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Heatmap and PCA-based evaluation of bioactive compounds and volatile profiles in aronia fruits under different drying methods

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Abstract: This study compares the bioactive compound contents and volatile profiles of products obtained from the 'Nero' variety of aronia (*Aronia melanocarpa* L.) fruits subjected to three different drying methods: freeze drying, vacuum drying, and hot air drying. Total phenolic content, total flavonoid content, and antioxidant activity were evaluated. The highest values were observed in the freeze-dried samples, with 67.9 mg GAE·g^{-1} dry weight (DW), 41.7 mg CE·g^{-1} DW, and 88.6% antioxidant activity, respectively. Vacuum drying resulted in moderate levels of bioactive compounds, while hot air drying yielded the lowest values. Volatile compound analysis, based on relative peak areas obtained from Gas Chromatography-Mass Spectrometry (GC-MS), indicated that freeze drying retained the highest levels of key aroma compounds, including hexanal (15.4%), ethyl acetate (13.9%), methyl acetate (5.7%), benzaldehyde (5.2%), 1-butanol (4.4%), linalool (3.5%), hexane (3.3%), and 2-nonanol (3.1%). The heatmap and ANOVA analyses consistently demonstrated that the drying method had a significant effect on volatile compound retention, with freeze drying identified as the most effective technique for preserving the native aroma profile. One-way ANOVA showed statistically significant differences between groups ($P < 0.001$). Multivariate analysis via Principal Component Analysis (PCA) revealed clear distinctions in both bioactive profiles and volatile compositions across the drying methods. Overall, freeze drying proved to be the most effective method for preserving both bioactive and volatile components in dried 'Nero' aronia fruits.

Keywords: *Aronia melanocarpa* L.; phenolic content; gas chromatography-mass spectrometry; multivariate analysis; postharvest processing

Aronia (*Aronia melanocarpa* L.) is a deciduous shrub of the *Rosaceae* family (Lancrajan 2012; Huang et al. 2022). Fruit colour varies among cultivars and can be red, purple, or black. Four species are generally recognised: *A. arbutifolia*, *A. melanocarpa* (Michx.), *A. prunifolia* (Marshall), and *A. mitschurinii* (Koç et al. 2024). The 'Nero' cultivar, used in this study, is sometimes classified as *Sorbaronia mitschurinii* due to its hybrid origin (*A. melanocarpa* × *Sorbus aucuparia*). However, it is more widely referred to in the literature as *Aronia melanocarpa* cv. 'Nero'. For consistency, this designation is adopted throughout the study. The 'Nero' shrub typically grows 1–2 m tall, flowers from May to June, and produces fruits that ripen between late August and September with diameters of 6.1–17.8 mm (Lancrajan 2012; Koc et al. 2024).

'Nero' is one of the most widely cultivated chokeberry genotypes, valued for its superior agronomic

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and biochemical traits compared with other cultivars. It produces relatively large, dark-black fruits with uniform size sand high yield potential, making it suitable for commercial production (Ceylan et al. 2024). Biochemically, it contains exceptionally high concentrations of anthocyanins, proanthocyanidins, and total polyphenols, surpassing cultivars such as 'Viking' and 'Galicjanka' (Gerasimov et al. 2023). These attributes confer stronger antioxidant activity and greater functional food potential. The cultivar also adapts well to diverse agro-ecological conditions, including those in Türkiye, and is increasingly favoured for both fresh consumption and processing (Xu et al. 2024a). Owing to these traits, 'Nero' has become a model cultivar in studies on bioactive compounds and technological processing methods in aronia fruits.

Aronia fruits are commonly consumed whole but are also processed into various products such as juices, juice concentrates, extracts, powders, jams, and fermented foods (Kaya and Özatay 2024). These fruits are notably rich in polyphenolic compounds, which are well recognised for their health-promoting properties. Numerous studies have demonstrated that the antioxidant and anti-inflammatory effects of polyphenols contribute to reducing the risk of cardiovascular diseases, metabolic disorders, inflammation, and neurodegenerative conditions (Kaya and Sariyer 2024).

Aronia is considered a functional fruit due to its richness in polyphenols, anthocyanins, flavonoids, and other bioactive constituents. In recent years, it has gained significant attention in the food, health, and cosmetic industries, primarily because of its potent antioxidant capacity. Owing to these properties, aronia is regarded as a valuable natural source of nutraceutical compounds, with considerable potential for supporting human health and nutrition (Gerasimov et al. 2023).

