agronomy

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Impact of Preharvest Bagging on the Volatile Profile of Vinalopó Table Grapes

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Abstract: The bagging technique is a traditional preharvest practice used in Vinalopó Bagged Table Grape production to improve fruit quality and protect clusters from environmental stress. However, its influence on grape volatile composition remains underexplored. This study analyzed the volatile profile of three grape varieties ('Dominga', 'Aledo', and 'Doña María') by comparing bagged and non-bagged clusters to assess the effect of bagging on aromatic compounds. Volatiles were extracted using headspace solid-phase microextraction (HS-SPME) and analyzed by gas chromatography–mass spectrometry (GC–MS). A total of 35 volatile compounds were identified and quantified, mainly aldehydes, terpenes, and alcohols. The highest concentration was found in non-bagged 'Dominga' grapes (57.17 mg kg^{-1}), and the lowest in bagged 'Doña María' grapes (16.36 mg kg^{-1}). Although total volatile content did not differ significantly between treatments, differences were observed in the relative abundance of chemical families. Bagged grapes showed higher proportions of aldehydes, such as hexanal and (E)-2-hexenal, contributing to green, fresh aromas, while non-bagged grapes exhibited more alcohols and esters, linked to fruity and overripe notes. This study offers new insights into the role of preharvest bagging in shaping grape volatile composition, contributing to a better understanding of its impact on fruit aroma and quality.

Keywords: table grape; preharvest bagging; volatile compounds; aroma profile; GC-MS analysis

Academic Editors: Dominika Średnicka-Tober, Renata Kazimierczak and Jackline Freitas Brilhante De Sao Jose

Received: 2 April 2025
Revised: 21 April 2025
Accepted: 26 April 2025
Published: 27 April 2025

Citation: Andreu-Coll, L.; Noguera-Artiaga, L.; Sendra, E.; Hernández, F. Impact of Preharvest Bagging on the Volatile Profile of Vinalopó Table Grapes. *Agronomy* **2025**, *15*, 1066. https://doi.org/10.3390/agronomy15051066

1. Introduction

Vinalopó Bagged Table Grapes, originating from the Vinalopó Valley in Alicante, Spain, are renowned for their exceptional quality and distinctive cultivation method, in which each bunch is individually enclosed in a paper bag during ripening. This product holds Protected Designation of Origin (PDO) status and includes several table grape varieties. The bagging technique, introduced in the early 20th century in Novelda, serves multiples purposes: it protects the grapes from pests, diseases, and environmental factors, while also slowing the ripening process, enhancing the fruit's organoleptic properties. According to the PDO specifications, grape clusters must remain bagged for at least 60 days before harvest, ensuring both protection and improved fruit quality. This traditional practice highlights the regional significance and meticulous agricultural methods that define the PDO [1].

The sensory attributes of Vinalopó grapes are largely influenced by their volatile compound composition. Volatile compounds, including alcohols, aldehydes, terpenes, ketones, and esters, play a pivotal role in defining the aroma and taste of fruits [2]. In grapes, these compounds are synthesized through metabolic pathways that can be affected by various factors such as cultivar type, climatic conditions, and agricultural practices. The bagging technique, by modifying the microenvironment around each cluster, can influence the biosynthesis and accumulation of these volatile compounds, thereby impacting the grape sensory qualities [3].

Preharvest bagging is a widely applied agronomic practice in fruit production, primarily used to improve external appearance and to protect against biotic and abiotic stressors. Beyond its protective role, bagging has been shown by several studies to influence important fruit quality traits such as soluble solids content, titratable acidity, firmness, and the accumulation of phenolic compounds. Moreover, it may affect the synthesis and accumulation of volatile compounds, thus modifying the sensory profile of the fruit [2,4–8]. While these effects have been investigated in various fruit crops, no studies to date have evaluated the impact of bagging on the volatile composition of PDO-certified Vinalopó table grape varieties, indicating the need to explore this aspect within the specific context of this traditional cultivation system.

The aim of this study was to determine the volatile composition of the berries from the grape varieties 'Dominga', 'Aledo', and 'Doña María', both bagged and non-bagged, to assess the differences in the volatile compound profile associated with the bagging technique. Understanding these differences could provide valuable insights into the impact of preharvest bagging on the aromatic quality and sensory characteristics of table grapes. This knowledge may contribute to optimizing grape cultivation practices, enhancing fruit quality and consumer perception, and offering a product with improved sensory attributes while maintaining the traditional techniques that define the Vinalopó Bagged Table Grape.

2. Materials and Methods

2.1. Plant Material and Sample Processing

Berries from the grape varieties 'Dominga', 'Aledo', and 'Doña María' were used for this study. 'Dominga' is an early- to mid-season variety (October–November) with large clusters of uniform, elongated, straw-yellow berries, featuring firm, non-pigmented pulp and a neutral flavor. 'Aledo' is a late-ripening variety (November–December) with large, loose clusters and ellipsoidal, waxy yellow berries with thick, crisp skin. 'Doña María' is a mid-season variety (September–October) producing large, conical, and loose clusters with very large, pale-yellow berries that have a thin skin and firm, juicy pulp with a characteristic honey-like aroma [1].

The grapevines of the 'Dominga', 'Aledo' and 'Doña María' varieties are cultivated in private farms in Novelda, Alicante, Spain. The climate of the area is strictly Mediterranean, with mild winters, low annual rainfall, and hot and dry summers. A bagging treatment was applied to grape clusters using white paper bags in August 2022, and these fruits were compared with those from control farms located in the same region, where no bagging treatment was implemented.

The sampling process was carried out in two stages: field and laboratory. In the field, grape clusters were harvested during the second week of October ('Doña María' variety) and the last week of November 2022 ('Dominga' and 'Aledo' varieties). For each variety and treatment (bagged and unbagged), three replicates were conducted, with 10 grape clusters collected per replicate, resulting in a total of 30 grape clusters per variety. Clusters were manually harvested at commercial ripening according to standard harvesting criteria for table grapes, based on visual indicators (such as color uniformity and berry size).

Soluble solids content (°Brix) and titratable acidity (g tartaric acid L^{-1}) were measured, and the maturity index (MI) was calculated as the ratio between soluble solids and acidity. These parameters are presented in Table 1. The sampling was distributed across different orientations and canopy heights to account for variability within the vineyard. All samples were immediately transported to the laboratory under controlled conditions, including careful handling to prevent mechanical damage, use of ventilated containers to reduce humidity buildup, and minimization of transport time (within two hours of harvest) to limit metabolic alterations.

Table 1. Total soluble solids (TSS), titratable acidity, and maturity index (MI) of bagged and unbagged 'Dominga', 'Aledo', and 'Doña María' grapes at harvest.

Variety × Treatment	SST (°Brix)	Titratable Acidity (g Tartaric Acid L^{-1})	MI
Bagged 'Dominga'	17.1 f [2]	2.55 d	67.0 a
Unbagged 'Dominga'	22.0 c	3.50 bc	63.0 b
Bagged 'Aledo'	19.0 e	3.34 c	57.0 c
Unbagged 'Aledo'	19.9 d	3.80 b	52.5 d
Bagged 'Doña María'	23.2 b	3.29 a	70.5 a
Unbagged 'Doña María'	25.4 a	4.58 a	55.6 cd
ANOVA	*** [1]	***	***

[1] ***, significant at $p < 0.001$. [2] Values (mean of three replications) followed by the same letter, within the same column, were not statistically different according to Tukey's multiple range test.

