

## Article

# Influence of Protein Matrix Modification on the Preservation of Volatile Organic Compounds in Thermally Processed Fresh-Cut American Cranberry (*Vaccinium macrocarpon* Aiton)

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## Abstract

The American cranberry (*Vaccinium macrocarpon* Aiton) is widely appreciated for its high nutritional value and characteristic volatile profile, making it an important raw material for the food industry, particularly in the sector of minimally processed products. However, thermal processing may significantly alter its volatile composition, leading to aroma losses and degradation of product quality. Therefore, understanding the impact of processing conditions on aroma stability is essential for developing new post-harvest technologies that preserve the volatile profiles of processed fruit products. The aim of this study was to evaluate the effect of thermal treatment and protein matrix modification on the volatile organic compound (VOC) profile of fresh-cut cranberry fruit. Samples were heated at 50, 100, and 200 °C, with and without gelatin addition as a protein-based matrix modifier. VOCs were analysed using headspace solid-phase microextraction coupled with gas chromatography–mass spectrometry (HS-SPME–GC–MS). High-temperature treatment (200 °C) resulted in substantial changes in the volatile composition of the reference cranberry fruit and contributed to the complete loss of characteristic native terpenoids. The addition of gelatin significantly improved the retention of hydrophobic volatiles under thermal stress, maintaining D-limonene at 20.3% of the total volatile fraction, whereas this compound was absent in samples processed without the modifier. Furthermore, gelatin successfully altered the course of thermally induced reactions, reducing undesirable furfural formation from 23.95% to 7.28%. These changes indicate that protein-assisted matrix modification can effectively limit aroma degradation and preserve the volatile profile during thermal processing. The findings demonstrate the potential of this approach as a novel technological strategy for enhancing aroma stability, reducing processing-related quality losses, and supporting the development of fresh-cut cranberry products with enhanced volatile compound retention.

**Keywords:** fresh-cut cranberry; post-harvest technology; matrix modification; volatile organic compounds (VOCs); HS-SPME–GC–MS

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## 1. Introduction

Large-cranberry (*Vaccinium macrocarpon* Aiton) is a globally valued berry raw material with high health-promoting potential, resulting from an above-average concentration of biologically active compounds, such as polyphenols, organic acids, and vitamins [1,2]. Apart from the universally recognised functional and antioxidant properties, the unique olfactory profile is a key factor determining the market quality, authenticity, and initial consumer acceptance of the fruit and its processed products [3]. The volatile organic compound (VOC) fraction of fresh cranberry fruit forms a highly complex system dominated by monoterpenes, sesquiterpenes, aldehydes, and esters. Together, these components are responsible for shaping the characteristic, fresh, resinous-fruity odour attributes of the raw material [1,3].

In recent years, the global food processing sector has observed a dynamic growth of interest in minimally processed foods, with particular emphasis on fresh-cut products [4]. These offer consumers high adaptive convenience while maintaining quality characteristics as close as possible to those of the fresh raw material. However, tissue fragmentation processes (slicing, dicing), which constitute an integral stage in the preparation of fresh-cut products, generate serious technological challenges in the field of post-harvest physiology and biochemistry [4,5]. The abrupt disruption of cellular structure integrity leads to a loss of intracellular compartmentalisation, intensification of oxidation processes, and a drastic increase in the mass transfer surface area. These phenomena result in accelerated desorption and destabilisation of native VOCs, leading to rapid degradation of the initial aromatic profile [5].

This situation becomes further complicated when minimally processed products are subjected to thermal processes, commonly applied to ensure microbiological stability or during the incorporation of fruits into complex, multi-component food systems [6]. Exposure to elevated temperature induces the non-enzymatic breakdown of thermolabile components, the sublimation of natural phenolic acids, and the activation of the Maillard reaction cascade and caramelisation [7,8]. Consequently, irreversible loss of native aroma markers occurs in favour of the *de novo* synthesis of volatile substances with caramel, burnt, and smoky characteristics, such as furfural or benzoic acid [9,10]. This phenomenon directly degrades the organoleptic properties and reduces the overall final quality of the finished product [11,12].

In view of the above, one of the priority directions in contemporary food engineering research is the search for innovative post-harvest technologies capable of effective protection, preservation, and stabilisation of the volatile profile of minimally processed raw materials [4,12]. A highly promising solution in this area is the controlled modification of the fruit matrix microenvironment using edible biopolymers [13]. Protein macromolecules, including commonly used gelatin, possess a specific spatial structure and the presence of amino acid side chains that exhibit the ability to form physicochemical sorption complexes with volatile organic ligands [13]. The introduction of an exogenous protein matrix into a fresh-cut product can therefore act as a molecular barrier trap limiting the diffusion of VOCs, as well as a factor modifying and controlling the pathways of thermally induced non-enzymatic browning reactions [10,13].

Considering these factors, the aim of this study was to investigate the effect of modifying the fruit matrix with an exogenous protein biopolymer (gelatin) on the stability, retention, and transformation pathways of the volatile organic compound (VOC) profile in minimally processed (fresh-cut) products from large-cranberry (*Vaccinium macrocarpon* Aiton), subjected to varied thermal treatment (50 °C, 100 °C, and 200 °C). The undertaken instrumental analyses (HS-SPME/GC-MS) aimed to verify the hypothesis of whether the targeted introduction of protein structures can constitute an effective post-harvest technology capable of the physicochemical immobilisation of valuable aroma components and

the successful inhibition of the emission of volatile products of thermal destruction. The developed solution aims to indicate a new processing pathway for enhancing the quality and preserving the optimal sensory and aromatic attributes of fresh-cut food under thermal processing conditions.

## 2. Materials and Methods

### 2.1. Research Material

The research material consisted of large-cranberry (*Vaccinium macrocarpon* Aiton) fruit of the 'Pilgrim' cultivar. The harvest of the raw material at harvest maturity was conducted in the third decade of October 2025 on a commercial plantation located in Nowiny (Radomyśl nad Sanem, Poland). The fruit was transported to the laboratory under refrigerated conditions (4 pm 1 °C) and subjected to testing procedures within 24 h of harvest. In order to implement the treatment aimed at protecting the volatile compound profile, chemically pure food-grade gelatin (porcine-derived, Type A, with a standard gel strength of approximately 180 Bloom, and purchased from Lidl Polska Sp. z o.o. Sp. k., Tarnowo Podgórne, Poland) was applied as an exogenous modifier of the fruit matrix and an amino group donor for the Maillard reaction. This commercially available culinary gelatin is characterised by an isoelectric point (*pI*) in the range of 7.0–9.0, and its well-defined macromolecular protein network serves as an effective matrix for the physical entrapment and chemical stabilisation of hydrophobic volatile organic compounds under thermal stress.