The 'Nero' variety, in particular, stands out for its high adaptability and rapidly expanding cultivation in Türkiye. Its production plays an increasingly important role in Turkish horticulture, offering considerable potential for both fresh consumption and industrial processing. However, aronia fruits have a relatively short shelf life and a limited harvest window, which restricts their availability for fresh use. Therefore, processing techniques are essential to extend shelf life and increase commercial value (Xu et al. 2024b). In this context, drying methods are widely used in the fruit and vegetable industry to prolong shelf life, ensure microbial stability, and preserve nutritional and functional components (Pateiro et al. 2022).

Drying involves the removal of water content from fruits, effectively preventing microbial growth and spoilage (Vidinamo et al. 2021). However, high processing temperatures and prolonged exposure to heat can negatively affect sensitive bioactive compounds, particularly polyphenols, flavonoids, and vitamins (Arkain and Alibas 2025). Therefore, the selection of an appropriate drying method is critical for maintaining the functional properties of the fruit (Villegas-Aguilar et al. 2024). The ability of the method to retain bioactive compounds directly influences the fruit's health-promoting potential and its value as a functional food ingredient (Yildiz et al. 2024).

The aim of this study is to compare the effects of freeze drying, vacuum drying, and hot air drying on the bioactive properties and volatile compound composition of *Aronia melanocarpa* cv. 'Nero'.

MATERIAL AND METHODS

'Nero' variety aronia fruits used in this study were harvested during the 2024 growing season under the ecological conditions of Çanakkale, Türkiye, at full ripeness. Only healthy, fully matured, and disease-free fruits were selected for analysis. After harvest, the fruits were immediately stored at 4 °C until further processing.

Drying methods

Three different drying methods were applied in the study:

Freeze drying (FD). The aronia fruits were first frozen at –50 °C and subsequently dried using a freeze dryer (Alpha 1-2 Ldplus, Martin Christ Gefriertrocknungsanlagen GmbH, Osterode am Harz, Germany) at a pressure of 0.1 mbar for 48 h. This method relies on the sublimation of water under low-temperature and vacuum conditions (Liu et al. 2022).

Freshly harvested aronia fruits were washed with tap water, stems and foreign materials were removed, and the samples were frozen at –50 °C for 24 h in a deep freezer (Arçelik, Türkiye). Approximately 200 g of fruit were evenly spread in a single layer on food-grade stainless steel trays (AISI 304) to ensure uniform exposure to the drying process. Freeze drying was performed in a laboratory freeze dryer (Alpha 1-2 Ldplus, Martin Christ Gefriertrocknungsanlagen GmbH, Germany). The process consisted of three steps: (*i*) freezing, in which samples were stabilised at –50 °C for 12 h, (*ii*) primary drying, carried out at 0.1 mbar while the temperature was gradually increased from –20 °C

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to 0 °C over 36 h; (*iii*) secondary drying, during which the temperature was raised to 20 °C under reduced pressure (0.01–0.05 mbar) for 12 h to remove bound water. Similar stepwise lyophilisation protocols have been reported for aronia and other small fruits (Skrovankova et al. 2015; Krakowska-Sieprawska et al. 2024).

Vacuum drying (VD). Fruit samples (200 g per tray) were placed in a single layer on food-grade stainless steel trays (AISI 304) and dried in a vacuum oven (VO400, Memmert GmbH + Co.KG, Germany) at 60 °C under a vacuum pressure of 200 mbar until constant weight was achieved. This approach minimises oxidative degradation of bioactive compounds by reducing oxygen availability during drying (López et al. 2017). The drying medium of 60 °C was selected as it provides an effective balance between efficient dehydration and the preservation of thermolabile compounds. Preliminary trials with aronia fruits indicated that this temperature ensured adequate drying within a reasonable time frame, while reducing the degradation of phenolic compounds and volatile constituents compared to higher temperatures. Furthermore, previous studies on small fruits, including aronia and berries, have successfully applied this temperature range for hot-air drying to maintain phenolic content and antioxidant capacity (Antony and Farid 2022).

Hot air drying (HAD). For this treatment, 200 g of fruit were evenly distributed food-grade stainless steel trays (AISI 304) and dried in a forced-air circulation oven (UN55, Memmert GmbH + Co.KG, Germany) at 60 °C for 12 h. Continuous airflow facilitated the evaporation of moisture from the fruit surface (Thi and Hwang 2016).

Dry weight of samples was determined gravimetrically by measuring constant weight after drying. The values are presented in Table 1.

Post drying sample preparation. The dried samples were ground into fine powder using a laboratory mill (IKA MF 10 basic, IKA-Werke GmbH & Co.KG, Germany) and stored at –20 °C until analysis.