In the laboratory, 30 grape clusters per variety and treatment were carefully inspected. From each cluster, approximately 10 berries were manually selected from different positions (top, middle, bottom) to ensure representativeness. Only intact and defect-free berries were chosen, discarding those with visible signs of dehydration, mechanical damage, or fungal infection. The selected berries were then randomly divided into three biological replicates per variety and treatment.

2.2. Extraction Procedure of Volatile Aroma Compounds and Chromatographic Analyses

Two grams of fresh grapes were added to a hermetic vial with a polypropylene cap and PTFE (polytetrafluoroethylene)/silicone septa, along with 1 g NaCl and β-ionone as the internal standard (10 µL of 1000 mg L^{-1} solution in ethanol). The extraction of the volatile compounds of the samples was carried out using the headspace solid-phase microextraction (HS-SPME) method, as described by Teruel-Andreu et al. [9]. A fiber of 50/30 mm DVB/CAR/PDMS (divinylbenzene/carboxen/polydimethylsiloxane) 1 cm in length was used to absorb the compounds during the extraction. Samples were exposed for 60 min at 40 °C, with constant agitation (500 rpm) by using a Shimadzu AOC-6000 Plus autosampler (Shimadzu Corporation, Kyoto, Japan). Volatile compounds were determined following the procedure described by Oliveira et al. [10] using a chromatograph Shimadzu GC2030 (Shimadzu Scientific Instruments, Inc., Columbia, MD, USA) for the isolation and identification of volatile compounds. The gas chromatograph was equipped with an SLB-5 MS column of 30 m × 0.25 mm × 0.25 µm (length, diameter, and film thickness, respectively) (Teknokroma, Barcelona, Spain). For the identification of compounds, the chromatograph was coupled with a Shimadzu TQ8040 NX mass spectrometer detector. The equipment and the gas chromatographic conditions were described in Teruel-Andreu et al. [9]. The volatile compounds were identified using three methods: (i) retention indices, which were calculated using a commercial alkane standard mixture (C8–24) (Sigma-Aldrich, Steinheim, Germany); (ii) retention times of the chemically pure compounds analyzed by gas chromatography–mass spectrometry (GC–MS); and (iii) comparison of the compound mass spectra with those in reference databases [11]. In addition, the relative intensity of each volatile compound was calculated as the ratio between the area of the specific molecule and the sum of the areas of all identified peaks (peak area normalization method) in the chromatogram. Compounds with a spectral similarity > 90% and with a deviation of less than 10 units of linear retention similarity were considered as correctly identified. Analyses were performed using three biological replicates per sample, and results are expressed as mean values in mg kg^{-1}.

2.3. Statistical Analysis

One-way analysis of variance (ANOVA) was used to assess the effect of grape variety on the concentration of volatile compounds. A two-way ANOVA was also performed to evaluate the effects of variety, bagging treatment (bagged vs. unbagged), and their combined influence. In both cases, Tukey's test was employed as the multiple range procedure to discriminate among means. Additionally, a Student's *t*-test was applied to assess the overall effect of bagging on volatile composition. Statistical significance was set at $p \leq 0.05$. All statistical analyses were performed using XLSTAT software version 9 [12]. Significantly different samples were labeled with different letters to facilitate interpretation of the results. The relative abundance of each chemical family according to variety and treatment was illustrated in the generated plots (Figures 1 and 2) using SigmaPlot 12.5. [13]. Furthermore, principal component analysis (PCA; Figure 3) was conducted using XLSTAT software version 9 [12].

3. Results and Discussion

To provide context for the interpretation of the volatile profiles, basic physicochemical parameters were measured at harvest. The basic physicochemical parameters—TSS, titratable acidity, and maturity index (MI)—are shown for each sample (Table 1). Notable differences were observed among the grape samples, with unbagged 'Doña María' showing the highest TSS and acidity and bagged 'Dominga' showing the lowest. The maturity index ranged from 52.46 to 70.49, reflecting variation in ripening status across varieties and treatments, which may contribute to differences in volatile compound accumulation.

The volatile compounds were determined using the HS-SPME standard method combined with GC–MS for the isolation, identification and their relative abundance determination. A total of 35 compounds were isolated, identified, and quantified across the three studied Vinalopó table grape varieties: 'Dominga', 'Aledo', and 'Doña María'. The identified compounds and their sensory descriptors are summarized (Table 1) according to FEMA [14], the SAFC Flavors and Fragrances Catalog [15], and the relevant literature [16].

The volatile compounds that were isolated can be grouped into seven chemical families.

- Aldehydes (n = 14): Hexanal, (E)-2-hexenal, benzaldehyde, octanal, benzeneacetaldehyde, nonanal, (E)-2-nonenal, decanal, β-citral, α-citral, undecanal, dodecanal, tetradecanal (2 isomers).
- Terpenes (n = 7): o-Cymene, D-limonene, γ -terpinene, terpinolene, caryophyllene, α-bergamotene, β-bisabolene.
- Alcohols (n = 6): 1-hexanol, fenchol, 1-terpinenol, β-terpineol, terpinen-4-ol, L-α-terpineol.
- Esters (n = 4): acetic acid hexyl ester, octanoic acid methyl ester, citronellol acetate, methyl laurate.
- Ketones (n = 2): 6-methyl-5-hepten-2-one, 2,6-di-tert-butyl-p-benzoquinone.
- Terpenoids (n = 1): linalool.
- Carboxilic acids (n = 1): hexanoic acid.

The volatile compounds shown in Table 2 are common to all varieties and treatments studied in this work. The volatile profile of 'Doña María', 'Dominga' and 'Aledo' bagged and unbagged grapes included many aldehydes, followed by terpenes and alcohols. This finding is aligned with those of other authors, who determined that the most abundant volatile compounds in table grapes include C6 compounds, terpenes, and alcohols [17]. By contrast, Li et al. [3] determined that esters and terpenes were the groups with greater contribution ratios to the total aroma compound content. These differences in volatile composition may be attributed to variations in grape variety, environmental factors such as climate and soil composition, and differences in vineyard management practices, for example, irrigation frequency or fertilization regimes. Additionally, genetic factors, ripening stage at harvest, and postharvest conditions, such as storage temperature and humidity control, may further contribute to the distinct volatile profiles observed across studies.

Table 2. Aromatic compounds found in bagged and unbagged 'Dominga', 'Doña María' and 'Aledo' grapes, analyzed by headspace solid-phase microextraction (HS-SPME).