### 2.2. Preparation of Fresh-Cut Products and Experimental Design

To simulate the processing conditions of minimally processed (fresh-cut) fruit, the raw material was subjected to quality selection, surface decontamination by washing in deionised water for 3 min, and subsequent drying in an air stream at ~20 °C for 30 min under a constant air-flow velocity of 1.0 m s<sup>-1</sup> to completely remove surface moisture without causing structural dehydration. The fruit was then longitudinally fragmented along its axis of symmetry, which allowed for the exposure of the internal pulp structures and the homogenisation of the surface area for mass transfer and volatile organic compound (VOC) release. The fresh-cut product prepared in this manner was used to establish two experimental series in a triplicate design:

- Series I (reference fresh-cut matrix): Exposure vessels (100 cm<sup>3</sup> conical Erlenmeyer flasks) were filled with 30.0 ± 0.1 g of the fragmented cranberry fruit.
- Series II (modified fresh-cut matrix): In order to investigate the effect of matrix modification on the retention of volatile organic compounds, 30.0 ± 0.1 g of fruit and 10.0 ± 0.1 g of gelatin in the form of a dry powder were introduced into the exposure vessels. The components were thoroughly mixed to ensure an even distribution of the powder across the fragmented fruit surfaces, establishing a specific dosage of 333.3 mg of gelatin per gram of fruit (component mass ratio of 3:1). This specific fruit-to-gelatin ratio was adopted to establish a robust model system. The high protein-to-substrate density was essential to ensure a tight macromolecular hydrogel network capable of physical volatile entrapment during extreme thermal stress (up to 200 °C), while simultaneously providing a sufficient concentration of amino-group donors to demonstrably alter the pathways of thermally induced reactions via covalent binding. From a practical standpoint, such concentrations align with industrial developments in high-protein edible coatings, texturized fruit snack matrices, and co-lyophilized intermediate food systems where high biopolymer concentrations are required to maintain structural and chemical integrity.

Immediately after dosing, the exposure vessels were hermetically sealed with multi-layer aluminium foil, providing a diffusion barrier that protected the profile of the VOCs released into the headspace and allowed for the lossless penetration of the septum by the SPME applicator needle.

### 2.3. Process Parameters of Thermal Treatment

In order to evaluate the thermal stability of volatile organic compounds and the susceptibility of the fresh-cut product to thermal degradation, the prepared samples were subjected to thermal treatment processes at a constant exposure time ( $t = 45$  min), applying varying levels of thermal stress:

- Variant T0—control sample (T0): freshly prepared fresh-cut product not subjected to thermal treatment.
- Variant T50 (mild thermal treatment): incubation in a water bath coupled with a magnetic stirrer (model ARE, VELP Scientifica, Usmate Velate, Italy) at a temperature of  $50 \pm 1$  °C.
- Variant T100 (standard pasteurisation): exposure in a chamber dryer with forced air circulation (model SLW 115 STD, POL-EKO-APARATURA, Wodzisław Śląski, Poland) at a temperature of  $100 \pm 1$  °C.
- Variant T200 (intensive thermal processing): exposure in a chamber dryer with forced air circulation (model SLW 115 STD, POL-EKO-APARATURA) at a temperature of  $200 \pm 1$  °C.

The utilisation of distinct heating media (a water bath for 50 °C and a forced-air chamber dryer for 100 °C and 200 °C) was dictated by the physical limitations of water as a heat-transfer fluid at high temperatures, as well as the technological intent to simulate different industrial processing regimes (mild liquid-phase conditioning vs. intensive dry-thermal processing). To standardise the volatile evaporation conditions and minimize the impact of external heat-transfer dynamics, all samples across all variants were processed inside identical, hermetically sealed, gas-tight glass exposure vessels, ensuring that VOC accumulation in the headspace depended strictly on the internal matrix temperature. Furthermore, this broad temperature gradient was strategically designed to anchor the experiment in both traditional and emerging food processing domains. While the 50 °C and 100 °C treatments closely simulate conventional industrial operations such as mild thermal conditioning, blanching, and pasteurisation of fresh-cut commodities, the inclusion of the extreme 200 °C parameter served a dual purpose. Mechanistically, it established an accelerated thermal degradation model necessary to rigorously evaluate the peak entrapment capacity of the protein matrix and observe the inhibition kinetics of advanced Maillard reaction by-products like furfural. From an applied perspective, this high-temperature boundary reflects the modern culinary and industrial utilisation of fresh-cut cranberry as a raw functional ingredient subjected to intensive thermal treatments, including commercial baking, roasting, and convective air-frying, where local product surfaces encounter severe thermal gradients.

### 2.4. Isolation of Sensory Markers by HS-SPME

The profiling of volatile organic compounds determining the sensory attributes of the processed fresh-cut product was performed using headspace solid-phase microextraction (HS-SPME). Immediately after the completion of the thermal operations, the exposure vessels were thermostated in a water bath at 45 °C to achieve sufficient analyte vapour pressure and to establish thermodynamic equilibrium within the system. The sorption of odour compounds was conducted using a commercial SPME fibre with a 100 µm station-

ary phase film thickness, coated with polydimethylsiloxane (PDMS) (Supelco, Bellefonte, PA, USA). The extraction time of the analytes from the headspace was 30 min in each case.

## 2.5. GC-MS Analysis and Identification of Volatile Odour Profiles

Following the completion of the sorption stage, the SPME fibre was subjected to direct thermal desorption in the injector of a Varian 450-GC gas chromatograph (Varian, Inc., Palo Alto, CA, USA) coupled with a 240-MS mass spectrometer (Varian, Inc., Palo Alto, CA, USA). The desorption process, releasing the analytes onto the column, was conducted at 250 °C for 5 min in splitless mode. The chromatographic separation of the components responsible for the sensory attributes was performed on an HP-5 capillary column (30 m length, 0.25 mm internal diameter, 0.25 µm stationary phase film thickness; Agilent Technologies, Santa Clara, CA, USA). Helium was used as the carrier gas at a constant flow rate of 1 cm min<sup>-1</sup>. The temperature programme of the column oven was as follows: the initial oven temperature was 50 °C and was maintained for 5 min, then increased at a rate of 10 °C min<sup>-1</sup> to a temperature of 300 °C, and held for 5 min. Chromatographic data acquisition and mass spectra (MS) recording were performed using MS Data Review software v. 6.9.1, integrated with the NIST07 mass spectral reference database (National Institute of Standards and Technology, Gaithersburg, MD, USA). The qualitative identification of the VOCs underlying the sensory characteristics of the cranberry was carried out by comparing the experimental mass spectra with the spectral profiles of the reference library.

## 2.6. Statistical Analysis

Trace compounds were defined as signals below the limit of quantification (LOQ), corresponding to a signal-to-noise ratio (S/N) of 10:1 and a relative peak area <0.01%. Data are expressed as mean ± standard deviation (SD) ( $n = 3$ ). Statistical analysis was performed using one-way ANOVA followed by Tukey's HSD test ( $p < 0.05$ ). Different lowercase superscript letters (a, b, c...) indicate significant differences among temperature treatments within each matrix, while uppercase letters (A, B) indicate significant differences ( $p < 0.05$ ) between the reference matrix (Table 1) and the gelatin-modified matrix (Table 2) at identical thermal conditions.

