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Enhancing the Aroma of Dealcoholized La Mancha Tempranillo Rosé Wines with Their Aromatic Distillates

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Abstract: The increasing demand for non-alcoholic beverages has led to the development of dealcoholized wines. However, current dealcoholization techniques often negatively impact wine aroma due to the loss of volatile compounds. This study investigates the impact of incorporating an aromatic distillate, collected during the spinning cone column (SCC) dealcoholization process, back into dealcoholized Tempranillo rosé wines. The aromatic distillate was added to dealcoholized wine in varying concentrations (0.5%, 1.0%, and 1.5% *v*/*v*). A total of 57 volatile compounds, including 25 varietal and 32 fermentative compounds, were identified and quantified using gas chromatography–mass spectrometry (GC-MS). The addition of the aromatic distillate significantly increased the concentration of several volatile compounds, notably C_6 compounds, terpenes, benzene compounds, and esters. The odor activity values (OAVs) reveal that increasing distillate concentrations led to a higher number of compounds with OAVs greater than 1, indicating enhanced individual aroma contributions. The fruity and sweet aromatic series were predominant in all samples, with their total intensity increasing with higher distillate concentrations. However, the addition of 1.5% *v*/*v* of the aromatic distillate (AW3) resulted in an alcohol content exceeding the legal limit for dealcoholized wine, classifying it as a reduced-alcohol wine. The study concludes that adding 1% *v*/*v* of the aromatic distillate to dealcoholized Tempranillo rosé wine effectively enhances the aroma profile while remaining within regulatory limits for dealcoholized wine. This approach presents a viable method for producing high-quality, aromatic, dealcoholized wines that meet consumer demand for non-alcoholic beverages.

Keywords: dealcoholized wine; aromatic distillate; volatile compounds; GC-MS; sensory analysis

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1. Introduction

Currently, new trends in wines due to the negative health effects of alcohol have had a considerable impact on the viticulture sector worldwide over the past twenty years [1,2]. The World Health Organization (WHO) is raising awareness about the harmful effects of alcohol on health, promoting the development of non-alcoholic beverages, and encouraging governments to increase taxes on alcoholic drinks (EU regulation No. 606/09, 2009) [3].

Considering this, the development of new non-alcoholic beverages, including dealcoholized wine, is becoming increasingly common in Spanish wineries [4–6]. However, there are legislative hurdles, as a product cannot be called "wine" if it does not reach a minimum ABV of 8.5% *v*/*v* of alcohol [1,3]. Nevertheless, due to new circumstances, the CAP reform (Common Agricultural Policy, EU regulation 2117/2021) [7], based on the OIV resolutions [8–10], includes dealcoholized wines, fully or partially, under the new EU rules [5].

Several innovative technological processes for dealcoholization have been explored, particularly post-fermentation techniques like the spinning cone column (SCC) [11,12]. This vertical distillation works in two phases: first, the aromatic fraction is captured under vacuum conditions and low temperatures for recovery and potential reintroduction into the wine, and second, the ethanol is removed using a higher temperature and lower vacuum conditions than in the first stage [11,13,14]. Although after the second stage of ethanol removal the aromatic fraction is added back to the wine, a total reconstitution of the volatile compounds does not take place since many of them may have been eliminated together with the ethanol in the dealcoholization stage and, therefore, are not found in the aromatic distillate coming from the first stage [15,16].

Every dealcoholization technique has its own advantages and disadvantages in terms of ethanol removal efficiency, sensory quality impact, and economic implications. However, all techniques experience a loss of volatile compounds, thus a reduction in the aromatic profile of volatile compounds due to the elimination of ethanol [15]. To mitigate this loss of volatile compounds during dealcoholization, various strategies have been studied to improve the aromatic profile of these wines [5,17,18]. Among these approaches, Liguori et al., in 2018 [19], proposed the addition of grape must to low-alcohol wines to restore some of the lost aromatic compounds, but this notably increased the sugar content of the final product. The incorporation of flower extracts in dealcoholized wine was investigated in other studies [16,17], as well as the introduction of glycosidic aromatic grape precursors from grape varieties classified as aromatic [5,18]. Another strategy, applicable when dealcoholization is performed using SCC, involves recovering volatile compounds from the extraction solution after dealcoholization and reintroducing them into low-alcohol beverages. In this case, the maximum recovery of the aromatic compounds in the first stage of the SCC technique is mandatory to obtain an adequate aromatic profile in the final wine [15]. Nevertheless, no bibliographic references have been found about this strategy, and their use is not approved as an oenological practice by the European Union (EU). However, the International Organization of Vine and Wine (OIV) states that although the regulation is a significant limitation, this strategy could be adopted in the case of special and aromatized wines.