Analytical methods

Total phenolic content (TPC) determination. *Extraction method:* five grams of dried aronia samples were weighed into Falcon tubes, and 20 mL of 80% methanol-water solution was added. The mixture was vortexed for 1 min to ensure homogenisation, then filtered through coarse filter paper into an Erlenmeyer flask. The filtrate was transferred to 50 mL Falcon tubes for further use.

Analysis method: in a clean Falcon tube, 100 µL of the extract and 3 400 µL of distilled water were mixed. Then, 2 000 µL of 10% Folin-Ciocalteu reagent was added, followed by vortexing for 15 s. Subsequently, 2 000 µL of sodium carbonate (Na_2CO_3) was added, and the solution was vortexed again for 15 s. The mixture was incubated in a water bath at 25 °C for 30 min. After incubation, absorbance was measured at 765 nm using a UV-Vis spectrophotometer, and results were expressed as gallic acid equivalents [mg GAE·g^{-1} dry weight (DW)] (Singleton and Rossi 1965; Slinkard and Singleton 1977).

Total flavonoid content (TFC) determination. *Extraction method:* the same methanolic extracts prepared for TPC determination were used for TFC analysis, based on the aluminium chloride colorimetric method.

Analysis method: in spectrophotometric cuvettes, 400 µL of the extract was mixed with 2.56 mL of distilled water. Then, 120 µL of 5% sodium nitrate ($NaNO_3$) solution was added, and the mixture was incubated at room temperature for 5 min. Afterward, 120 µL of 10% aluminium chloride ($AlCl_3$) solution was added and allowed to react for 6 min. Finally, 800 µL of 1 M sodium hydroxide (NaOH) was added. Absorbance was measured at 510 nm using a spectrophotometer. Results were expressed as catechin equivalents (mg CE·g^{-1} DW).

Antioxidant activity (AA) – (2,2-diphenyl-1-picrylhydrazyl (DPPH) method). *Extraction method:* the same extracts used for TPC and TFC were also used

Table 1. Dry weight of aronia fruits obtained by different drying methods

Drying method	Fresh weight	Final dry weight	Dry matter yield (%)
	(g)		
Freeze drying	200	48.5 ± 1.2^{a}	24.2^{a}
Vacuum drying	200	44.6 ± 1.5^{b}	22.3^{b}
Hot air drying	200	41.2 ± 1.8^{c}	20.6^{c}

$^{a–c}$different letters in the same column indicate statistically significant differences among drying methods (ANOVA, LSD post-hoc test; $P < 0.05$); values represent mean ± SD ($n = 3$)

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to determine antioxidant activity via the DPPH radical scavenging method.

Analysis method: the antioxidant activity was assessed based on the ability of the extract to scavenge the stable DPPH free radical. Aliquots of 25 µL, 50 µL, and 75 µL of the extract were added to separate spectrophotometric cuvettes. For the blank, 50 µL of methanol was used. Subsequently, 1.95 mL of 0.1 mmol·L^{-1} DPPH solution was added to each cuvette. Samples were incubated in the dark at room temperature for 30 min using a shaker incubator. After incubation, absorbance was measured at 517 nm using a Shimadzu UV-1800 spectrophotometer (Japan). The percentage of radical inhibition was calculated using the formula:

$$\text{Inhibition (\%)} = [(A_0 - A_1)/A_0] \times 100 \quad (1)$$

where: A_0 – the absorbance of the control (DPPH + methanol); A_1 – the absorbance of the sample.

A higher percentage inhibition value indicates a higher antioxidant capacity. The antioxidant activity was determined based on the DPPH radical scavenging method and expressed as percentage inhibition.

Volatile component analysis

Volatile compounds in aronia samples were analysed using Gas Chromatography-Mass Spectrometry (GC-MS) (Agilent Technologies, GC 6890, MS 6890N, USA). Solid Phase Microextraction (SPME) was used for sample preparation. Separation was performed with an HP-INNOWax column (60 m × 0.25 mm i.d. × 0.25 µm film thickness, Agilent Technologies). Five g of dried sample and 1 g of sodium chloride (NaCl) were placed in a 40 mL SPME vial (Supelco, USA). The mixture was incubated in a water bath at 40 °C for 20 min. A DVB/Carboxen/PDMS-coated SPME fibre (2 cm, 50/30 µm; Supelco, USA) was then exposed to the sample headspace at 40 °C for an additional 20 min. After extraction, the fibre was inserted into the GC-MS injection port for thermal desorption. The carrier gas (helium) flow was maintained at 1.0 mL·min^{-1}. The oven temperature program started at 40 °C (held for 10 min), increased at 5 °C·min^{-1} to 250 °C, and was held at 250 °C for 10 min. The total run time was 62 min. MS conditions were as follows: interface temperature, 280 °C; ionisation energy, 70 eV; mass range, 35–350 amu; scan rate, 4.45 scans·s^{-1}. Volatile compounds were identified by comparing mass spectra with those in the NIST (2008) and Wiley (McLafferty 2005) libraries. Quantification of volatile compounds was performed based on their relative peak area (%) from GC-MS chromatograms, without the use of an internal standard. Results are presented as relative percentage of the total ion chromatogram.