**Table 1.** Effect of thermal treatment temperature on the volatile organic compound (VOC) profile in fresh-cut products from large-cranberry (*Vaccinium macrocarpon* Aiton) reference matrix (without modifier).

| No. | RT<br>[min] | Peak Share in the Chromatogram<br>[%] |                            |                            |                            | Systematic Substance Name                        | Ordinary Compound<br>Name       | m/z<br>(100%)/M <sup>+</sup> | No CAS     |
|-----|-------------|---------------------------------------|----------------------------|----------------------------|----------------------------|--------------------------------------------------|---------------------------------|------------------------------|------------|
|     |             | T50                                   | T100                       | T200                       | T0                         |                                                  |                                 |                              |            |
| 1   | 4.88        | -                                     | -                          | 23.95 ± 02.11 <sup>B</sup> | -                          | 2-Furaldehyde                                    | Furfural                        | 96/96                        | 98-01-1    |
| 2   | 6.49        | 3.74 ± 0.23 <sup>aA</sup>             | 7.5 ± 0.11 <sup>bB</sup>   | -                          | 11.27 ± 0.97 <sup>bA</sup> | Phenylethylene                                   | Styrene                         | 104/104                      | 100-42-5   |
| 3   | 8.22        | 2.63 ± 0.09 <sup>bB</sup>             | 1.67 ± 0.04 <sup>a</sup>   | -                          | 3.94 ± 0.11 <sup>cA</sup>  | -                                                | Benzaldehyde                    | 77/106                       | 100-52-7   |
| 4   | 8.30        | -                                     | -                          | 2.8 ± 0.28                 | -                          | 5-Methylfurfural                                 | -                               | 110/110                      | 620-02-0   |
| 5   | 8.53        | -                                     | trace                      | -                          | -                          | 2H-Pyran, 2-ethenyltetrahydro-2,6,6-trimethyl-   | Limetol                         | 139/154                      | 7392-19-0  |
| 6   | 9.65        | -                                     | -                          | 3.45 ± 0.16 <sup>A</sup>   | -                          | 4-methyl-1-isopropylbenzene                      | p-Cymene                        | 119/134                      | 99-87-6    |
| 7   | 9.74        | -                                     | 2.66 ± 0.24 <sup>bA</sup>  | 1.3 ± 0.07 <sup>a</sup>    | -                          | -                                                | D-Limonene                      | 68/136                       | 138-86-3   |
| 8   | 9.79        | 2.63 ± 0.08 <sup>a</sup>              | 2.45 ± 0.03 <sup>a</sup>   | -                          | 3.91 ± 0.08 <sup>b</sup>   | 1,3,3-Trimethyl-2-oxabicyclo[2.2.2]octane        | Eucalyptol                      | 43/154                       | 470-82-6   |
| 9   | 10.89       | 3.56 ± 0.07 <sup>b</sup>              | 7.36 ± 0.17 <sup>c</sup>   | 2.47 ± 0.02 <sup>aA</sup>  | -                          | 4-Isopropylidene-1-methylcyclohexene             | Terpinolene                     | 93/136                       | 586-62-9   |
| 10  | 11.00       | -                                     | -                          | trace                      | -                          | -                                                | methyl benzoate                 | 105/136                      | 93-58-3    |
| 11  | 11.17       | -                                     | -                          | 1.65 ± 0.02 <sup>A</sup>   | -                          | α-trans-γ-trans-Sorbic acid                      | Sorbic acid                     | 97/112                       | 110-44-1   |
| 12  | 11.24       | -                                     | -                          | trace                      | -                          | -                                                | 2-methyl benzofuran             | 131/132                      | 4265-25-2  |
| 13  | 12.33       | 5.88 ± 0.16 <sup>aA</sup>             | 5.88 ± 0.21 <sup>a</sup>   | -                          | -                          | -                                                | 4-Ethylphenol                   | 107/122                      | 123-07-9   |
| 14  | 12.51       | -                                     | 4.81 ± 0.21 <sup>b</sup>   | 53.34 ± 0.13 <sup>cB</sup> | 2.98 ± 0.04 <sup>aA</sup>  | -                                                | Benzoic acid                    | 105/122                      | 65-85-0    |
| 15  | 12.72       | 9.42 ± 0.25 <sup>aB</sup>             | 14.95 ± 0.18 <sup>bA</sup> | -                          | 26.4 ± 0.17 <sup>cA</sup>  | (S)-2-(4-Methyl-3-cyclohexenyl)-2-propanol       | α-Terpineol                     | 59/154                       | 98-55-5    |
| 16  | 12.83       | -                                     | 0.56 ± 0.03 <sup>A</sup>   | -                          | -                          | 1,5,5-Trimethyl-6-methylene-cyclohexene          | -                               | 121/136                      | 514-95-4   |
| 17  | 13.68       | -                                     | 1.03 ± 0.00 <sup>A</sup>   | -                          | -                          | -                                                | Isopulegol                      | 41/154                       | 89-79-2    |
| 18  | 13.99       | 5.88 ± 0.34 <sup>b</sup>              | 6.84 ± 0.21 <sup>b</sup>   | -                          | 2.55 ± 0.02 <sup>aA</sup>  | 4-Ethyl-2-methoxyphenol                          | 4-Ethylguaicol                  | 137/152                      | 2785-89-9  |
| 19  | 14.32       | -                                     | 1.3 ± 0.04                 | -                          | -                          | 2H-Benzocyclohepten-2-one                        | -                               | 55/180                       | 55103-67-8 |
| 20  | 14.34       | -                                     | -                          | 1.07 ± 0.01                | -                          | -                                                | 4-hydroxy-3-methyl acetophenone | 135/150                      | 876-02-8   |
| 21  | 15.07       | -                                     | -                          | 1.55 ± 0.03 <sup>A</sup>   | -                          | 1,2-dihydro-1,1,6-trimethylnaphthalene           | dehydro-ar-ionene               | 157/172                      | 30364-38-6 |
| 22  | 15.12       | -                                     | -                          | 1.0 ± 0.04 <sup>A</sup>    | -                          | 1H-Inden-1-one, 2,3-dihydro-3,3,4,5-tetramethyl- | -                               | 173/188                      | 56298-98-7 |