In this context, the aim of this study was to research the incorporation of the aromatic distillate from dealcoholization into the final dealcoholized wine in different concentrations as a potential technique for improving the volatile profile of dealcoholized La Mancha Tempranillo rosé wines.

2. Materials and Methods

2.1. Reagents and Standards

Dichloromethane, ethanol, and methanol were procured from Merck (Darmstadt, Germany), while anhydrous sodium sulfate was obtained from Panreac (Barcelona, Spain). Ultrapure water, with a conductivity of 0.000006 S/m, was produced using the Milli-Q purification system by Millipore (Bedford, MA, USA). LiChrolut EN resins were supplied by Merck (Darmstadt, Germany). An alkane solution (C_7–C_{24}, Supelco, Bellefonte, PA, USA) dissolved in dichloromethane was used to determine the linear retention index (RI) of each volatile compound. Pure standards were acquired from Sigma-Aldrich (Madrid, Spain), Merck (Darmstadt, Germany), Fluka (Madrid, Spain), Lancaster (Strasbourg, France), and Firmenich (Geneva, Switzerland). This information is shown in Table S1.

2.2. Samples

2.2.1. Initial Tempranillo Rosé Wine

The Tempranillo rosé dealcoholized wines were supplied by a wine cellar of the La Mancha region. To produce the initial wine, grapes were manually harvested and transported in 50 kg boxes to the winery, where a series of assessments were conducted to classify the grapes according to their quality. The process followed the traditional red wine vinification scheme. The grapes underwent destemming and crushing, and sulfur dioxide was added in the form of $K_2S_2O_7$ (100 mg/kg), with a sulfur dioxide yield of 50%. The must was inoculated with the yeast *Saccharomyces cerevisiae cerevisiae* (CECT nº 10835), and then maceration was carried out with the solid parts of the grape in stainless steel tanks for 12–18 h before racking. After racking, the rosé must was transferred to self-emptying tanks where it was maintained at a constant temperature of 17–18 °C until the completion of alcoholic fermentation, reaching a constant density of 995 g/L. The final wines were then subjected to the dealcoholization process.

2.2.2. Dealcoholized Wine

The dealcoholization process was conducted using the spinning cone column (SCC) technique under the conditions outlined by Sam et al. in 2021 [16]. Initially, a portion of the rosé control wine was processed at a vacuum pressure of 0.04 atm and a temperature of 28 °C using the SCC to recover volatile compounds, which constitute approximately 1% of the total wine volume. The obtained dearomatized wine was then subjected to a slightly higher vacuum pressure and a temperature of 38 °C to remove the alcohol, constituting the dealcoholizing process. Then, the wines were racked, bottled, and stored at 4 °C. The aromatic distillate obtained from the first stage of dealcoholization of wines was stored at −4 °C in order to incorporate it into the final wine.

2.2.3. Incorporation of Aromatic Distillate to Dealcoholized Wines

The aromatic distillate, with an ethanol concentration of 55% v/v, obtained from the dealcoholizing process, was added to the dealcoholized wine in different concentrations. The samples were prepared by adding the aromatic distillate in a proportion of 0.5, 1.0, and 1.5% v/v (AW1, AW2, and AW3, respectively) to one liter of Tempranillo rosé dealcoholized wine in order not to exceed the 1% v/v of ethanol. A control sample wine (CW) was also analyzed. All samples were prepared in duplicate.

2.3. Conventional Analysis

The methodologies recommended by the International Organisation of Vine and Wine (O.I.V., 2022) [20] were employed to measure the standard physicochemical parameters. The analysis encompassed pH, total acidity, volatile acidity, ethanol content, and both free and total SO_2 for every sample studied. The analysis was carried out in duplicate.

2.4. Analysis of Free Minor Volatile Compounds

2.4.1. Isolation of Free Minor Volatile Compounds

Analysis of free volatile compounds of control dealcoholized wine, aromatic distillate, and the samples with added aromatic distillate were carried out 24 h after the addition of the aromatic distillate. A total of 100 mL of the sample, containing 40 µL of 4-nonanol as the internal standard (1.04 g/L in absolute ethanol), was processed through SPE cartridges (500 mg) from Merck at a flow rate of 1 mL/min, following the method described by Sánchez-Palomo et al. in 2006 [21]. To elute the minor free volatile compounds, 10 mL of dichloromethane was used, and this extract was finally concentrated under a nitrogen stream to a final volume of 200 µL. All samples were analyzed in duplicate.