Code	Volatile Compound	Chemical Family	RT [1] (min)	Kovat Index (KI) [2]		Sensory Descriptors
				Exp.	Lit.	
V1	Hexanal	Aldehydes	4.69	823	819	Apple, fatty, green, fresh [3,4]
V2	(E)-2-Hexenal	Aldehydes	6.13	862	855	Almond, apple, fruity, vegetable [4]
V3	1-Hexanol	Alcohols	6.49	870	870	Green, woody, sweet [4]
V4	Benzaldehyde	Aldehydes	10.6	953	955	Almond, cherry, sweet [4]
V5	Hexanoic acid	Carboxilic acids	11.41	969	977	Cheesy, fatty, sour, pungent [3,4]
V6	6-methyl-5-Hepten-2-one	Ketones	11.84	978	986	Citrus, mushroom, oily, green [3,4]
V7	Octanal	Aldehydes	12.86	998	1001	Fat, green, oil, pungent, fruity [3,4]
V8	Acetic acid hexyl ester	Esters	13.40	1007	1010	Floral, green, apple, cherry [3,4]
V9	o-Cymene	Terpenes	14.07	1018	1018	Citrus, fresh, solvent [3,4]
V10	D-Limonene	Terpenes	14.33	1022	1028	Citrus, mint [3]
V11	Benzeneacetaldehyde	Aldehydes	15.06	1033	1043	Cocoa, coffee, wine-line [3,4]
V12	γ-Terpinene	Terpenes	16.09	1050	1055	Bitter, citrus, herbaceous [3,4]
V13	Terpinolene	Terpenes	17.78	1077	1084	Pine [3,4]
V14	Linalool	Terpenoids	18.78	1092	1098	Floral, citrus, sweet [3,4]
V15	Nonanal	Aldehydes	19.09	1097	1101	Fat, grape, floral, citrus, melon [3,4]
V16	Fenchol	Alcohols	19.86	1109	1110	Camphor, lemon [3]
V17	Octanoic acid methyl ester	Esters	20.38	1116	1120	Oily, cheese [4]
V18	1-Terpinenol	Alcohols	21.05	1126	1120	Grapefruit, anise, citrus, fruity [3,4]
V19	β-Terpineol	Alcohols	21.90	1139	1144	Anise, fresh, mint, oil, lilac [3,4]
V20	(E)-2-Nonenal	Aldehydes	22.71	1151	1156	Paper, waxy, fatty [3,4]
V21	Terpinen-4-ol	Alcohols	23.95	1169	1162	Grapefruit, citrus, anise, fresh [3,4]
V22	L-α-Terpineol	Alcohols	24.94	1183	1192	Anise, fresh, mint, oil, lilac [3,4]
V23	Decanal	Aldehydes	25.85	1197	1203	Floral, green, apple, cherry, [3,4]
V24	β-Citral	Aldehydes	27.81	1229	1238	Lemon [3,4]
V25	α-Citral	Aldehydes	29.64	1259	1267	Lemon [3,4]
V26	Undecanal	Aldehydes	31.97	1297	1306	Floral, orange, fatty, rose [3,4]
V27	Citronellol acetate	Esters	34.21	1341	1348	Floral, geranium, rose [3,4]
V28	Dodecanal	Aldehydes	37.16	1399	1409	Green, waxy, floral, sweet [3,4]
V29	Caryophyllene	Terpenes	37.46	1406	1414	Fried, spicy, woody [3,4]

Table 2. *Cont.*

Code	Volatile Compound	Chemical Family	RT [1] (min)	Kovat Index (KI) [2] Exp.	Lit.	Sensory Descriptors
V30	α-Bergamotene	Terpenes	38.21	1422	1414	Fruity, sweet [3,4]
V31	2,6-di-tert-butyl-p-Benzoquinone	Ketones	39.38	1448	1458	Fennel, fatty [4]
V32	β-Bisabolene	Terpenes	41.58	1498	1506	Floral [3]
V33	Tetradecanal	Aldehydes	41.76	1502	1503	Honey, hay [5]
V34	Methyl laurate	Esters	42.34	1516	1524	Coconut, creamy, fatty, soapy [3,4]
V35	Tetradecanal	Aldehydes	45.98	1605	1615	Honey, hay [5]

[1] RT: Retention time. [2] KI: KI (Exp.) = experimental Kovats index; (Lit.) = literature Kovats index. [3] FEMA (2024) [14]. [4] SAFC (2011) [15]. [5] Schirack et al. (2006) [16].

The concentration of each volatile compound in μg 100 g^{-1} and their total content in mg kg^{-1} (Tables 3–5) were used to compare differences among samples. The standard deviation remained below 20% in all cases. Statistical significance is indicated, where relevant, in Tables 4 and 5 using the following notation: not significant, (NS, $p > 0.05$), * ($p < 0.05$), ** ($p < 0.01$), and *** ($p < 0.001$). The samples which presented the highest concentration of volatile compounds were unbagged 'Dominga', followed by unbagged 'Aledo', whose values are 57.17 mg kg^{-1} and 43.30 mg kg^{-1}, respectively. By contrast, the unbagged 'Doña María' variety had the lowest values, followed by bagged 'Doña María' (14.67 and 18.05 mg kg^{-1} respectively). The variety which had the highest concentration of volatile compounds was 'Dominga' (46.9 mg kg^{-1}) and the lowest concentration of volatile compounds was found in the 'Doña María' variety (16.36 mg kg^{-1}). Regarding treatment, there were no significant differences between bagged and unbagged grapes in the total concentration of volatile compounds (29.72 mg kg^{-1} in bagged grapes and 38.42 mg kg^{-1} in unbagged grapes), as shown in Table 2. This aligns with the findings of Ubeda et al. [18], who reported that the white grape variety they studied, Arra-15, exhibited no significant variation in total volatile content throughout the ripening process, although changes in specific C6 compounds and terpenes were reported.

Table 3. Concentration of volatile compounds in Vinalopó Table Grapes: bagged vs. unbagged treatments.

Volatile Compound	Concentration (μg 100 g^{-1})		
	Student's *t*-Test	Bagged	Unbagged
Aldehydes			
Hexanal	0.004 [1]	822	370
(E)-2-Hexenal	0.04	1024	631
Benzaldehyde	0.13	1.94	3.68
Octanal	0.04	7.23	22.3
Benzeneacetaldehyde	0.08	2.43	7.75
Nonanal	0.63	55.9	60.8
(E)-2-Nonenal	0.04	10.9	3.40
Decanal	0.32	12.3	14.6
β-Citral	0.50	1.14	1.26
α-Citral	0.48	0.95	1.12
Undecanal	0.11	1.37	2.06
Dodecanal	0.10	3.91	4.93
Tetradecanal	0.99	0.97	0.97
Tetradecanal	0.58	0.87	0.99

Table 3. *Cont.*

Volatile Compound	Concentration (µg 100 g^{-1})		
	Student's *t*-Test	Bagged	Unbagged
Terpenes			
o-Cymene	0.85	13.9	14.4
D-Limonene	0.24	364	260
γ-Terpinene	0.34	37.8	29.4
Terpinolene	0.93	3.94	4.03
Caryophyllene	0.56	2.37	2.74
α-Bergamotene	0.94	3.27	3.34
β-Bisabolene	0.85	2.42	2.53
Alcohols			
1-Hexanol	0.003	450	1710
Fenchol	0.73	1.12	1.20
1-Terpinenol	0.18	1.77	2.27
β-Terpineol	0.77	1.82	1.71
Terpinen-4-ol	0.47	2.58	2.98
L-α-Terpineol	0.56	5.44	6.04
Esters			
Acetic acid, hexyl ester	0.04	117	656
Octanoic acid methyl ester	0.11	0.432	0.682
Citronellol acetate	0.54	0.33	0.42
Methyl laurate	0.95	1.54	1.56
Ketones			
6-methyl-5-Hepten-2-one	0.16	1.65	2.86
2,6-di-tert-butyl-p-Benzoquinone	0.95	1.94	1.92
Carboxilic acids			
Hexanoic acid	0.70	10.9	9.76
Total (mg kg^{-1})	0.36	29.72	38.42

[1] Significant at $p < 0.05$.