|    |       |                            |                            |                          |                            |                                                                          |                                            |         |            |
|----|-------|----------------------------|----------------------------|--------------------------|----------------------------|--------------------------------------------------------------------------|--------------------------------------------|---------|------------|
| 23 | 15.46 | trace                      | -                          | -                        | -                          | 1-(2.6.6-Trimethylcyclohexa-1.3-dien-1-yl)-2-buten-1-one                 | Damascenone                                | 69/190  | 23696-85-7 |
| 24 | 16.36 | 3.96 ± 0.17 <sup>b</sup>   | 2.60 ± 0.07 <sup>a</sup>   | -                        | 5.72 ± 0.11 <sup>cA</sup>  | -                                                                        | Aristolene                                 | 105/204 | 6831-16-9  |
| 25 | 16.78 | 21.27 ± 2.13 <sup>bB</sup> | 8.87 ± 0.04 <sup>a</sup>   | trace                    | 21.91 ± 0.01 <sup>bB</sup> | [S-(E.E)]-1-Methyl-5-methylene-8-(1-methylethyl)-1.6-cyclodecadiene      | Germacrene D                               | 161/204 | 23986-74-5 |
| 26 | 16.94 | trace                      | -                          | -                        | -                          | (1S,4S)-1.4-dimethyl-7-propan-2-ylidene-2.3.4.5.6.8-hexahydro-1H-azulene | beta-Guaiene                               | 161/204 | 88-84-6    |
| 27 | 17.09 | -                          | -                          | 1.5 ± 0.06               | -                          | 4H-1-Benzopyran. 4.4.5.8-tetramethyl-                                    |                                            | 173/188 | 82391-06-8 |
| 28 | 17.94 | 1.67 ± 0.02 <sup>bA</sup>  | 1.05 ± 0.01 <sup>aA</sup>  | -                        | -                          | -                                                                        | 2-nonadecene (Z)                           | 83/266  |            |
| 29 | 18.95 | trace                      | -                          | -                        | -                          | (7S,9aS)-4.4.7.9a-tetramethyl-1.2.3.6.8.9-hexahydro-benzo[7]annulen-7-ol | widdrol                                    | 43/222  | 6892-80-4  |
| 30 | 19.99 | trace                      | 1.43 ± 0.07                | -                        | -                          | -                                                                        | Benzyl benzoate                            | 105/212 | 120-51-4   |
| 31 | 20.15 | 16.45 ± 1.04 <sup>cB</sup> | 11.54 ± 0.46 <sup>b</sup>  | -                        | 8.61 ± 0.03 <sup>a</sup>   | -                                                                        | 1-Tetracosene                              | 55/336  | 10192-32-2 |
| 32 | 20.99 | trace                      | -                          | -                        | -                          | -                                                                        | Butyl cyclohexyl phthalate                 | 149/304 | 84-64-0    |
| 33 | 21.97 | 8.46 ± 0.21 <sup>c</sup>   | 6.19 ± 0.71 <sup>b</sup>   | 3.59 ± 0.01 <sup>a</sup> | 7.66 ± 0.34 <sup>bA</sup>  | (5β.8α.9β.10α.12α)-Atis-16-ene                                           | (-)-Atisirene                              | 257/272 | 20230-48-2 |
| 34 | 21.5  | trace                      | -                          | -                        | -                          | methyl 14-methyl pentadecanoate                                          | pentadecanoic acid                         | 74/270  | 5129-60-2  |
| 35 | 22.41 | trace                      | -                          | -                        | -                          | -                                                                        | manoyl oxide                               | 275/290 | 596-84-9   |
| 36 | 22.92 | 10.37 ± 0.08 <sup>cA</sup> | 9.03 ± 0.214 <sup>cB</sup> | 2.32 ± 0.06 <sup>a</sup> | 4.7 ± 0.01 <sup>bA</sup>   | 5.5.9-trimethyl-14-methylidenetetracyclo(11.2.1.01.10.04.9)hexadecane    | KAUR-16-ENE                                | 257/272 | 562-28-7   |
| 37 | 23.17 | 3.25 ± 0.04 <sup>b</sup>   | 2.28 ± 0.01 <sup>a</sup>   | -                        | -                          | -                                                                        | 7.10.13-Hexadecatrienoic acid methyl ester | 79/264  | 56554-30-4 |

NOTE: trace—below the limit of quantification (LOQ), determined at a signal-to-noise (S/N) ratio of 10:1 (corresponding to a relative area percentage <0.01%); lowercase superscript letters (a, b, c,) represent the statistical significance of the temperature effect within each respective matrix; uppercase letters (A, B) represent significant difference ( $p < 0.05$ ) between the reference matrix (Table 1) and the gelatin-modified matrix (Table 2) at the identical thermal threshold.

**Table 2.** Effect of thermal treatment temperature on the volatile organic compound (VOC) profile in fresh-cut products from large-cranberry (*Vaccinium macrocarpon* Aiton) with the application of a modifier.