2.4.2. Identification and Quantification of Volatile Compounds by Gas Chromatography Coupled with Mass Spectrometry (GC-MS)

Minor volatile compounds of the wine samples and aromatic distillate were analyzed by gas chromatography coupled with mass spectrometry (GC-MS). An Agilent 6890N GC system, equipped with a Mass Selective Detector (model 5973 inert) and a DB-WAX column (60 m × 0.25 mm × 0.25 µm) from Agilent Technologies, Inc. (Santa Clara, CA, USA), was utilized for this purpose. In the splitless mode, 1 µL extract was injected at 250 °C, with helium as the carrier gas at a flow rate of 1 mL/min. The oven temperature program started at 70 °C for 5 min, increased by 1 °C/min to 95 °C, held for 10 min, then increased by 2 °C/min to 200 °C, and maintained for 40 min. The mass spectrometer operated in the electron impact mode with an electron energy of 70 eV, recording the global run time in the full scan mode (40–450 m/z mass range) with an ion source temperature of 230 °C.

Volatile compounds were identified by comparing their mass spectra with those of authentic compounds and data system libraries (NBS75K). To confirm the identity of each volatile compound, the linear retention index (LRI) was calculated using the retention times of a mixture of straight-chain alkanes (C_7–C_{24}) under the same chromatographic conditions.

Response factors for each volatile compound were determined by injecting commercially available standards at concentrations typically found in wines. The standards and the samples were added in the same amounts as the internal standard. For compounds not commercially available, the response factors of structurally similar compounds were used. These response factors were then applied to calculate the concentration of each compound. All determinations were carried out in duplicate.

2.5. Odor Activity Values

The contribution of each volatile compound to the aroma of the dealcoholized wines with added aromatic distillate was determined using the odor activity values (OAVs), which can be used as an approach to determine the sensory profile of samples. The OAV of each compound was calculated as the relation of c/t, with c being the concentration of each volatile compound in the wine sample and t being the odor threshold of the compound available in the literature [22–24]. The individual contributions of volatile compounds to the wine aroma were considered when the OAV was higher than one, while a synergetic effect with other volatile compounds was considered when the OAV was between 0.1 and 1. Moreover, aromatic descriptors were assigned to each of the volatile compounds in the wines, and according to this, the compounds were categorized into an aromatic series. This classification was used to determine the sensory profile of the wine based on its chemical composition [25–27].

2.6. Sensory Descriptive Analysis

A sensory evaluation of wines was carried out by a trained panel of eleven assessors (six females and five males, with ages ranging between 34 and 60 years) with extensive experience in the sensory analysis of wines. The assessors were trained based on the international standards, UNE-EN ISO 8586:2014 [28], which include the detection and recognition of tastes and odors, as well as the use of scales. Sensory sessions were conducted in a standard sensory analysis chamber, UNE-EN ISO 8589:2010 [29], equipped with 8 separate booths. Wine samples were stored at 10 °C and presented at 18 °C in standard wine-tasting glasses according to the standard UNE 87022:1992 [30] and covered with a watch glass to reduce the loss of aroma compounds. Forty milliliters (40 mL) of wine was served to each assessor, who sniffed and tasted the wine to detect aromas and flavors. The assessor rated the intensity of each aroma sensory descriptor using an unstructured 10 cm scale from 0 (not perceptible) to 10 (strongly perceptible). All wines were in the same year of their production and evaluated in duplicate.

2.7. Statistical Analysis

Statistical analysis was conducted using SPSS for Windows version 28.0 (SPSS Inc., Chicago, IL, USA). A one-way ANOVA was applied to compare data from the conventional analysis results, the volatile compounds, and the sensory analysis to identify significant differences between the mean values among the studied dealcoholized wine (control wine and those with 10, 20, and 30 mL of aromatic distillate added). In all cases, if significant differences were found, the Student–Newman–Keuls test was used with a significance level of $p < 0.05$ to determine between which wine samples these differences occurred.

3. Results and Discussion

3.1. Conventional Analysis

The values of the physicochemical parameters of the dealcoholized control wine, aromatic distillate, and samples with different volumes of aromatic distillate added are shown in Table 1. In general, no significant differences were observed between the samples for the studied parameters, except for the alcohol concentration. The aromatic distillate contained 55% alcohol, while the control dealcoholized wine had 0.0% v/v of ethanol. Thus, increasing the amount of added aromatic distillate proportionally increased the alcohol percentage in each sample. Consequently, only CW, AW1, and AW2 can be classified as dealcoholized wine according to the regulations, while AW3 is classified as having a reduced ethanol content [7]. Regarding the other parameters, the values were within the limits accepted by the OIV [20].

Table 1. Conventional analysis of control dealcoholized wine (CW) and dealcoholized wines with added aromatic distillate in concentrations of 0.5 (AW1), 1 (AW2), and 1.5 (AW3) % *v*/*v*. Mean ± standard deviation.