Table 4. Concentration of volatile compounds in Vinalopó Table Grapes by variety.

Volatile Compound	Concentration (µg 100 g^{-1})			
	ANOVA	'Dominga'	'Aledo'	'Doña María'
Aldehydes				
Hexanal	*** [1]	411 b [2]	1052 a	327 b
(E)-2-Hexenal	NS	1078 a	775 a	628 a
Benzaldehyde	NS	2.70 a	3.01 a	2.73 a
Octanal	**	28.3 a	8.13 a	7.87 a
Benzeneacetaldehyde	NS	3.30 a	7.10 a	4.86 a
Nonanal	NS	48.8 a	68.5 a	57.7 a
(E)-2-Nonenal	***	14.4 a	4.10 ab	2.94 b
Decanal	NS	11.9 a	14.1 a	14.3 a
β-Citral	NS	1.20 a	1.06 a	1.33 a
α-Citral	NS	1.17 a	1.03 a	0.91 a
Undecanal	NS	1.89 a	1.73 a	1.54 a
Dodecanal	NS	5.31 a	4.18 a	3.77 a
Tetradecanal	NS	1.14 a	0.96 a	0.81 a
Tetradecanal	NS	0.83 a	0.88 a	1.08 a

Table 4. *Cont.*

Volatile Compound	Concentration (μg 100 g^{-1})			
	ANOVA	'Dominga'	'Aledo'	'Doña María'
Terpenes				
o-Cymene	NS	10.6 a	14.8 a	16.9 a
D-Limonene	NS	196 a	362 a	378 a
γ-Terpinene	NS	19.9 a	38.2 a	42.7 a
Terpinolene	NS	2.74 a	3.68 a	5.53 a
Caryophyllene	NS	3.51 a	2.20 a	1.96 a
α-Bergamotene	NS	4.88 a	2.60 b	2.45 b
β-Bisabolene	NS	3.33 a	1.822 a	2.271 a
Alcohols				
1-Hexanol	**	1793 a	1348 ab	97 b
Fenchol	NS	1.31 a	1.28 a	0.90 a
1-Terpinenol	NS	2.09 a	1.99 a	1.99 a
β-Terpineol	*	2.27 a	1.82 ab	1.21 b
Terpinen-4-ol	NS	3.62 a	2.52 a	2.20 a
L-α-Terpineol	NS	5.94 a	6.05 a	5.24 a
Esters				
Acetic acid, hexyl ester	**	1013 a	141 ab	4.36 b
Octanoic acid methyl ester	NS	0.36 a	0.57 a	0.75 a
Citronellol acetate	NS	0.57 a	0.22 a	0.34 a
Methyl laurate	NS	1.91 a	1.51 a	1.24 a
Ketones				
6-methyl-5-Hepten-2-one	**	4.12 a	1.78 b	0.87 b
2,6-di-tert-butyl-p-Benzoquinone	NS	2.22 a	1.92 a	1.65 a
Carboxilic acids				
Hexanoic acid	NS	6.61 a	12.9 a	11.6 a
Total (mg kg^{-1})	*	46.92 a	38.92 ab	16.36 b

[1] NS = not significant at $p < 0.05$; *, **, ***, significant at $p < 0.05$, 0.01 and 0.001, respectively. [2] Values (mean of three replications) followed by the same letter, within the same row, were not statistically different according to Tukey's multiple range test.

Table 5. Concentration of volatile compounds in bagged and unbagged 'Dominga', 'Doña María' and 'Aledo' grapes.

Volatile Compound	Concentration (μg 100 g^{-1})						
	ANOVA	Bagged 'Dominga'	Unbagged 'Dominga'	Bagged 'Aledo'	Unbagged 'Aledo'	Bagged 'Doña María'	Unbagged 'Doña María'
Aldehydes							
Hexanal	*** [1]	538 b [2]	284 b	1579 a	524 b	350 b	304 b
(E)-2-Hexenal	NS	1317 a	840 a	1110 a	441 a	644 a	611 a
Benzaldehyde	NS	3.23 a	2.16 a	1.33 a	4.70 a	1.26 a	4.19 a
Octanal	**	8.23 b	48.4 a	7.58 b	8.68 b	5.88 b	9.85 b
Benzeneacetaldehyde	NS	3.80 a	2.81 a	2.11 a	12.1 a	1.37 a	8.34 a
Nonanal	NS	54.5 a	43.1 a	66.6 a	70.4 a	46.6 a	68.9 a
(E)-2-Nonenal	***	25.2 a	3.55 b	4.93 b	3.28 b	2.52 b	3.37 b
Decanal	NS	12.8 a	11.0 a	13.3 a	14.9 a	10.7 a	17.9 a
β-Citral	NS	1.11 a	1.28 a	1.19 a	0.927 a	1.11 a	1.56 a
α-Citral	NS	1.19 a	1.15 a	0.99 a	1.06 a	0.67 a	1.14 a
Undecanal	NS	1.39 a	2.38 a	1.44 a	2.01 a	1.28 a	1.80 a
Dodecanal	NS	4.28 a	6.34 a	3.88 a	4.47 a	3.56 a	3.99 a
Tetradecanal	NS	1.06 a	1.23 a	1.09 a	0.82 a	0.76 a	0.86 a
Tetradecanal	NS	0.71 a	0.94 a	1.11 a	0.65 a	0.78 a	1.37 a

Table 5. *Cont.*

Volatile Compound	Concentration (μg 100 g^{-1})						
	ANOVA	**Bagged 'Dominga'**	**Unbagged 'Dominga'**	**Bagged 'Aledo'**	**Unbagged 'Aledo'**	**Bagged 'Doña María'**	**Unbagged 'Doña María'**
Terpenes							
o-Cymene	NS	11.0 a	10.2 a	13.9 a	15.8 a	16.7 a	17.1 a
D-Limonene	NS	186 a	206 a	373 a	351 a	533 a	223 a
γ-Terpinene	NS	19.1 a	20.6 a	39.6 a	36.7 a	54.6 a	30.7 a
Terpinolene	NS	2.88 a	2.61 a	4.04 a	3.32 a	4.89 a	6.18 a
Caryophyllene	NS	2.83 a	4.18 a	2.46 a	1.929 a	1.81 a	2.12 a
α-Bergamotene	NS	4.77 a	4.99 a	2.68 a	2.52 a	2.37 a	2.52 a
β-Bisabolene	NS	3.56 a	3.09 a	1.95 a	1.69 a	1.73 a	2.81 a
Alcohols							
1-Hexanol	**	1090 ab	2497 a	175 b	2523 a	85.4 b	109 b
Fenchol	NS	1.43 a	1.19 a	1.12 a	1.44 a	0.81 a	0.98 a
1-Terpinenol	NS	2.09 a	2.09 a	1.64 a	2.35 a	1.58 a	2.39 a
β-Terpineol	*	2.87 a	1.67 ab	1.52 ab	2.12 ab	1.08 b	1.34 ab
Terpinen-4-ol	NS	3.89 a	3.36 a	1.76 a	3.28 a	2.10 a	2.30 a
L-α-Terpineol	NS	6.74 a	5.13 a	5.03 a	7.06 a	4.56 a	5.92 a
Esters							
Acetic acid, hexyl ester	**	338 b	1689 a	7.96 b	275 b	4.20 b	4.52 b
Octanoic acid methyl ester	NS	0.09 b	0.63 ab	0.59 ab	0.55 ab	0.62 ab	0.874 a
Citronellol acetate	NS	0.65 a	0.49 a	0.13 a	0.32 a	0.22 a	0.46 a
Methyl laurate	NS	1.71 a	2.11 a	1.36 a	1.65 a	1.56 a	0.93 a
Ketones							
6-methyl-5-Hepten-2-one	**	3.96 ab	4.28 a	0.602 c	2.95 abc	0.398 c	1.34 bc
2,6-di-tert-butyl-p-Benzoquinone	NS	2.43 a	2.00 a	2.06 a	1.77 a	1.34 a	1.97 a
Carboxilic acids							
Hexanoic acid	NS	6.93 a	6.29 a	11.8 a	13.9 a	14.0 a	9.07 a
Total (mg kg^{-1})	*	36.66 ab	57.17 a	34.44 ab	43.40 ab	18.05 ab	14.67 b