Statistical analysis

All analyses were conducted in triplicate, and the results are presented as mean ± SD. Statistical evaluations were performed using SAS 9.4 software (SAS Institute Inc., USA). One-way analysis of variance (ANOVA) followed by Tukey's post hoc test was applied to determine significant differences among means, with the significance level set at $P < 0.001$. Principal Component Analysis (PCA) was conducted to assess the multivariate relationships among the bioactive components. For volatile aroma analysis, a heatmap was generated to visualise the relative percentage distribution of each volatile compound across the different drying methods. Since only dried samples were analysed, the relative peak areas of each compound were normalised by setting the highest value among the drying methods to 100%, and the others were expressed proportionally. Data visualisation was performed using Python (version 3.9) with the Seaborn (version 0.11.2) and Matplotlib (version 3.4.3) libraries, while data handling was carried out using Pandas (version 1.2.5). This approach effectively illustrates which drying method preserves each compound most efficiently, thereby clarifying the impact of drying on the volatile profiles of aronia fruits.

RESULTS AND DISCUSSION

Effects of drying methods on proximate composition. Dry matter yields of aronia fruits obtained by different drying methods are presented in Table 1. These values provide an important basis for interpreting the subsequent biochemical and nutritional analyses, since moisture content and dry weight are critical indicators of drying efficiency and sample stability.

The dry matter yields of aronia fruits significantly varied depending on the drying method applied ($P < 0.05$). FD resulted in the highest dry weight (48.5 ± 1.2 g) and yield (24.2%), confirming its effectiveness in preserving solid components under low temperature and vacuum conditions. VD produced intermediate values (44.6 ± 1.5 g, 22.3%), reflecting partial structural losses due to moderate heat (60 °C) despite reduced oxygen levels. The lowest dry matter yield was obtained in HAD (41.2 ± 1.8 g, 20.6%), likely due to cell shrinkage and moisture evaporation during forced air circula-

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tion. Overall, these results indicate that FD is the most efficient method in terms of dry matter preservation, consistent with previous reports on aronia and other small fruits (Güneş 2023; Demircan et al. 2024).

In this study, three different drying methods – freeze drying, vacuum drying, and hot air drying – were applied to 'Nero' aronia fruits to evaluate their effects on bioactive compounds, antioxidant activity, and volatile profiles. Freeze drying resulted in the highest retention of TPC, flavonoids, and antioxidant activity, with values of 67.9 mg GAE·g^{-1} DW, 41.7 mg CE·g^{-1} DW, and 88.6% inhibition, respectively (Figure 1). These findings are consistent with Liu et al. (2025), who reported superior preservation of bioactive components in freeze-dried samples compared to other drying methods.

In contrast, hot air drying produced the lowest values across all parameters: 40.7 mg GAE·g^{-1} DW for TPC, 28.8 mg CE·g^{-1} DW for flavonoids, and 60.9% inhibition in antioxidant activity. These results are in agreement with Xu et al. (2024b), who observed significant degradation of phenolic compounds under hot air drying conditions. Vacuum drying yielded intermediate outcomes 51.8 mg GAE·g^{-1} DW, 33.9 mg CE·g^{-1} DW, and 71.8% inhibition demonstrating the partial effectiveness of reducing oxidative degradation by limiting oxygen exposure.

The ANOVA test revealed statistically significant differences among the three drying methods in terms of bioactive compound retention ($P < 0.001$). This outcome underscores the critical role of the selected drying technique in preserving the nutritional and antioxidant qualities of aronia fruits.

Several previous studies have examined the influence of drying temperature on the stability of bioactive compounds. For instance, Ceylan et al. (2024) reported that high drying temperatures, such as those used in hot air drying, contribute to greater degradation of phenolics due to prolonged thermal exposure a finding that aligns with our results. In contrast, vacuum and freeze drying, which operate at lower temperatures, exhibited enhanced preservation of these sensitive compounds.