| No. | RT<br>[min] | Peak Share in the Chromatogram<br>[%] |                           |                           |                               | Systematic Substance Name                       | Ordinary Compound<br>Name        | <i>m/z</i><br>(100%)/M <sup>+</sup> | No CAS     |
|-----|-------------|---------------------------------------|---------------------------|---------------------------|-------------------------------|-------------------------------------------------|----------------------------------|-------------------------------------|------------|
|     |             | T50                                   | T100                      | T200                      | T0                            |                                                 |                                  |                                     |            |
| 1   | 4.85        | -                                     | -                         | 7.28 ± 0.02 <sup>A</sup>  | -                             | 2-Furaldehyde                                   | Furfural                         | 96/96                               | 98-01-1    |
| 2   | 6.50        | 4.75 ± 0.04 <sup>bB</sup>             | 2.47 ± 0.03 <sup>aA</sup> | -                         | 11.27 ± 0.97<br><sup>cA</sup> | Phenylethylene                                  | Styrene                          | 104/104                             | 100-42-5   |
| 3   | 8.23        | 2.0 ± 0.01 <sup>aA</sup>              | -                         | -                         | 3.94 ± 0.11 <sup>bA</sup>     | -                                               | Benzaldehyde                     | 77/106                              | 100-52-7   |
| 4   | 8.29        | -                                     | -                         | 7.03 ± 0.14               | -                             | 5-methylfuran-2-carbaldehyde                    | 5-methylfurfural                 | 110/110                             | 620-02-0   |
| 5   | 9.65        | -                                     | -                         | 7.35 ± 0.00               | -                             | 4-methyl-1-isopropylbenzene                     | p-Cymene                         | 119/134                             | 99-87-6    |
| 6   | 9.74        | Trace                                 | 2.4 ± 0.01 <sup>a</sup>   | 20.3 ± 0.07 <sup>bB</sup> | -                             | (R)-4-Isopropenyl-1-methyl-1-cyclohexene        | D-Limonene                       | 68/136                              | 138-86-3   |
| 7   | 9.79        | 1.58 ± 0.00 <sup>a</sup>              | 5.84 ± 0.07 <sup>cB</sup> | -                         | 3.91 ± 0.08 <sup>b</sup>      | 1.3.3-Trimethyl-2-oxabicyclo[2.2.2]octane       | Eucalyptol                       | 43/154                              | 470-82-6   |
| 8   | 10.34       | -                                     | -                         | 2.21 ± 0.01 <sup>A</sup>  | -                             | -                                               | Delta-3-carene                   | 93/136                              | 13466-78-9 |
| 9   | 10.9        | -                                     | -                         | 18.28 ± 0.01 <sup>B</sup> | -                             | 1-isopropyl-4-methylcyclohexa-1.3-diene         | α-terpinene                      | 121/136                             | 99-86-5    |
| 10  | 10.9        | -                                     | 7.2 ± 0.04                | -                         | -                             | 1-methyl-4-(propan-2-ylidene)cyclohex-1-ene     | Terpinolene                      | 93/136                              | 586-62-9   |
| 11  | 11.0        | -                                     | -                         | 5.75 ± 0.02 <sup>B</sup>  | -                             | -                                               | methyl benzoate                  | 105/136                             | 93-58-3    |
| 12  | 11.18       | -                                     | -                         | trace                     | -                             | 2-hydroxy-3-methylcyclopent-2-en-1-one          | Cyclotene hydrate                | 112/112                             | 80-71-7    |
| 13  | 12.24       | 5.7 ± 0.04 <sup>A</sup>               | -                         | -                         | -                             | -                                               | 4-Ethylphenol                    | 107/122                             | 123-07-9   |
| 14  | 12.43       | -                                     | trace                     | 9.03 ± 0.11 <sup>bA</sup> | 2.98 ± 0.04 <sup>aA</sup>     | -                                               | Benzoic acid                     | 105/122                             | 65-85-0    |
| 15  | 12.71       | 6.80 ± 0.08 <sup>aA</sup>             | 28.8 ± 0.21 <sup>bB</sup> | -                         | 26.4 ± 0.17 <sup>bA</sup>     | -                                               | α-Terpineol                      | 59/154                              | 98-55-5    |
| 16  | 13.67       | -                                     | 1.3 ± 0.01 <sup>B</sup>   | -                         | -                             | -                                               | Isopulegol                       | 41/154                              | 89-79-2    |
| 17  | 13.89       | -                                     | -                         | 2.82 ± 0.03 <sup>B</sup>  | -                             | 2-methyl-5-[(5-methylfuran-2-yl)methyl]furan    | 2,2'-Methylenebis(5-methylfuran) | 43/176                              | 13679-43-1 |
| 18  | 13.92       | -                                     | -                         | 2.28 ± 0.07               | -                             | a-furancarboxylic acid                          | 2-furoic acid                    | 112/112                             | 88-14-2    |
| 19  | 13.97       | 4.34 ± 0.11 <sup>b</sup>              | -                         | -                         | 2.55 ± 0.02 <sup>aA</sup>     | 4-Ethyl-2-methoxyphenol                         | 4-Ethylguaicol                   | 137/152                             | 2785-89-9  |
| 20  | 14.33       | -                                     | -                         | trace                     | -                             | 2-Isopropyl-5-methylphenol                      | Thymol                           | 135/150                             | 89-83-8    |
| 21  | 15.07       | -                                     | -                         | 11.76 ± 0.09 <sup>B</sup> | -                             | 1.2-dihydro-1.1.6-trimethylnaphthalene          | dehydro-ar-ionene                | 157/172                             | 30364-38-6 |
| 22  | 15.12       | -                                     | -                         | 4.07 ± 0.04 <sup>B</sup>  | -                             | 3.3.5.6-tetramethyl-2.3-dihydro-1H-inden-1-one. |                                  | 173/188                             | 56298-98-7 |
| 23  | 15.58       | trace                                 | -                         | -                         | -                             | -                                               | Octadecane                       | 57/254                              | 593-45-3   |

|    |       |                            |                           |             |                           |                                                                        |                          |         |            |
|----|-------|----------------------------|---------------------------|-------------|---------------------------|------------------------------------------------------------------------|--------------------------|---------|------------|
| 24 | 16.36 | 5.40 ± 0.31 <sup>aA</sup>  | -                         | -           | 5.72 ± 0.11 <sup>aA</sup> | -                                                                      | Aristolene               | 105/204 | 6831-16-9  |
| 25 | 16.67 | 8.21 <sup>b</sup>          | 2.20 <sup>a</sup>         | -           | -                         | -                                                                      | Ethyl 9-tetradecenoate   | 55/254  | 68862-25-9 |
| 26 | 16.78 | 27.67 ± 0.03 <sup>cB</sup> | 8.60 ± 0.18 <sup>aB</sup> | -           | 21.91 <sup>b</sup>        | [S-(E.E)]-1-Methyl-5-methylene-8-(1-methylethyl)-1,6-cyclodecadiene    | Germacrene D             | 161/204 | 23986-74-5 |
| 27 | 17.09 | -                          | -                         | trace       | -                         | -                                                                      | 1,5,8-trimethyltetraline | 159/174 | 21693-51-6 |
| 28 | 17.94 | 1.95 ± 0.05 <sup>A</sup>   | -                         | -           | -                         | -                                                                      | 1-hexadecanol            | 55/254  | 36653-82-4 |
| 29 | 19.99 | trace                      | -                         | -           | -                         | -                                                                      | Benzyl benzoate          | 105/212 | 120-51-4   |
| 30 | 20.15 | 19.89 ± 0.46 <sup>cB</sup> | 6.80 ± 0.19 <sup>aA</sup> | -           | 8.61 ± 0.03 <sup>bA</sup> | -                                                                      | 1-Tetracosene            | 55/336  | 10192-32-2 |
| 31 | 21.23 | trace                      | -                         | -           | -                         | -                                                                      | Hentriacontane           | 57/436  | 630-04-6   |
| 32 | 21.49 | -                          | 3.15 ± 0.00               | -           | -                         | -                                                                      | methyl palmitate         | 74/270  | 112-39-0   |
| 33 | 21.97 | 5.74 ± 0.02 <sup>a</sup>   | -                         | trace       | 7.66 <sup>bA</sup>        | (5β,8α,9β,10α,12α)-Atis-16-ene                                         | (-)-Atisirene            | 257/272 | 20230-48-2 |
| 34 | 22.91 | 5.92 ± 0.09 <sup>c</sup>   | 2.81 ± 0.01 <sup>a</sup>  | trace       | 4.70 <sup>b</sup>         | 5,5,9-trimethyl-14-methylidenetetra-cyclo(11.2.1.01.10.04.9)hexadecane | Kaur-16-ene              | 257/272 | 562-28-7   |
| 35 | 23.11 | -                          | 3.36 ± 0.17               | -           | -                         | methyl 9-cis,12-cis-octadecadienoate                                   | methyl linoleate         | 67/294  | 112-63-0   |
| 36 | 23.17 | -                          | 8.11 ± 0.09               | -           | -                         | 9-octadecenoic acid (Z)-, methyl ester                                 | methyl oleate            | 55/296  | 112-62-9   |
| 37 | 28.95 | -                          | -                         | 1.77 ± 0.03 | -                         | 2,6,10,15,19,23-heksametylo-2,6,10,14,18,22-tetrakosaheksaen           | Squalene                 | 69/410  | 111-02-4   |

NOTE: trace—below the limit of quantification (LOQ), determined at a signal-to-noise (S/N) ratio of 10:1 (corresponding to a relative area percentage <0.01%); overcase superscript letters (a, b, c) represent the statistical significance of the temperature effect within each respective matrix; uppercase letters (A, B) represent significant difference ( $p < 0.05$ ) between the reference matrix (Table 1) and the gelatin-modified matrix (Table 2) at the identical thermal threshold.