[1] NS = not significant at $p < 0.05$; *, **, ***, significant at $p < 0.05$, 0.01 and 0.001, respectively. [2] Values (mean of three replications) followed by the same letter, within the same row, were not statistically different according to Tukey's multiple range test.

Among the determined compounds in this work, hexanal, (E)-2-hexenal and 1-hexanol are predominant and significantly contribute to the characteristic green and grassy aroma of table grapes, which are also predominant in 'Dominga', 'Doña María' and 'Aledo' varieties. Bagged 'Aledo' grapes showed the highest concentration of hexanal, while no significant differences were observed between varieties for (E)-2-hexenal. However, Blanch et al. [19] provided a detailed analysis of the volatile profiles in seedless table grape varieties, highlighting the prominence of esters, terpenoids, and aldehydes in their volatile composition. Similarly, Kaya et al. [20] reported that in Italia and Bronx Seedless table grapes, the most abundant volatile families were esters, fatty acids, terpenes, and C6 compounds, with hexanoic acid, ethyl 3-hydroxybutyrate, ethyl octanoate, 2-hexenoic acid, and octanoic acid being the most prominent components. These findings highlight the strong influence of varietal characteristics and metabolic pathways on the composition and distribution of volatile compounds in table grapes, emphasizing the role of both genetic factors and agronomic conditions in shaping their aromatic profile. Among esters, compounds such as ethyl acetate and ethyl butyrate were identified by Blanch et al. [19] as key contributors to fruity and sweet aromas. Although several studies have examined volatile compounds in table grapes, no published data are currently available for the specific volatile profiles of the 'Aledo', 'Doña María', or 'Dominga' varieties, which limits direct comparisons with the existing literature. Table 6 presents odor thresholds [21–23], the odor activity values (OAVs > 1) and relative odor contributions (ROCs) of the volatile compounds considered perceptible in the grape samples analyzed. For instance, hexyl acetate was detected at relatively high concentrations, particularly in unbagged 'Dominga'. However, due to its elevated odor threshold, its calculated OAV remained below 1 and, therefore, it was excluded from the present analysis. Among the compounds identified, hexanal and (E)-2-nonenal exhibited the highest sensory relevance across all samples, as reflected by their elevated OAV and ROC values. Hexanal showed consistently high values in all varieties and treatments, with a maximum OAV of 3510 and a ROC of 78.6% in bagged 'Aledo', confirming its prominent role in contributing to the green and grassy aroma of table grapes. Even in the sample with the lowest relative contribution (bagged 'Doña María'), hexanal still accounted for 46.1% of the total ROC. Similarly, (E)-2-nonenal, due to its low odor threshold, exhibited notable OAVs despite its low concentration. The highest value was recorded in bagged 'Dominga' (OAV = 1325; ROC = 13.0%), with ROC values ranging from 5.81% to 12.9%, indicating a secondary but consistent contribution to the aroma profile. In contrast, (E)-2-hexenal, although it was detected at relatively high concentrations in several samples, displayed limited sensory impact, with ROC values ranging from 2.72% (unbagged 'Aledo') to 7.02% (bagged 'Aledo'). Other aldehydes such as nonanal, dodecanal, and benzeneacetaldehyde exhibited low OAV and ROC values in all treatments, suggesting minimal olfactory significance. Among the terpenes, D-limonene and o-cymene showed perceptible contributions, particularly in bagged 'Doña María', where D-limonene reached an OAV of 533 and a ROC of 32.3%, indicating a more subtle role in contributing citrus and floral aromatic nuances. These observations suggest a potential effect of the bagging technique on the accumulation of specific aroma-active aldehydes and terpenes, especially in the 'Aledo' variety, and highlight the influence of both varietal and treatment-related factors on the aromatic profile of table grapes.

The relative abundance of each chemical group is presented (Figures 1 and 2). Figure 1 compares both variety and bagging treatment, while Figure 2 focuses exclusively on the effect of the bagging treatment. Bagged 'Aledo' grapes showed more than 80% relative abundance of aldehydes, the highest among all groups studied, while bagged 'Doña María' grapes presented over 30% terpenes, also the highest recorded. Unbagged 'Aledo' grapes displayed nearly 60% alcohols, representing the highest alcohol content observed. Overall, bagged grapes demonstrated a greater proportion of aldehydes, while unbagged grapes were characterized by higher levels of alcohols and esters. Canturk et al. [17] reported that C6 compounds accounted for 73.2% to 89.0% of the total volatile compounds detected in novel hybrid table grape varieties and terpenes represent up to 11.3% of the volatile profile in some hybrids. The abundance of 1-hexanol, which plays a role in the overall aroma with green, herbaceous, woody and sweet notes, ranged from 0.2% to 7.3% in the grapes studied by Canturk et al. [17], and from 0.25 to 2.35% in the seedless table grape varieties determined by Blanch et al. [19]. By contrast, the result of this work showed that the relative content of these alcohols ranged from 7.4% in unbagged 'Doña María' grapes to 58% in 'Aledo' unbagged grapes.

Regarding terpenes, the average relative abundance in unbagged grapes was 8.24%, while bagged grapes reached 14.39%. The bagged 'Doña María' variety showed the highest proportion in this group of compounds, with terpenes representing 34.09% of its volatile profile. Cantürk et al. [17] reported that terpenes can account for up to 11.3% of the volatile profile in some hybrids, while Li et al. [3] identified genetic markers involved in terpene biosynthesis, with terpene levels ranging from 10.26% to 41.67%. These findings, along with our results, suggest that the variability observed between varieties may be influenced by genetic factors affecting terpene production, as well as by processing conditions such as the bagging technique, which could alter the expression or retention of these compounds. These differences may therefore reflect a complex interaction between compositional, genetic, and environmental factors.