	CW		AW1		AW2		AW3	
% alcohol (% ABV)	0.00	±0.03	0.55 [c]	±0.02	0.825 [b]	±0.05	1.1 [a]	±0.07
Total acidity (g/L tartaric acid)	4.91 [a]	±0.04	4.95 [a]	±0.02	4.89 [a]	±0.03	4.87 [a]	±0.05
pH	3.34 [a]	±0.01	3.25 [a]	±0.06	3.31 [a]	±0.05	3.28 [a]	±0.07
Free SO_2 (mg/L)	28.67 [a]	±1.15	28.52 [a]	±1.25	28.46 [a]	±1.31	28.6 [a]	±1.32
Total SO_2 (mg/L)	55.33 [a]	±0.58	55.10 [a]	±1.15	55.27 [a]	±1.05	55.32 [a]	±0.8
Residual sugars (g/L)	66.73 [a]	±0.40	66.7 [a]	±0.35	66.6 [a]	±0.25	66.65 [a]	±0.31

[a,b,c]: Different superindexes in the same row indicate significant differences at a 0.05 level according to Student–Newman–Keuls statistical test ($p \leq 0.05$) between CW, AW1, AW2, and AW3.

3.2. Volatile Composition

Gas chromatography coupled with mass spectrometry (GC-MS) allowed us to identify and quantify a total of 57 volatile compounds, comprising 25 varietal compounds and 32 compounds formed mainly during alcoholic fermentation, in Tempranillo rosé dealcoholized wine, the aromatic distillate, and samples of dealcoholized wine with different volumes of aromatic distillate added. Tables 2 and 3 show, respectively, the mean concentration and relative standard deviation of the varietal compounds and compounds formed mainly during alcoholic fermentation. One-way ANOVA and the Student–Newman–Keuls' test were employed according to the factor volume of the aromatic distillate added. Table S1 of Supplementary Materials shows the total concentrations of the main groups of minor volatile compounds identified in the samples. Table S2 of Supplementary Materials shows the mean concentration and relative standard deviation of the varietal and compounds formed mainly during alcoholic fermentation of original rosé Tempranillo wine prior to the dealcoholization process. As can be observed in Tables S2 and 2, the total dealcoholizing process decreased the concentration of higher alcohols, esters, acids, and C_6 compounds in the wines while not modifying the concentration of terpenes, benzenic compounds, and C_{13}-norisoprenoids in agreement with previous research [4].

Table 2. Mean concentration (μg/L) and relative standard deviation (n = 2) of free volatile compounds identified in aromatic distillate (AD), control dealcoholized wine (CW), and dealcoholized wines with added aromatic distillate in concentrations of 0.5 (AW1), 1 (AW2), and 1.5 (AW3) % *v*/*v*.

Compound	AD	RSD%	CW	RSD%	AW1	RSD%	AW2	RSD%	AW3	RSD%
2-hexanol	83.5	±10.0	n.d.		n.d.		n.d.		n.d.	
1-hexanol	3340	±2	96.6 [d]	±0.3	1420 [c]	±4	2865 [b]	±1	3663 [a]	±1
(E)-3-hexen-1-ol	63.0	±5.0	n.d.		33.9 [c]	±7.1	65.6 [b]	±2.6	82.9 [a]	±1.3
(Z)-3-hexen-1-ol	635,635	±1	22.7 [c]	±1.6	269.9 [b]	±2.7	490 [a]	±7	521 [a]	±1
(E)-2-hexen-1-ol	10.8	±5.2	n.d.		n.d.		5.52 [a]	±3.86	6.70 [b]	±9.93
2-ethyl-1-hexanol	15.6	±2.5	5.26 [c]	±2.07	8.06 [b]	±1.3	9.00 [b]	±8.86	13.3 [a]	±4.4