Volatile compound analysis performed via GC-MS showed that freeze drying preserved a broader spectrum of aroma-active volatiles compared to other methods. This observation is consistent with Yildiz et al. (2024), who reported that low-temperature drying reduces volatilisation losses. Heatmap analysis further confirmed that hot air drying resulted in substantial losses of key aroma compounds, whereas vacuum and freeze drying more effectively preserved the volatile profiles of aronia fruits.

In conclusion, freeze drying proved to be the most effective method for preserving both the bioactive compounds and the volatile aroma profile of 'Nero' aronia fruits. The findings of this study are largely consistent with those of Liu et al. (2025) and Xu et al. (2024b), who also reported superior retention of functional and volatile compounds in freeze-dried fruits. These results suggest that freeze drying should be prioritised for maintaining the health-promoting properties and sensory characteristics of aronia. Nevertheless, vacuum drying also demonstrated considerable potential, offering a reasonable compromise between drying time and the preservation of bioactive constituents.

One of the notable strengths of this study is the comprehensive comparison of three different drying methods applied to 'Nero' variety aronia fruits cultivated under Turkish agro-ecological conditions. The use of multivariate statistical techniques, particularly PCA, enabled a multidimensional evaluation of the impact of drying methods on the overall bioactive profile. PCA results showed a clear separation of freeze-dried samples from those processed by other methods,

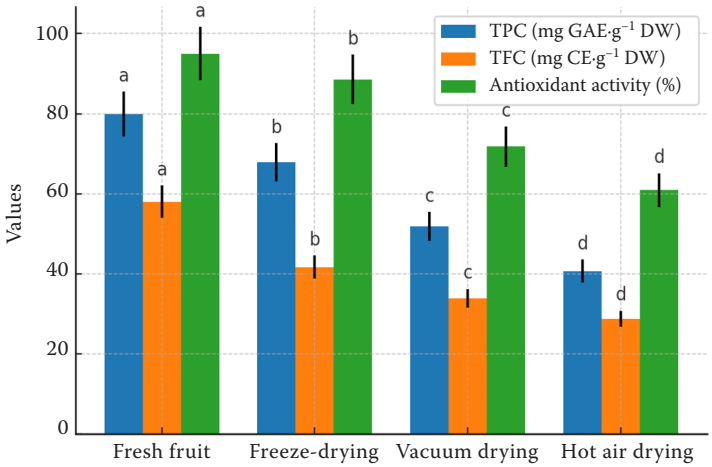

Figure 1. Effects of different drying methods on total phenolic content (TPC), total flavonoid content (TFC), and antioxidant activity of *Aronia melanocarpa* cv. Nero fruits

[a–d]different letters indicate statistically significant differences among treatments according to one-way ANOVA followed by Tukey's test ($P < 0.05$); values are expressed as mean ± SD ($n = 3$); DW – dry weight

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especially with regard to phenolic content, flavonoid levels, and antioxidant activity. These findings were further supported by visual representation (Figure 2). Similarly, Liu et al. (2017) employed PCA to assess the effects of various drying methods on aronia-like fruits and reported that freeze drying more effectively preserved the bioactive profile. These results are largely consistent with the outcomes of the present study, reinforcing the conclusion that freeze drying is the most effective method for maintaining the nutritional quality of aronia fruits.

The PCA biplot shows the distribution of 'Nero' aronia fruit samples subjected to freeze drying, vacuum drying, and hot air drying, based on TPC, flavonoid content, and antioxidant activity. The first two principal components (PC1 and PC2) explain 100% of the total variance (99.7% and 0.3%, respectively). Freeze-dried samples are positioned on the positive side of PC1, clearly associated with higher levels of flavonoids and TPC, while also positively correlated with antioxidant activity. Vacuum-dried samples appear in an intermediate position, reflecting moderate values across the measured parameters. Hot air-dried samples are separated on the negative side of PC1 and slightly negative on PC2, corresponding to lower values of bioactive compounds. The loading vectors indicate that all three bioactive traits contribute strongly to the separation pattern, highlighting freeze drying as the most effective method for preserving the chemical quality of aronia fruits.

Effect of drying methods on volatile aroma components. The effect of different drying methods on the volatile aroma components of aronia fruits was assessed using a heatmap, which visualised the percentage changes in individual compounds across each drying technique (Figure 3). The results indicate that drying methods influenced the retention of volatile compounds to varying degrees and caused the loss of specific aroma components depending on the method applied. To statistically verify these differences, the results of one-way ANOVA followed by Tukey's post hoc test are presented in Figure 3.