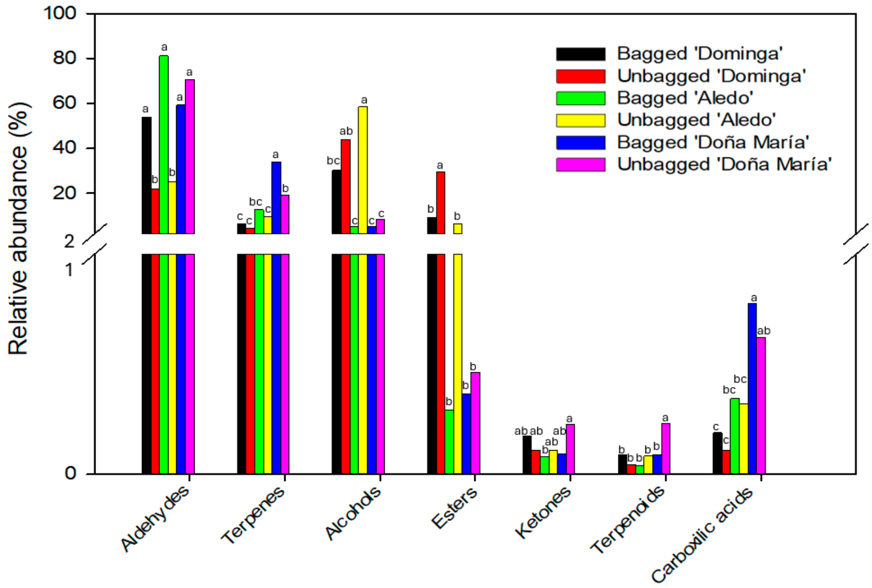

Figure 1. Relative abundance of chemical families of volatile compounds identified in bagged and unbagged 'Dominga', 'Doña María', and 'Aledo' grapes. Bars with the same letter within the same chemical family were not significantly different at $p < 0.05$, according to Tukey's multiple range test.

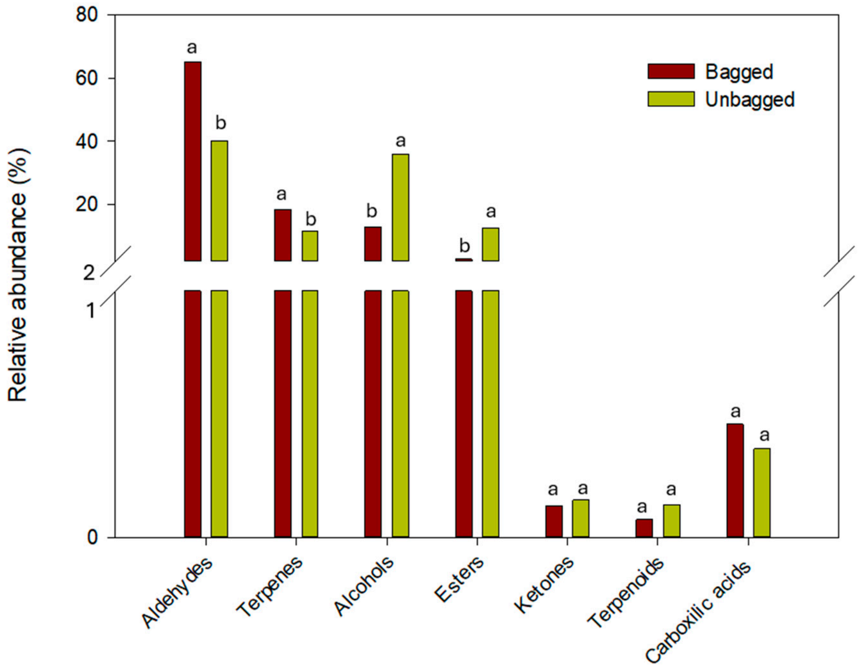

Figure 2. Relative abundance of chemical families of volatile compounds identified in bagged and unbagged grapes. Bars with the same letter within the same chemical family were not significantly different at $p < 0.05$, according to Student's *t*-test.

Table 6. Odor activity values (OAV) and relative odor contributions (ROC) in bagged and unbagged 'Dominga', 'Doña María' and 'Aledo' grapes.

Volatile Compound	Odor Threshold ($\mu g\ 100\ g^{-1}$)	Bagged 'Dominga'		Unbagged 'Dominga'		Bagged 'Aledo'		Unbagged 'Aledo'		Bagged 'Doña María'		Unbagged 'Doña María'	
		OAV	ROC (%)	OAV	ROC (%)	OAV	ROC (%)	OAV	ROC (%)	OAV	ROC (%)	OAV	ROC (%)
Hexanal [1]	0.45 [2]	1196	39.0 [3]	631	43.2	3510	78.6	1164	58.9	777	46.1	675	49.2
(E)-2-Hexenal	8.20	161	5.23	102	7.02	135	3.03	53.7	2.72	78.6	4.66	74.5	5.44
1-Hexanol	250.00	4.36	0.14	9.99	0.68	0.70	0.02	10.1	0.51	0.34	0.02	0.44	0.03
Octanal	0.40	20.6	0.67	121	8.28	18.9	0.42	21.7	1.10	14.7	0.87	24.6	1.80
o-Cymene	0.50	22.0	0.72	20.4	1.40	27.7	0.62	31.6	1.60	33.4	1.98	34.2	2.50
D-Limonene	1.00	186	6.05	206	14.1	373.	8.35	351	17.7	533	31.6	222	16.3
Benzeneacetaldehyde	0.63	6.02	0.20	4.46	0.31	3.35	0.08	19.2	0.97	2.18	0.13	13.2	0.97
Linalool	0.60	5.37	0.18	4.32	0.30	2.40	0.05	5.98	0.30	2.68	0.16	5.73	0.42
Nonanal	2.80	19.5	0.63	15.4	1.05	23.8	0.53	25.1	1.27	16.6	0.99	24.6	1.80
(E)-2-Nonenal	0.02	1325	43.2	187	12.8	259	5.81	172	8.72	132.	7.85	177.6	13.0
Decanal	0.93	13.8	0.45	11.8	0.81	14.2	0.32	16.0	0.81	11.5	0.68	19.3	1.41
Undecanal	0.50	2.79	0.09	4.77	0.33	2.89	0.06	4.02	0.20	2.56	0.15	3.6	0.26
Dodecanal	0.05	80.7	2.63	120	8.19	73.2	1.64	84.4	4.27	67.2	3.98	75.2	5.49
Caryophyllene	1.00	2.83	0.09	4.18	0.29	2.46	0.06	1.93	0.10	1.81	0.11	2.12	0.15
2,6-di-tert-butyl-p-Benzoquinone	0.11	22.1	0.72	18.2	1.25	18.7	0.42	16.1	0.81	12.1	0.72	17.9	1.31

[1] Only compounds with OAV ≥ 1 are included. [2] References [21–23]. [3] ROC (%) was calculated as $(OAV_i/\sum OAV_i) \times 100$, considering only compounds with OAV ≥ 1 in each sample.