Table 2. *Cont.*

Compound	AD	RSD%	CW	RSD%	AW1	RSD%	AW2	RSD%	AW3	RSD%
C_6 COMPOUNDS	**4148**		**124 [d]**		**1732 [c]**		**3436 [b]**		**4287 [a]**	
Cis-linalool oxide furanic	35.9	±1.4	n.d.		n.d.		11.5 [b]	±3.3	22.5 [a]	±0.3
Linalool	70.5	±3.9	n.d.		5.25 [c]	±0.57	10.1 [b]	±1.70	16.2 [a]	±2.4
β-terpineol acetate	8.9	±9.6	n.d.		n.d.		n.d.		n.d.	
α-terpineol	46.9	±1.4	22.8 [c]	±2.9	25.2 [c]	±0.8	35.1 [b]	±3.9	39.9 [a]	±3.5
Trimethyl dihydronaphtalene	58.5	±1.9	n.d.		n.d.		8.26 [b]	±4.40	11.8 [a]	±0.3
Nerol	12.5	±2.5	n.d.		n.d.		n.d.		n.d.	
β-citronellol	32.9	±0.9	n.d.		8.57 [c]	±1.75	9.76 [b]	±4.90	12.2 [a]	±1.4
β-damascenone	56.7	±1.0	43.1 [d]	±1.5	45.9 [c]	±0.8	49.9 [a]	±0.6	48.0 [b]	±0.2
Geraniol	38.2	±0.5	n.d.		n.d.		n.d.		33.9 [a]	±0.0
3,7-dimethyl-1,7-octadienol	50.7	±3.0	22.4 [c]	±3.0	24.8 [c]	±7.10	27.1 [b]	±0.2	30.4 [a]	±6.5
TERPENIC COMPOUNDS	**360**		**88.3 [d]**		**107 [c]**		**151 [b]**		**214 [a]**	
Benzaldehyde	25.8	±3.7	6.59 [c]	±1.56	9.55 [b]	±0.13	9.86 [a,b]	±6.20	10.7 [a]	±0.7
Benzyl alcohol	66.1	±11.5	25.6 [d]	±8.3	38.6 [c]	±0.5	43.2 [b]	±0.7	47.7 [a]	±3.5
Phenol	32.8	±2.7	14.0 [b]	±9.5	19.8 [a]	±7.4	21.0 [a]	±3.8	19.9 [a]	±2.1
Syringol	48.1	±0.8	8.50 [b]	±9.40	29.6 [a]	±1.3	34.2 [a]	±7.1	32.8 [a]	±4.9
2,3-dihydro-benzofurane	403	±1	162 [c]	±5	259 [b]	±4	345 [a]	±5	341 [a]	±2
Benzoic acid	96.6	±0.6	64.3 [c]	±9.7	68.4 [c]	±4.5	80.6 [b]	±9.4	88.1 [a]	±1.7
Benceneacetic acid	77.4	±2.0	28.2 [d]	±5.5	33.0 [c]	±2.9	69.2 [b]	±1.8	63.2 [a]	±1.9
Zingerone	78.5	±0.8	51.2 [b]	±6.4	50.2 [b]	±7.4	64.0 [a]	±1.0	60.6 [a]	±3.3
Homovanillic acid	269	±3	49.9 [d]	±1.8	70.9 [c]	±1.1	163 [b]	±2	230 [a]	±6
BENCENIC COMPOUNDS	**1098**		**411 [d]**		**579 [c]**		**830 [b]**		**895 [a]**	

n.d.: not detected; [a,b,c,d]: different superindexes in the same row indicate significant differences at a 0.05 level according to Student–Newman–Keuls statistical test ($p \leq 0.05$) between CW, AW1, AW2, and AW3.

Table 3. Mean concentration (µg/L) and relative standard deviation (n = 2) of volatile compounds formed during alcoholic fermentation identified in aromatic distillate (AD), control dealcoholized wine (CW), and dealcoholized wines with added aromatic distillate in concentrations of 5 (AW1), 10 (AW2), and 15 (AW3) % *v*/*v*.

Compound	AD	RSD%	CW	RSD%	AW1	RSD%	AW2	RSD%	AW3	RSD%
ϒ-heptalactone	20.7	±2.3	7.34 [d]	±7.94	9.77 [c]	±2.47	13.1 [b]	±4.5	15.9 [a]	±2.1
LACTONES	**20.7**		**7.34 [d]**		**9.77 [c]**		**13.1 [b]**		**15.9 [a]**	
Butyric acid	44.5	±7.5	48.4 [b]	±6.4	50.9 [b]	±2.4	64.8 [a]	±1.7	68.2 [a]	±0.1