### 3. Results and Discussion

#### 3.1. Characterisation of the Volatile Organic Compound (VOC) Profile of the Thermally Intact Fresh-Cut Matrix

Headspace analysis of freshly fragmented large-cranberry fruit (variant T0 (control) (Series I)) revealed a dominant proportion of substances from the terpenoid group, with a particular emphasis on  $\alpha$ -terpineol (26.40%) and germacrene D (21.91%) (Table 1). This profile exhibits a high correlation with literature reports regarding the volatile metabolites of the genus *Vaccinium*, where monoterpenes and sesquiterpenes are indicated as the main determinants of the native, resinous, and fruity organoleptic attributes of the raw material [1,14]. The presence of eucalyptol (3.91%) and benzaldehyde (3.94%) in the control sample defines the baseline volatile composition of the unprocessed raw material, which has also been confirmed by other authors dealing with the chemotaxonomy of berry fruits [3]. The high relative proportion of styrene (11.27%) in the thermally intact reference matrix can be interpreted as a direct physiological tissue response to the mechanical stress induced by the preparation of the fresh-cut product. The literature extensively describes the phenomenon of the activation of the phenylpropanoid pathway and the enzymatic decarboxylation of cinnamic acid to styrene in plant cells whose structural integrity has been disrupted as a result of tissue fragmentation [4]. From a biological perspective, the volatile profile of the fresh raw material is characterised by high lability, which depends on multiple factors. The mechanical damage to cellular barriers during fresh-cut preparation significantly increases the mass transfer surface area, which facilitates the desorption of native VOCs and induces direct contact between intracellular enzymes (e.g., lipoxygenases) and lipid substrates, serving as a starting point for further transformations under the influence of exogenous factors [5].

#### 3.2. Effect of Moderate Thermal Treatment (50 °C and 100 °C) on the Rearrangement of the Odour Profile—A Comparative Study of the Matrices

The implementation of moderate thermal treatment in the 50 °C and 100 °C variants revealed diametric differences in the release kinetics and stability of VOCs between the reference matrix (Table 1) and the protein-modified matrix (Table 2). The most spectacular protective effect of the applied post-harvest technology was recorded for germacrene D. In the reference matrix subjected to a temperature of 50 °C, this compound remained a significant constituent of the volatile profile, reaching 21.27% of the total volatile fraction (Table 1). However, in the presence of gelatin (Variant T50, Table 2), the relative proportion of germacrene D was further elevated, reaching a notably higher level of 27.67%. A similar relationship was observed at 100 °C, where the proportion of  $\alpha$ -terpineol in the modified matrix was as high as 28.30%, whereas in the sample without the protein addition, it was reduced to 14.95%. This phenomenon provides direct evidence for the immobilisation of hydrophobic monoterpene and sesquiterpene molecules within the gelatin biopolymer network structure. According to the macromolecular theories described by Guichard [13], proteins exhibit the ability to bind volatile organic ligands via van der Waals interactions and hydrophobic forces within their structural loops. As a result, the escape of these key volatile compounds from the headspace of the fresh-cut product is inhibited, which allows for the enhanced retention of the aroma profile despite the impact of the thermal factor.

However, it is worth noting that in the gelatin-modified matrix, the proportion of 4-ethylphenol at 100 °C was completely eliminated, and the proportion of 4-ethylguaiacol decreased from 6.84% (pure matrix) to 4.14%. This suggests that the functional groups of the protein may partially restrict the non-enzymatic breakdown of phenolic acids or bind

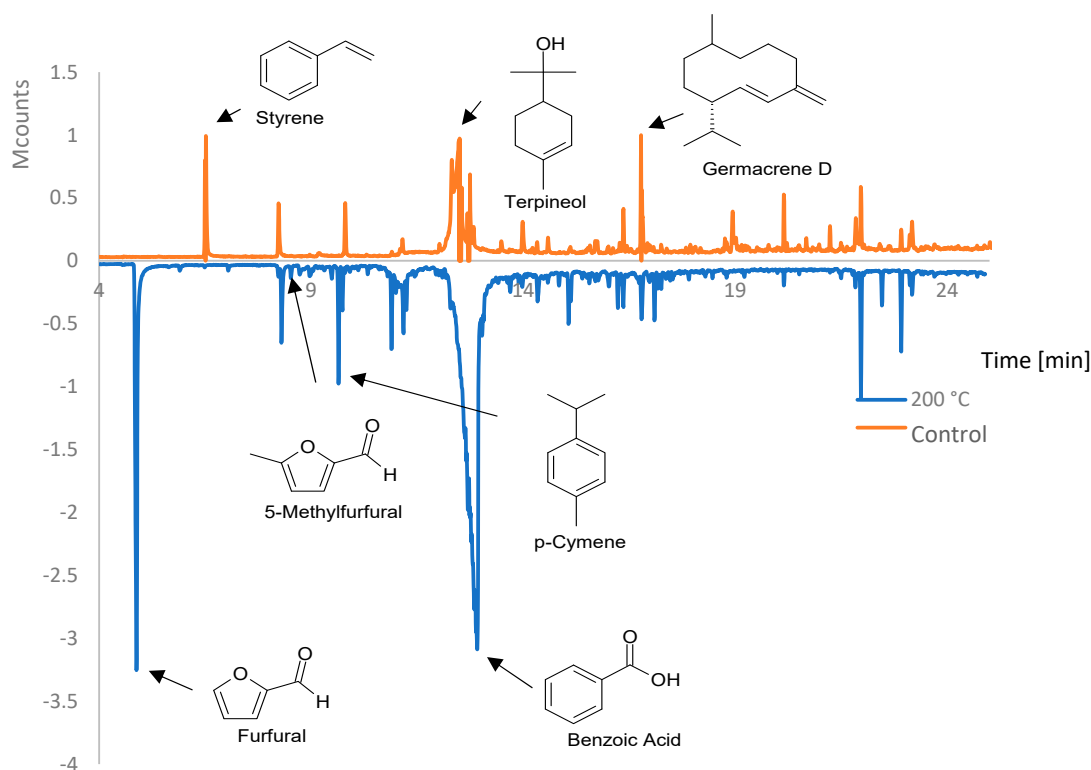
the resulting ring compounds, thereby limiting the accumulation of these undesirable volatile components [14,15].

