratios, which are notable for their functional and health-related properties.

RPO, with its high tocopherol content, stands out as a potent antioxidant source, while BPO, which contains squalene, holds potential as a valuable ingredient in skincare formulations due to the moisturizing properties of squalene.

The volatile profile of these oils was explored for the first time, revealing a robust aromatic profile in the blueberry lipid and establishing a foundation for future research aimed at optimizing the flavor profile for various applications.

Overall, while the extraction method was chosen to ensure comprehensive analytical coverage and reveal the full compositional potential of berry pomace oils, future studies should prioritize the use of more sustainable, industry-aligned techniques to optimize extraction conditions and confirm these findings for practical application.

CRedit authorship contribution statement

Georgia Soultani: Investigation. **Nadia Mulinacci:** Writing – review & editing. **Pedro Ferreira-Santos:** Writing – review & editing, Investigation, Data curation. **Matteo Bordiga:** Supervision, Methodology. **Lorenzo Cecchi:** Writing – review & editing, Investigation, Data curation. **Yassine Jaouhari:** Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Martakos Ioannis:** Writing – review & editing, Investigation. **Thomaidis Nikolaos:** Validation. **Coisson Jean:** Writing – review & editing, Validation, Supervision, Project administration, Methodology, Funding acquisition. **Vincenzo Disca:** Writing – review & editing, Investigation. **Francesca Carrà:** Writing – review & editing, Investigation. **Fabiano Travaglia:** Writing – review & editing, Supervision, Formal analysis, Data curation.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.foohum.2026.101004](https://doi.org/10.1016/j.foohum.2026.101004).

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