Table 3. *Cont.*

Compound	AD	RSD%	CW	RSD%	AW1	RSD%	AW2	RSD%	AW3	RSD%
Hexanoic acid	4035	±2	1974 [c]	±2	2599 [b]	±4	3295 [a]	±3	3250 [a]	±2
(E)-3-hexenoic acid	40.7	±1.1	27.6 [b]	±2.5	35.3 [a]	±1.2	35.4 [a]	±3.5	37.7 [a]	±8.3
(E)-2-hexenoic acid	48.2	±0.1	18.8 [b]	±4.2	31.1 [a]	±5.1	32.9 [a]	±1.6	28.4 [a]	±8.3
Octanoic acid	11,568	±3	1816 [c]	±2	3203 [b]	±4	4700 [a]	±3	4790 [a]	±2
Decanoic acid	7853	±0	155 [d]	±2	905 [c]	±5	1636 [b]	±2	2108 [a]	±1
Dodecanoic acid	784	±6	n.d.		96.3 [c]	±3.5	453 [b]	±0	289 [a]	±8
ACIDS	**24,399**		**4048 [c]**		**6931 [b]**		**10,233 [a]**		**10,590 [a]**	
Isobutanol	595	±5	48.2 [d]	±2.3	166 [c]	±0	201 [b]	±5	239 [a]	±1
1-butanol	1295	±6	n.d.		6.23 [c]	±8.01	9.45 [b]	±3.01	14.0 [a]	±0.8
1-pentanol	50.8	±2.4	n.d.		17.1 [c]	±1.3	23.8 [b]	±1.3	33.5 [a]	±7.7
2-methyl-2-buten-1-ol	50.9	±1.6	n.d.		5.80 [c]	±8.29	8.40 [b]	±3.39	10.0 [a]	±4.0
2.3-Butanediol (*levo*)	n.d.	n.d.	46.4 [a]	±4.8	43.2 [a]	±3.2	45.5 [a]	±1.5	47.4 [a]	±2.7
2.3-Butanediol (*meso*)	n.d.	n.d.	7.86 [a]	±3	7.41 [a]	±2.01	7.65 [a]	±0.21	7.95 [a]	±1.35
3-metilthio-1-propanol	n.d.		179 [b]	±3	182 [b]	±3	195 [a]	±1	198 [a]	±3
2-phenylethanol	2531	±1	12,358 [a]	±2	12,475 [a]	±4	13,390 [a]	±2	13,183 [a]	±0
ALCOHOLS	**4525**		**12,639 [a]**		**12,903 [a]**		**13,882 [a]**		**13,734 [a]**	
Ehtyl butanoate	584	±1	n.d.		119 [a]	±2	525 [b]	±2	638 [a]	±1
Ethyl isovalerate	36.3	±9.8	n.d.		8.47 [c]	±7.01	10.5 [b]	±1.2	31.5 [a]	±3.3
Isoamyl acetate	4348	±2	48.4 [d]	±0.6	446 [c]	±3	875 [b]	±0	1243 [a]	±1
Ethyl hexanoate	16,471	±7	n.d.		915 [c]	±4	2249 [b]	±2	3320 [a]	±1
Hexyl acetate	329	±0	3.49 [d]	±1.4	40.6 [c]	±9.5	89.9 [b]	±1.1	133 [a]	±1
Ethyl *cis*-3-hexanoate	31.8	±9.7	n.d.		n.d.		n.d.		18.0 [a]	±0.7
Ethyl 2-hexanoate	31.8	±1.9	n.d.		6.81 [c]	±1.16	14.0 [b]	±2.0	19.1 [a]	±3.7
Ethyl octanoate	50,156	±3	25.26 [d]	±5.5	2734 [c]	±4	6163 [b]	±3	7910 [a]	±00
Methyl decanoate	30.3	±5.9	n.d.		n.d.		4.49 [b]	±4.63	6.68 [a]	±0.74
Ethyl decanoate	18,264	±3	n.d.		911 [c]	±4	2446 [b]	±1	2913 [a]	±1
Ethyl 9-decenoate	1176	±2	n.d.		72.7 [c]	±2.7	173 [b]	±2	230 [a]	±1
2-phenethyl acetate	392	±2	9.59 [d]	±4.08	22.6 [c]	±2.4	40.8 [b]	±0.5	55.3 [a]	±1.0
ESTERS	**91,853**		**86.8 [d]**		**5277 [c]**		**12,593 [b]**		**16,521 [a]**	

n.d.: not detected; [a,b,c,d]: different superindexes in the same row indicate significant differences at a 0.05 level according to Student–Newman–Keuls statistical test ($p \leq 0.05$) between CW, AW1, AW2, and AW3.

3.2.1. Varietal Compounds

C_6 compounds encompass the major group of varietal volatile compounds in every sample studied, with the exception of the control wine. In the aromatic distillate, every compound of this group could be identified, though only three compounds could be found in the control dealcoholized wine and in a reduced concentration, which brings to light that most of these compounds were lost in the dealcoholizing process [4,31]. As the amount of the aromatic distillate added to the samples increased, these compounds began to appear, and their concentration increased. However, this did not occur proportionally, as the increase from AW2 and AW3 was not as significant as the growth from AW1 to AW2. 1-hexanol, followed by *(Z)*-3-hexen-1-ol, showed the highest concentrations in all cases, with both imparting herbaceous aromas to the wine [24]. The results of C_6 compounds obtained in the Tempranillo rosé wines prior to dealcoholization (Table S2), which are in concordance with other studies [4], are similar to the concentrations observed in sample AW3, outlining that almost a total recovery of C_6 compounds can be achieved with the addition of an aromatic distillate to dealcoholized wine.