### 3.3. High-Temperature Thermolysis (200 °C)—Protein-Carbohydrate Interactions and Inhibition of Quality Destruction

The process conducted at 200 °C led to the complete elimination of native monoterpenes and sesquiterpenes, which signifies the degradation of the fraction responsible for the native sensory attributes of the cranberry. In the headspace of the reference matrix, benzoic acid became the dominant component, reaching a relative proportion of 53.34% (compared to 2.98% in the control sample). Large-cranberry is a raw material naturally characterised by a high content of benzoic acid, occurring both in its free state and as ester-bound forms [16]. Extreme thermal stress (200 °C) induces processes of rapid ester bond thermolysis and the sublimation of benzoic acid from cell wall structures. A similar phenomenon of intense benzoic acid release under conditions of rigorous heat treatment of berry products has been reported in studies concerning the physicochemical stability analysis of fruit preserves [17].

A key distinguishing feature of the 200 °C variant in the pure sample was also the intensive *de novo* synthesis of products of advanced non-enzymatic browning (the Maillard reaction) and caramelisation, represented by furfural (2-furaldehyd) with a proportion of 23.95% and 5-methylfurfural (2.80%). These compounds were not identified at lower temperatures or in the reference sample T0. Their presence in the headspace is a classic indicator of the thermal degradation of pentoses, dehydration of ascorbic acid, and cyclisation of simple sugar breakdown products occurring in the acidic environment of cranberry juice [12,13]. Furfural and its derivatives are documented in the literature to introduce empyreumatic (caramel and burnt) volatile notes to the matrix, which, consequently, can impair the overall aroma profile quality of products that are expected to retain fresh characteristics.

Visual confirmation of the described thermal destruction of the unmodified fruit matrix is provided by the comparison of the chromatographic profile of the control sample and the variant subjected to 200 °C (Figure 1). In the high-temperature profile, a drastic disappearance of signals corresponding to native terpenoids is visible, accompanied by the simultaneous appearance of dominant, intense peaks with a high peak area, identified as benzoic acid and furfural.



**Figure 1.** Comparative alignment of GC-MS chromatograms of the control sample (T0) and the large-cranberry sample after thermal treatment at 200 °C (T200) without the addition of a protein modifier (Series I).

It is crucial to acknowledge that the data presented herein represent relative peak area percentages, a methodology that can be subject to mathematical shifts such as passive percentage inflation due to the loss of highly volatile fractions (e.g., primary terpenes in the control matrix) or dilution effects from the emergence of new thermal by-products. However, a comparative audit of the raw chromatographic abundances confirms a true suppression of furfural in the presence of the protein modifier. To fully elucidate this phenomenon, it is necessary to distinguish the temperature-dependent hierarchy between physical entrapment, hydrophobic interactions, and chemical covalent pathways. At moderate thermal thresholds (50 °C and 100 °C), the retention of volatile organic compounds is primarily driven by physical embedding and non-covalent hydrophobic forces within the native macromolecular loops of the hydrated gelatin hydrogel. However, at the extreme threshold of 200 °C, the biopolymer network undergoes extensive thermal denaturation and partial pyrolytic degradation, rendering physical entrapment secondary. Under these conditions, the dramatic suppression of furfural is governed predominantly by chemical kinetics. In the absence of direct structural or macromolecular evidence, such as Fourier-transform infrared spectroscopy (FTIR) or size-exclusion chromatography (SEC), to confirm the synthesis of complex, non-volatile melanoidins, this phenomenon is most accurately attributed to competitive substrate utilisation. The abundance of nucleophilic  $\epsilon$ -amino groups ( $\epsilon$ -NH<sub>2</sub>) from lysine side chains in the gelatin matrix actively engages in rapid Maillard cascade reactions with native reducing sugars. By aggressively consuming these carbohydrate precursors, the protein modifier competitively suppresses the alternative acidic dehydration and caramelization pathways that would otherwise lead to intensive furfural accumulation in the control matrix.

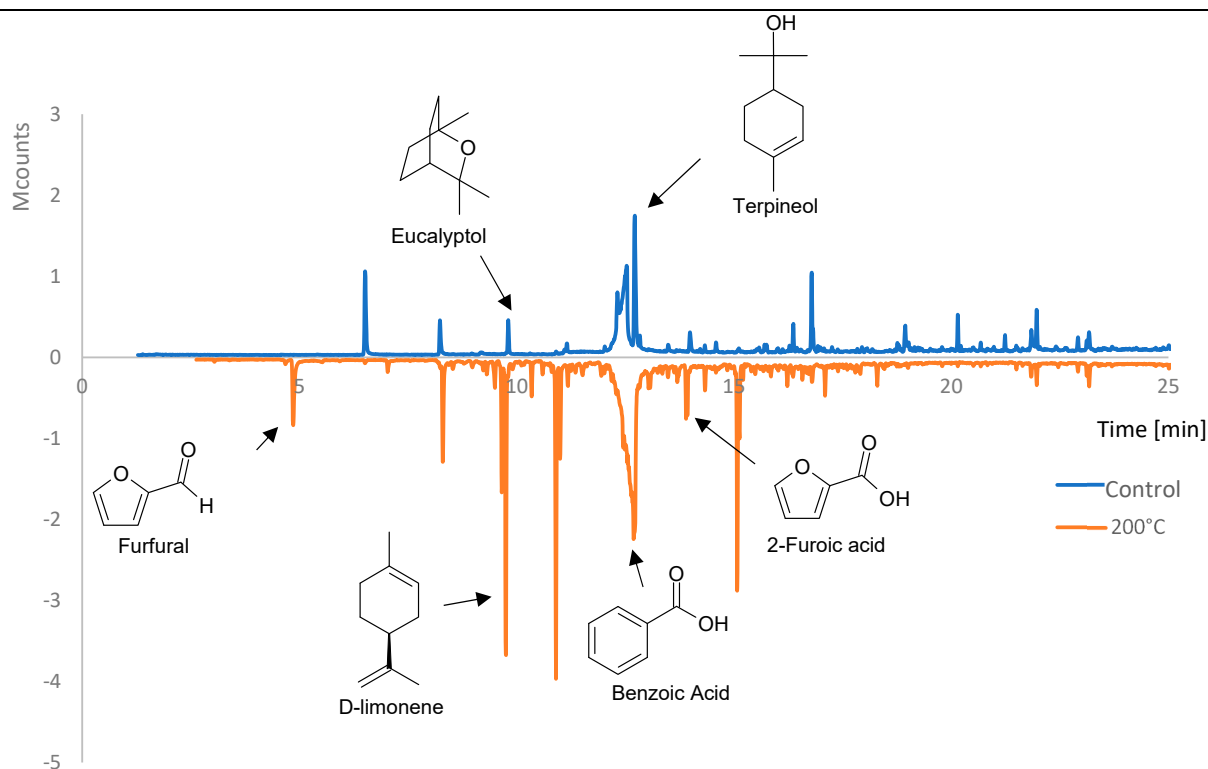
The implementation of the protein modification technology (Table 2, Variant T200) led to a drastic remodelling of these chemical transformation pathways, which is illus-

trated by the comparison of the chromatograms of the control sample with the gelatin-modified variant (Figure 2). In the presence of the biopolymer, the following were recorded:

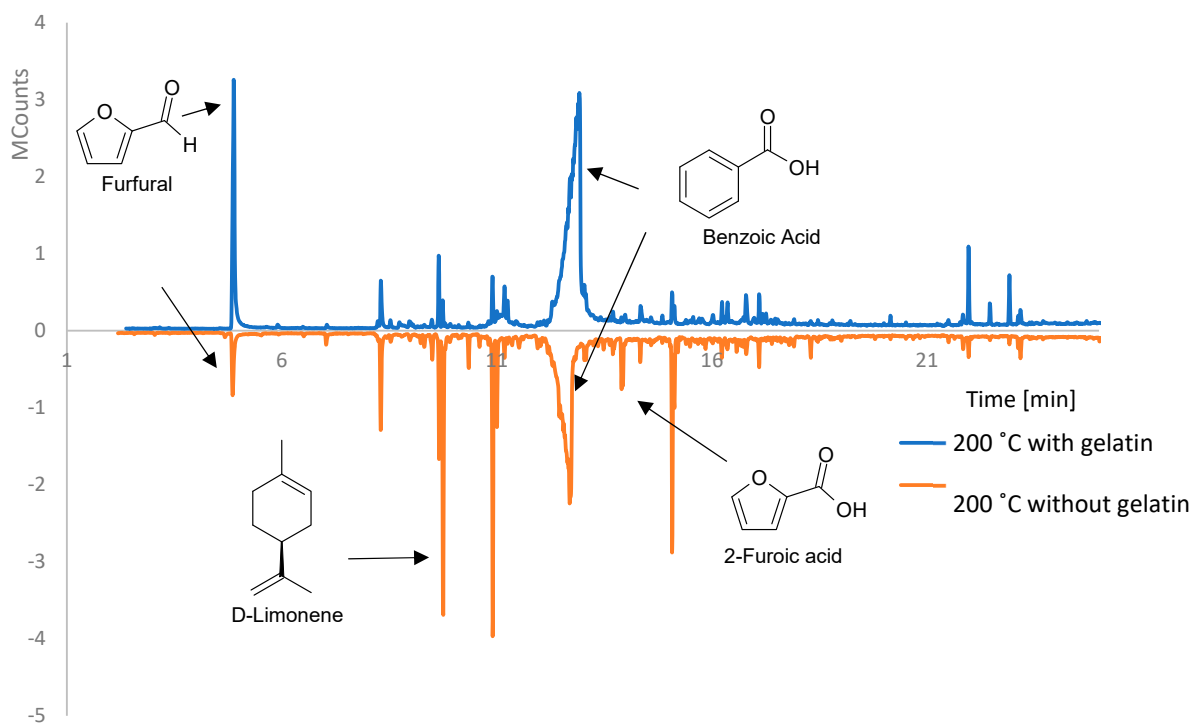
- Inhibition of benzoic acid release: The proportion of benzoic acid in the headspace dropped from 53.34% to a mere 9.03%. This indicates a strong chemical affinity of the amino and carboxylic groups of gelatin towards free benzoic acid, which blocked its rapid sublimation from the cellular structures of the fresh-cut tissue [18].
- Reduction in furfural emission: The proportion of free furfural was reduced from 23.95% to 7.28%. This decrease, combined with the simultaneous appearance of new furan derivatives (e.g., 2,2'-methylenebis(5-methylfuran) at a level of 2.82% and 2-furoic acid—2.28%), provides classic evidence for the involvement of free amino groups of gelatin in the advanced stages of the Maillard reaction [19,20]. Instead of the free evaporation of furfural into the gas phase, it underwent condensation and addition reactions with the amino acids of gelatin, leading to the formation of non-volatile macromolecular melanoidins.

Additionally, in the T200 variant with gelatin, the unique presence of D-limonene at a very high level (20.30%) and  $\alpha$ -terpinene (18.28%) was recorded, which did not occur in the pure matrix at this temperature. This proves that the presence of the protein matrix drastically alters the thermolysis conditions within the microenvironment of the fresh-cut product, protecting monoterpene rings from complete breakdown and enabling their isomerisation, which leads to a unique volatile profile with potentially desirable citrus-spicy sensory attributes [21,22].

Direct and most crucial evidence for the stabilising efficiency of the implemented post-harvest technology is provided by the direct overlay of the chromatographic profiles of both investigated series at 200 °C (Figure 3). The graphical comparison clearly illustrates the effect of “quenching” (attenuating) the intensity of the benzoic acid and furfural signals in the sample containing gelatin, while simultaneously exposing the retention bands characteristic of the protected terpene components (D-limonene,  $\alpha$ -terpinene). This confirms the macromolecular nature of the interactions between the protein matrix and VOCs, predestining this system for the role of an effective preserving agent for the volatile profile of minimally processed cranberry fruit.



**Figure 2.** Comparative alignment of GC-MS chromatograms of the control sample (T0) and the large-cranberry sample after thermal treatment at 200 °C with the addition of a protein modifier (Series II).



**Figure 3.** Comparative alignment of GC-MS chromatograms of the large-cranberry sample after thermal treatment at 200 °C without the addition of a modifier (Series I) and with the addition of a protein modifier (Series II).

#### 4. Conclusions

The conducted research on the volatile organic compound (VOC) profile of large-cranberry fruit contributes to the development of modern post-harvest technologies aimed at the stabilisation and quality enhancement of minimally processed products. It was demonstrated that tissue fragmentation, characteristic of fresh-cut technology, drastically increases the thermolability of native odour markers. In the reference matrix, heat treatment (50–200 °C) led to a complete loss of valuable terpene fractions and the generation of volatile substances with a smoky and empyreumatic character (benzoic acid up to 53.34%, furfural up to 23.95%), which drastically reduces the baseline aromatic quality of the raw material. The application of an innovative treatment, based on the modification of the fruit matrix with an edible protein biopolymer (gelatin), allowed for the effective prevention of these unfavourable changes. GC-MS analysis confirmed that the introduction of the protein matrix acts as a physicochemical barrier for valuable volatile components, retaining germacrene D and  $\alpha$ -terpineol at moderate temperatures, and drastically inhibiting the emission of furfural and benzoic acid at 200 °C. Mechanistically, this thermal mitigation is driven by temperature-dependent pathways, transitioning from physical entrapment at lower temperatures to competitive chemical kinetics that consume carbohydrate precursors under extreme thermal stress. Ultimately, the controlled modification of the food matrix demonstrates a significant protective potential against thermally induced aromatization, primarily by altering the reaction kinetics and partitioning behaviour of volatiles, although absolute quantification is required for industrial validation.

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