The analysis of volatile compounds in bagged and unbagged table grapes revealed significant differences in their aromatic profiles, closely associated with ripening dynamics. Unbagged grapes exhibited higher concentrations of alcohols and esters, compounds typically linked to advanced ripening and fermentation processes [18]. Among the most prominent volatiles detected in unbagged samples were 1-hexanol, 6-methyl-5-hepten-2-one, octanoic acid, and octanoic acid methyl ester. Other authors determined that alcohols such as ethanol and ethyl acetate are associated with the natural progression of the fruit ripening and over-maturity, but in their study, only terpenoids reflected a common increasing trend during ripening and they suggested that it depends on the variety [18]. The alcohol 1-hexanol, detected at higher concentrations in unbagged grapes, is known to contribute to fresh, green, and herbaceous aromas at moderate levels. However, in overripe or damaged fruits, excessive accumulation of 1-hexanol can lead to undesirable sensory attributes, including fermented or off-putting notes. Studies have identified 1-hexanol as a key volatile compound in mature and overripe guava fruits, with concentrations reaching 3.020 and 2.379 $\mu g\ kg^{-1}$, respectively, indicating its association with fruit overripening [24]. This elevated concentration of alcohols in unbagged grapes may be attributed to overripening, which enhances metabolic processes leading to increased alcohol production [25]. Similarly, 6-methyl-5-hepten-2-one, known for its pungent and sweat-like aroma, has been associated with fruit senescence and oxidative stress [26]. The presence of octanoic acid and its derivative octanoic acid methyl ester further supports the hypothesis of advanced metabolic processes in unbagged grapes. Octanoic acid contributes to a fatty, waxy, and slightly rancid odor, while octanoic acid methyl ester adds a sweet and fruity aroma, which, at high concentrations, becomes waxy and less fresh [14,24,27]. These compounds collectively suggest a more advanced ripening stage and potentially less desirable aroma profile for unbagged grapes.

Unbagged grapes showed higher concentrations of esters, which are associated with fruity and sweet aromas, and in some cases floral notes, as previously reported in table grapes and other fruit species [20]. Among the studied varieties, 'Dominga' grapes showed a particularly high accumulation of these compounds, suggesting a varietal influence on ester biosynthesis, which has been reported by other authors [14,24,28]. Kaya et al. [20] determined that esters are key contributors to the aromatic complexity of grapes, with compounds such as ethyl acetate, ethyl isobutyrate, butyl acetate, and ethyl hexanoate playing a significant role in defining the volatile profile of different grape varieties. Studies have shown that the Italia variety accumulates higher concentrations of these esters compared to Bronx Seedless, reinforcing the role of genetic factors in volatile compound formation [20]. Additionally, the ripening stage has been found to significantly impact ester composition and some studies linked elevated ester content to fruit overripeness. Kaya et al. [20] reported that esters tend to increase in concentration as ripening progresses, a trend that has also been observed in other fruit species. In apples, Ferenczi et al. [29] found that overripe and alcoholic sensations are predicted to increase two weeks after the initiation of ripening in response to an increase in the production of ethyl esters. In the case of wine, the results of Sancho-Galán et al. [30] indicated that the concentration of esters, such as ethyl acetate, increased in wines produced from overripe grapes, which is associated with fermentation-derived aromas. While our study did not specifically assess the relationship between overripeness and ester degradation, the higher ester concentrations observed in unbagged grapes suggest that these grapes were at a more advanced ripening stage. This aligns with previous findings, indicating that esters may serve as potential ripeness markers in table grapes.

On the other hand, bagged grapes exhibited higher concentrations of aldehydes such as hexanal, and 2-nonenal, which are associated with fresher and greener aromatic notes.

Hexanal was significantly elevated in bagged grapes and is known for its green, grassy, and fresh aroma [14,15]. Studies have shown that the odor activity value of hexanal reaches high levels in mature grapes, indicating its key role in aroma formation [31]. Furthermore, 2-nonenal, which has been detected in grapes and is linked to green and fresh aromatic notes, was also found in higher concentration in bagged grapes, which agreed with other studies [3,31]. According to Oliveira et al. [25], aldehydes reach their highest concentration at intermediate fruit maturation, with levels ranging from 2 to 12 times higher than those found in unripe or fully ripe fruits. Their relative proportion follows a similar trend, increasing during the early maturation stages and subsequently declining in ripe fruits. However, in the study of He et al. [2] the peak of aldehydes was achieved in unbagged grapes and a lower concentration of C6 aldehydes was found in the harvested berries under bagged treatments.

Research has also demonstrated that the variety, the timing and duration and type of bagging influence the volatile organic compounds in grapes, directly affecting their aromatic profile. In the study of He et al. [2], all bunch bagging treatments had a strong effect on volatile composition and these treatments decreased the concentration of four key compounds—nerol, benzaldehyde, benzeneacetaldehyde, and p-cymene—while leading to an increase in phenol and 3,4-dimethylbenzaldehyde. Similarly, Ubeda et al. [18] observed that the evolution of C6 compounds during ripening was strongly dependent on the grape cultivar. In 'Crimson', 'Magenta', and 'Krissy' varieties, most aldehydes and alcohols increased throughout the ripening process until harvest, suggesting a cultivar-dependent pattern in the accumulation of these volatile compounds. Regarding the effect of colored paper bags, Ji et al. [32] reported that fruit bagging is an effective technique for enhancing grape aroma in 'Kyoho' grapes by modulating the production of volatile compounds. Guo et al. [33] confirmed that grape bagging delayed fruit coloring, sugar accumulation, berry weight gain, and overall ripening. Additionally, their study discovered that bagged berries of both 'Cabernet Sauvignon' and 'Carignan' cultivars synthesized higher amounts of melatonin compared to unbagged berries, suggesting a novel approach in viticulture for managing grape development and metabolic processes. The effects of bagging on volatile composition also depend on the type of bag used. Wang et al. [8] found that different bagging materials altered the total amount of aromatic components in Muscat-flavored grapes, with black bags having the most pronounced negative impact, followed by brown and yellow bags, while white bags resulted in the least reduction in aromatic compounds. Furthermore, Ji et al. [32] reported that while red, green, blue, and white paper bags favored the accumulation of esters, they simultaneously inhibited the accumulation of aldehydes, alcohols, terpenes, ketones, and acids, highlighting the complex role of bagging in shaping the aromatic profile of grapes.

Principal component analysis (PCA) was performed to obtain an easier and complete understanding of the relationship among the studied cultivars and their volatile compounds (Figure 3). The first principal component (F1) accounted for 43.39%, and the second one accounted for (F2) 21.99% of the total variance. It is important to remember that the higher the distance between two parameters, the lower their correlation. Although the first two principal components explained 65% of the total variance, the PCA was used as an exploratory tool to observe general patterns and sample distribution based on volatile composition.