Terpenic compounds are characteristic of aromatic grape varieties, which explains why, in this research, this group of compounds does not represent a majority group, as the Tempranillo variety is considered a neutral variety. However, it is still considered a qualitatively important group as they have a relevant role in the aroma of wines because of their floral attributes and their low odor perception thresholds [24]. Terpenes mostly disappeared during the dealcoholizing process, being only identified in this sample α-terpineol and hydroxycitronellol and in reduced concentrations in CW. These results are in agreement with Osorio et al., 2023 [4]. As the amount of added aromatic distillate increased, the number of identified terpenic compounds in the samples, as well as the total concentration of this group of compounds, also increased. Certain compounds, such as β-terpineol acetate and nerol, were only detected in the aromatic distillate and not in the dealcoholized wine samples, even after adding various volumes of the distillate. This suggests that some compounds are lost during the dealcoholization process and are not recovered with the addition of the aromatic distillate. As expected, terpenes did not represent an important group in the aroma profile of Tempranillo rosé wines, even if dealcoholization did not take place (Table S2), coinciding with the results obtained in the descriptive sensory analysis.

C_{13} norisoprenoids constitute a quantitatively minor group regardless of the sample, with only two compounds detected in all studied wines: TDN and β-damascenone. It can be remarked that the concentration of β-damascenone was higher than 43 µg/L in all studied wines, exceeding its olfactory perception threshold of 0.05 µg/L [24] and indicating its individual contribution to the wine's aroma. The aroma of β-damascenone is characterized by notes of sweet and fruity aromas [24]. The concentration of this compound did not vary significantly between samples, obtaining values quite similar to the aromatic starting distillate, thus indicating that this compound is recovered practically in its entirety after the addition of aromatic distillate to the dealcoholized wines. However, if we compare the obtained concentrations in these samples with the concentration of β-damascenone in the original wine prior to dealcoholization (Table S2), it can be observed that the values are considerably reduced. This points out that β-damascenone can also be lost during the second stage of the SCC technique with the elimination of alcohol. On the other hand, TDN was not found in the control dealcoholized wine but was present in the aromatic distillate. This compound appeared in AW2 and AW3 but at much lower concentrations compared to the aromatic distillate.

Benzene compounds impart sweet, fruity, and spicy aromas to wines [24,32]. Seven compounds, regardless of the sample, were identified, making it the second most quantitatively significant group among the varietal compounds, consistent with the findings of Osorio Alises et al. in 2023 [4]. As observed in Table 2, unlike terpenic compounds, these were not completely lost during the wine dealcoholization process, and their concentration increased independently of the volume of aromatic distillate added. Moreover, no significant differences were found between AW2 and AW3. The most important benzenic compounds in all studied wines were vanillin derivatives, especially homovanillic acid and zingerone, which impart sweet aromas.

Different to what was observed in the case of C_6 compounds, benzene compounds were not totally recovered with the addition of the aromatic distillate to dealcoholized wine, as can be observed in Table S2. This suggests that a part of these compounds can be lost with the elimination of alcohol during the SCC technique, which explains the different ratio between benzene compounds and C_6 compounds in CW and AW1, AW2, and AW3 (Table 2). Notwithstanding all this, similar values of benzene compounds in dealcoholized Tempranillo rosé wines were obtained by other authors [4].

3.2.2. Volatile Compounds Formed Principally During Alcoholic Fermentation

Fermentative aroma compounds are those formed principally during alcoholic fermentation due to the metabolism of yeasts, and their impact on wine aroma may be positive or negative [23]. These compounds were identified in higher concentrations than varietal volatile compounds.

Acids, which constitute a quantitative important group of volatile compounds, may play an important role in the complexity of wine aroma. This study brings to light that they were not affected by the dealcoholizing process in terms of the variety of compounds identified but in terms of their concentration, which was significantly reduced compared to control wine (CW) (Tables 3 and S1). The concentration of acids in the aroma of AW1, AW2, and AW3 gradually increased; however, no significant differences were found between AW2 and AW3. Acids were not completely recovered with the addition of aromatic distillate to dealcoholized wine compared to the original Tempranillo wine prior to the dealcoholization process (Table S2). This was mainly due to the loss of hexanoic, octanoic, and decanoic acids, which were the acids that, although they were found in higher concentrations, were also the compounds that suffered the greatest decrease after the dealcoholizing process. This coincides with the results of Osorio Alises et al. in 2023 [4] for dealcoholized rosé wines of the same variety and Saha et al. in 2013 [33] for wines with reduced or no alcohol content. These same acids were also the major components in the aromatic distillate-added samples, regardless of the amount incorporated.