The analysis revealed that compounds such as linalool, limonene, ethanol, and acetic acid exhibited highly significant differences among drying methods ($P < 0.001$), whereas others like methyl acetate, hexane, and decanal showed no significant variation ($P > 0.05$).

In this study, 13 volatile compounds were consistently detected across aronia samples subjected to different drying methods. These compounds primarily aldehydes such as benzaldehyde, alcohols like *cis*3hexenol and hexanol, and esters like ethyl hexanoate correspond well with the dominant volatiles previously reported in *Aronia melanocarpa*. For instance, Wang et al. (2024) identified a wide range of volatile compounds in chokeberry juice, with benzaldehyde among the most abundant, and confirmed several aroma-active compounds through gas chromatography-olfactometry sensory analysis. Similarly, Butorová et al. (2016) reported volatile compounds across chokeberry cultivars, including alcohols, aldehydes, esters, ketones, acids, and terpenoids. In line with these findings, Kraujalytė et al. (2013) also affirmed the presence of key volatiles such as benzaldehyde, hexanal, *trans*-2-hexenal,

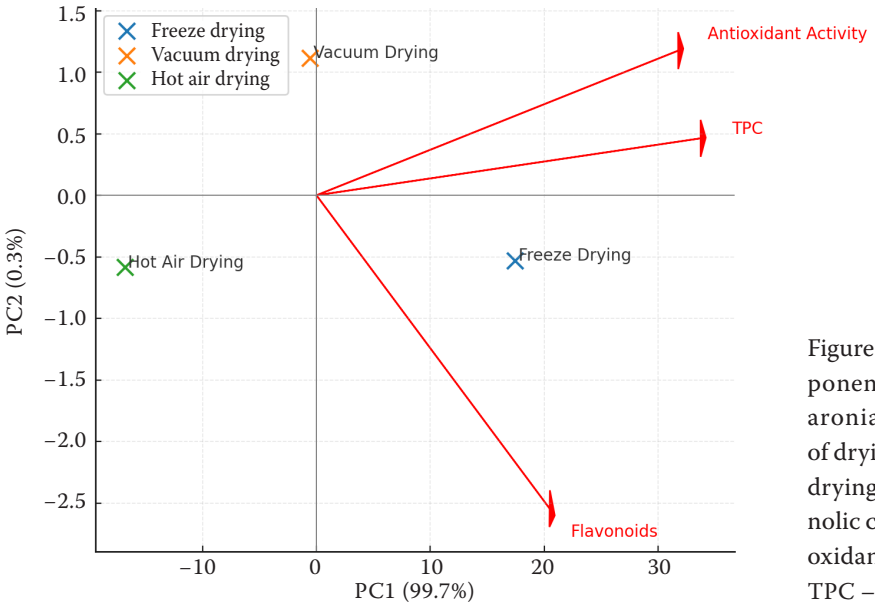

Figure 2. Two-dimensional Principal Component Analysis (PCA) biplot of 'Nero' aronia fruits showing the distribution of drying methods (freeze drying, vacuum drying, hot air drying) based on total phenolic content, flavonoid content, and antioxidant activity values.
TPC – total phenolic content

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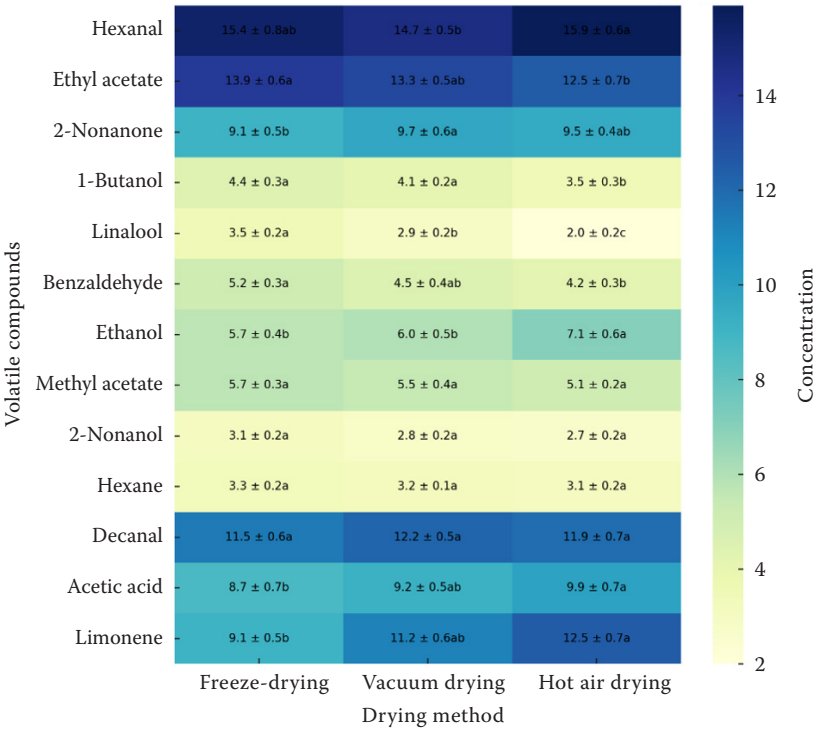