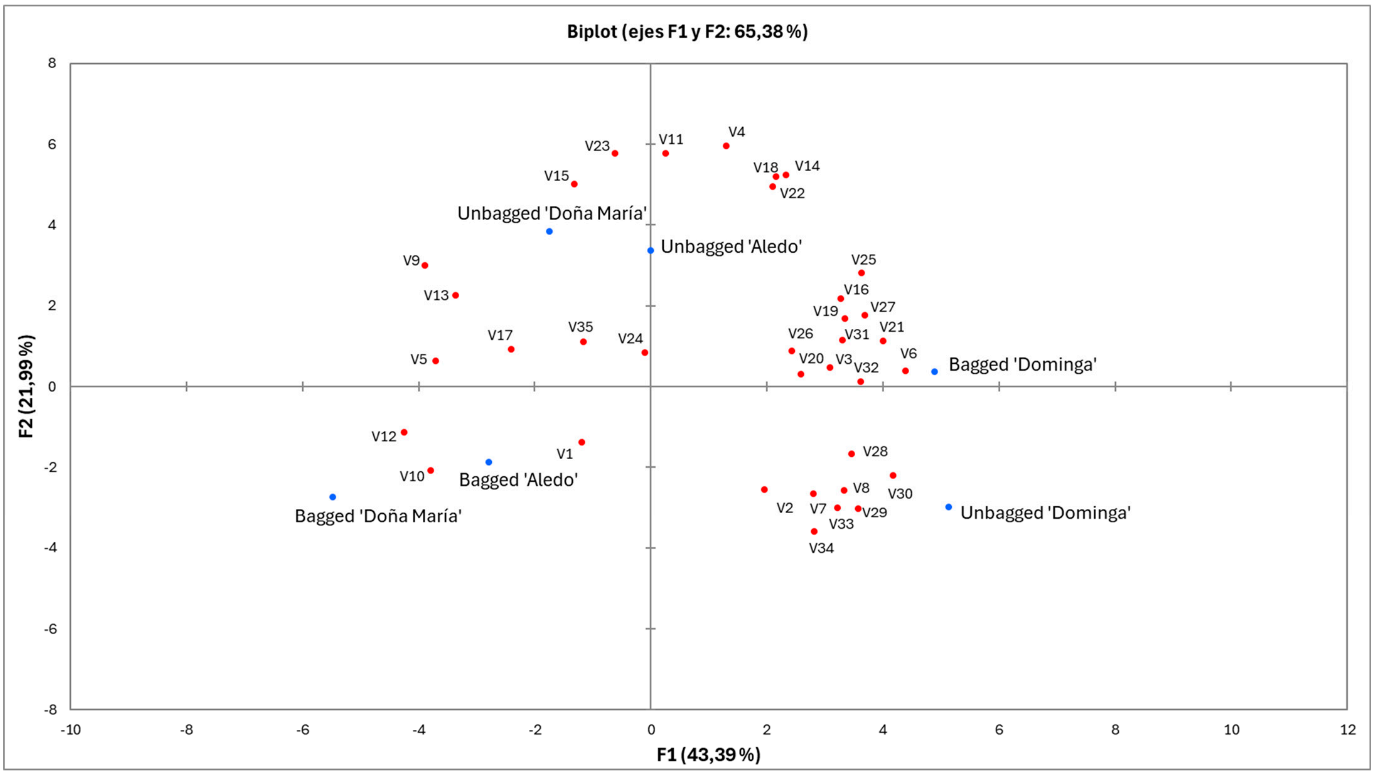

Figure 3. Principal component analysis (F1 and F2) of bagged and unbagged 'Dominga', 'Doña María', and 'Aledo' grapes.

F1 was positively linked with 6-methyl-5-hepten-2-one, terpinen-4-ol, citronelol acetate,β-bisabolene, 2,6-di-tert-butyl-p-benzoquinone, 1-hexanol, (E)-2-nonenal, undecanal, β-terpineol, fenchol, α-citral, α-bergamotene, dodecanal, acetil acid hexyl ester, caryophyllene, tetradecanal (isomer 1), octanal, (E)-2-hexenal and methyl laurate; and negatively with γ-terpinene, D-limonene, hexanoic acid, o-cymene, terpinolene, and octanoic acid methyl ester. F2 was positively linked with nonanal, γ-terpinene, decanal, benzaldehyde, 1-terpinenol, L- α-terpineol and linalool, and inversely linked with methyl laurate.

The principal components F1 and F2 were able to establish differences among samples. Bagged 'Aledo' and bagged 'Doña María', which were positioned in the lower left part of the graph, were correlated with the presence of volatile compounds with fresh and citrus notes, such as γ-terpinene, D-limonene, and hexanal. On the other hand, unbagged 'Doña María', situated in the upper left part of the graph, was positively linked with compounds exhibiting fatty and wine-like notes, mainly nonanal and benzeneacetaldehyde. With respect to F1, both bagged and unbagged 'Dominga', the most aromatic variety, were positioned on the right side of the graph and were associated with terpenes and other characteristic compounds of the variety, such as α-bergamotene, caryophyllene, 1-hexanol. Furthermore, unbagged 'Dominga' is linked to esters, such as acetic acid hexyl ester and methyl laurate, which are associated with a more advanced ripening stage [18,20].

4. Conclusions

The results of this study provide valuable insights into the impact of preharvest bagging on the volatile composition of 'Dominga', 'Aledo', and 'Doña María' grapes. A total of 35 volatile compounds were identified, with aldehydes, terpenes, and alcohols being the predominant chemical families. 'Dominga' showed the highest volatile compound concentration, while 'Doña María' had the lowest. Although no significant differences were observed in the total volatile content between bagged and unbagged grapes, notable variations were found in the relative composition of specific volatile families. Bagged grapes demonstrated a higher proportion of aldehydes, particularly hexanal and (E)-2-hexenal, compounds associated with fresh and green sensory attributes. In contrast, unbagged grapes exhibited higher concentrations of alcohols and esters, which are linked to fruity and overripe aromas. These findings align with previous studies indicating that the bagging technique modifies the microenvironment of grape clusters, influencing the biosynthesis of volatile compounds. The results suggest that preharvest bagging may contribute to the preservation of fresher aromatic notes while reducing the accumulation of volatiles related to advanced ripening. Further research is needed to explore the long-term effects of bagging on postharvest quality, consumer acceptance, and potential applications in optimizing sensory attributes in commercial grape production.

Author Contributions: Conceptualization, L.A.-C. and F.H.; methodology, L.N.-A.; validation, F.H. and E.S.; formal analysis, L.A.-C. and L.N.-A.; investigation, L.A.-C.; resources, F.H. and E.S.; data curation, L.A.-C. and L.N.-A.; writing—original draft preparation, L.A.-C.; writing—review and editing, L.N.-A., F.H. and E.S.; visualization, F.H. and E.S.; supervision, F.H. and E.S.; project administration, F.H. and E.S.; funding acquisition, F.H. and E.S. All authors have read and agreed to the published version of the manuscript.

Funding: The GCMS has been acquired thanks to Grant EQC2018–004170-P funded by MCIN/AEI/10.13039/501100011033 and by ERDF A way of making Europe. Project AICO/2021/326 financed by the Autonomous Community of the Comunidad Valenciana through Conselleria de Innovación, Universidades, Ciencia y Sociedad Digital.

Data Availability Statement: The raw data supporting the conclusions of this article will be made available by the authors on request.

Acknowledgments: The authors thank the Protected Designation of Origin (PDO) 'Uva de Mesa Embolsada del Vinalopó' for their support, and J. A. Martínez Cazorla, the owner of the vineyards, for his kind collaboration.

Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

ANOVA	Analysis of variance
GC–MS	Gas chromatography–mass spectrometry
HS-SPME	Headspace solid-phase microextraction
KI	Kovat Index
NS	Not significant
PCA	Principal component analyses
PDO	Protected Designation of Origin
RT	Retention time

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