Figure 3. Heatmap illustrating the effects of different drying methods on the volatile aroma components of 'Nero' aronia fruits; the values are expressed as relative peak area percentages (% of total peak area); compounds are listed according to their retention times on the Gas Chromatography-Mass Spectrometry (GC-MS) column

Different letters indicate statistically significant differences ($P < 0.05$)

cis-3-hexenol, ethanol, hexan-1-ol, and ethyl ethanoate in *A. melanocarpa* extracts using SPME-GC-MS. The selection of 13 volatile compounds was guided by methodological rigor and analytical reliability. Specifically, only those volatiles that were both abundant and consistently detected across biological replicates (typically exceeding 1% of the total peak area) were retained for further evaluation. Minor constituents and degradation by-products, which exhibited low reproducibility and limited sensory significance, were deliberately excluded. This targeted approach was adopted to enhance the robustness and interpretative clarity of the dataset, thereby allowing the identification of a representative volatile 'fingerprint' of *Aronia melanocarpa*. While previous GC-MS investigations have reported a larger array of volatiles sometimes numbering several dozen, many of these were detected sporadically or at trace levels, often as artifacts of drying or matrix degradation. Such compounds, although analytically detectable, were deemed to have limited value in defining the core aroma profile of aronia. A total of 13 volatile compounds were characterised in dried aronia fruits, with aldehydes, alcohols, and esters being the most prominent classes. Among them, hexanal, t*rans*-2-hexenal, benzaldehyde, hexanol, *cis*-3-hexenol, *trans*-2-hexenol, hexyl acetate, and *cis*-3-hexenyl acetate appear to be the key contributors to the distinctive aroma of aronia. Similar findings have been reported by Đorđević et al. (2022), Nour (2022) and Tolić et al. (2015), who also highlighted the predominance of C6 aldehydes and alcohols as typical markers of fresh and processed aronia berries. Although previous studies using GC-MS coupled with SPME have reported dozens of volatiles, including drying-derived degradation products, the current work focused on abundant and reproducible compounds consistently detected across biological replicates. Minor volatiles present at trace levels (< 1% of the total profile) were excluded due to their low repeatability and limited sensory relevance. Therefore, the 13 compounds presented here represent the core volatile fingerprint of aronia fruits, while acknowledging that additional compounds may occur at low concentrations or under specific degradation pathways.

Hot air drying. Hot air drying resulted in the lowest retention of volatile compounds in aronia fruits. This finding aligns with the results of Ceylan et al. (2024), who reported that hot air drying leads to significant losses of volatile components due to thermal degradation. In particular, compounds such as 1-butanol (3.5%) and linalool (2.0%) were substantially reduced, and these decreases were statistically significant ($P < 0.05$, ANOVA). The pronounced decline can be explained by the physicochemical characteristics of these substances: 1-butanol, with its relatively low boiling point and high volatility, is prone to rapid evaporation, while linalool, a thermolabile monoterpene alcohol, is highly sensitive to oxidative degradation at elevated temperatures. These losses are therefore attributed to oxidative

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reactions and thermal breakdown caused by prolonged exposure to heat. Although hot air drying generally causes a reduction in most volatile compounds, hexanal exhibited a slight but statistically significant increase (from 15.4% to 15.9%, $P < 0.05$), which can be attributed to enhanced lipid oxidation under high temperatures, leading to additional formation of this aldehyde despite the overall decline in aroma integrity. Similarly, minor increases observed in other aldehydes can be explained by their generation as secondary oxidation products rather than retention of original volatiles.

Vacuum drying. Vacuum drying achieved moderate success in preserving volatile compounds. High retention rates were observed for components such as decanal (12.2%), 2-nonanone (9.7%) and acetic acid (9.2%). However, hexanal levels declined slightly from 15.4% in freeze-dried samples to 14.7% in vacuum-dried ones. This suggests that although vacuum drying limits oxygen exposure and thus reduces oxidation, it is not as effective as freeze drying in preserving volatile profiles. These findings are consistent with Coşkun et al. (2024), who reported reduced loss of bioactive components with vacuum drying, though not to the same extent as freeze drying.

Freeze drying. Freeze drying resulted in the highest retention of volatile compounds among all drying methods evaluated. Compounds such as ethyl acetate (13.9%), 1-butanol (4.4%), linalool (3.5%), benzaldehyde (5.2%), methyl acetate (5.7%), 2-nonanol (3.1%), and hexane (3.3%) remained at levels comparable to those of fresh fruits. These compounds are recognised as characteristic aroma constituents of *Aronia melanocarpa*, directly originating from the fruit's native metabolic profile rather than degradation pathways (Huang and Xu 2024; Liu et al. 2025). The preservation of these key volatiles confirms that freeze drying is highly effective in maintaining the authentic aroma fingerprint of aronia.

In contrast, volatile compounds that appeared in higher concentrations under hot-air drying but were less abundant in freeze-dried samples are more likely to represent degradation products arising from lipid oxidation and Maillard reactions rather than genuine fruit-derived volatiles. This interpretation is consistent with previous reports demonstrating that high-temperature drying promotes oxidative and thermally induced volatiles, which alter the natural fruit aroma profile (Antal et al. 2011; Ceylan et al. 2024; Li et al. 2025).

Thus, by operating at low temperature under vacuum, freeze drying minimises oxidative and thermal degradation reactions, selectively protecting native aroma compounds while suppressing the formation of secondary degradation products. This makes freeze drying the most appropriate technique for preserving both the sensory and chemical integrity of aronia fruits.

The heatmap clearly indicates that freeze drying best preserved the fresh aroma profile of the fruit. These findings are supported by the literature, which frequently highlights freeze drying as the most effective technique for maintaining fruit aroma (Cengiz et al. 2025). By operating at low temperatures under vacuum, freeze drying minimizes oxidation and thermal degradation, thereby preserving key aroma compounds (Antal 2024).

These results demonstrate that the efficiency of volatile compound preservation in aronia fruits varies significantly depending on the drying method used. Freeze drying provided the best overall retention of aroma and bioactive components, while vacuum drying offered a reasonable alternative. Hot air drying, by contrast, caused the most pronounced losses, particularly due to high temperatures and extended drying times. This study offers practical insights for selecting appropriate drying techniques for aronia fruits, especially when targeting high-quality aroma profiles for food industry applications.

CONCLUSION

This study comprehensively evaluated the effects of three different drying methods freeze drying, vacuum drying, and hot air drying on the preservation of bioactive compounds and volatile aroma constituents in 'Nero' aronia fruits. The results for total phenolic content, flavonoid levels, antioxidant activity, and volatile compounds demonstrate that the choice of drying method plays a critical role in determining the nutritional and sensory quality of the final product. Freeze drying yielded the highest retention across all evaluated parameters and was identified as the most effective technique in minimising losses of functional and aromatic components. Characteristic volatiles originally present in fresh fruit, such as ethyl acetate, linalool, benzaldehyde, and 1-butanol, were well preserved by freeze drying. In contrast, volatile compounds that appeared at higher levels in hot-air dried samples (e.g. aldehydes and Maillard-derived compounds) can be attributed to degradation and oxidation pathways rather than the native fruit profile. Thus, by protecting authentic metabolites while suppressing the formation of degradation products, freeze drying maintains

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the genuine aroma fingerprint of aronia. Vacuum drying showed intermediate performance, while hot air drying resulted in significant degradation of sensitive compounds. Notably, freeze drying by providing a low-temperature, oxygen-free environment proved to be more suitable for preserving both phenolic compounds and antioxidant activity. The findings of this study offer valuable guidance for selecting drying methods in the processing of aronia and similar high-value fruits for functional food production. In industrial applications, method selection should consider not only processing efficiency and cost but also the preservation of nutritional and sensory quality. Freeze drying should be the preferred method, especially for aronia-based products aimed at pharmaceutical or nutraceutical applications. Future research should focus on optimising drying parameters such as temperature, duration, residual moisture, and vacuum level in conjunction with evaluations of shelf life and sensory attributes. Moreover, multidisciplinary studies involving different aronia cultivars, bioavailability assessments, and consumer acceptability trials are essential to strengthen the scientific foundation for industrial-scale applications.

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