Regarding alcohols, a total of eleven were identified and quantified, although they were not present in all of the studied samples, as many were undetected in the control wine due to their volatilization during the dealcoholization process. The only alcohols retained in the dealcoholized control wine were 2-phenylethanol, 3-methylthio-1-propanol, isobutanol, and 2,3-butanediol (*levo* and *meso*). This coincides with the predominant identified alcohols in the samples with different concentrations of aromatic distillate added, although the concentrations increased proportionally as more of the aromatic distillate was added. Furthermore, several alcohols were additionally identified in the samples as more of the aromatic distillate was added. As reported in Table 3, no significant differences were found between samples regarding the total concentration of alcohols, though certain compounds (isobutanol, 1-butanol, 1-pentanol, 1-octanol, etc.) presented significant differences between samples.

The concentration of isobutanol, associated with peppery notes in red wines [34], and 3-methylthio-1-propanol and propanol, which impart cooked vegetable notes, is notable, but 2-phenylethanol was the predominant compound in all samples [4], with no significant differences between samples after the addition of different volumes of the aromatic distillate. The latter is associated with rose-like aromas [22], and it is crucial for the wine aroma as it exceeded its olfactory perception threshold (10,000 µg/L) in all studied samples, contributing individually to the wine's aroma [24].

Esters were the predominant group of fermentative aroma compounds, except in the control dealcoholized wine, where a reduced variety and concentration of these compounds were observed. As can be observed in Table S2, even though the aromatic distillate was added to dealcoholized wines, the concentrations of esters were not the same as in the Tempranillo wine prior to dealcoholization. This loss is likely associated with the process of alcohol reduction, during which these esters are also removed [4,16]. A total of twelve compounds were identified in the samples, although not all were present in every sample.

The samples with added aromatic distillate exhibited higher concentrations of esters than the CW, likely due to the formation of complexes between the alcohols and carboxylic acids (Table 3). Significant differences were noted among the samples, as the ester concentrations varied greatly with increased essence dosage.

Ethyl octanoate was the ester identified in higher concentrations in the studied wines, coinciding with Osorio et al. in 2023 [4]. This compound is related to fruity and sweet aromas of wines and exhibited a low olfactory perception threshold (5 μg/L), which was greatly exceeded in AW2 and AW3. Alongside ethyl octanoate, other esters with significant contributions to the wine aroma of AW1, AW2, and AW3 samples included isoamyl acetate, imparting a banana-like note, ethyl hexanoate with a green apple aroma, and ethyl decanoate, presenting fruity and caramel notes [22,24].

3.3. Influence of the Addition of Aromatic Distillate on Odor Activity Values (OAVs)

In Table 4, the odor descriptors, odor thresholds obtained from bibliographic references, and the odor activity values (OAVs) of the 19 volatile compounds with OAV $\geq$ 0.1 in control wine and samples with the addition of different volumes of the aromatic distillate are shown. The compounds with OAV 1 are expected to contribute individually to the aromatic profile of the sample, while those with an OAV $\geq$ 0.1 may have a synergetic effect on the aromatic profile [4].

Only six compounds were found to individually contribute to the aroma across all samples: β-damascenone, ethyl octanoate, hexanoic acid, octanoic acid, isoamyl acetate, and 2-phenylethanol. A noticeable difference in the calculated odor activity value (OAV) for esters, particularly ethyl octanoate, was observed between the control wine and samples with varying volumes of aromatic distillate added. This aligns with previous observations, where ester concentrations significantly decreased during the dealcoholizing process due to their loss in complexes formed with alcohols. Increasing the ester concentration in the samples through distillate addition also raised their OAV.

Conversely, the OAV of β-damascenone remained consistently high across all samples, imparting sweet and fruity notes to the wine's aroma [24]. It can be concluded that as the concentration of added distillate increases, so do the OAVs and the individual aromatic compounds contributing to the aroma of dealcoholized wine. AW3 exhibited a total of thirteen compounds with OAVs greater than 1. In this sample, along AW2, notable esters such as ethyl hexanoate, ethyl butyrate, and ethyl decanoate contributed to fruity and sweet aromas, while terpenes and benzenoid compounds like linalool, geraniol, and 2-phenylethanol provided floral notes. Additionally, high OAVs were recorded for octanoic, hexanoic, and decanoic acids, which are characterized by rancid and fatty notes, potentially considered a sensory defect.

Based on the calculated odor activity values (OAVs) for each sample, the chemical composition of the wine aromas was correlated with their sensory profiles. These compounds were grouped into different aromatic series according to the associated aromatic descriptors (Table 4). For calculating the total intensity of each series, only compounds with an OAV greater than 0.1 were considered [26]. The aromatic series used in this study were fruity, floral, green, sweet, and fatty, excluding series 5 and 7, known as spicy and others, respectively. As can be observed in Table 5, the most important aromatic series identified in the aroma of the studied wines were fruity and sweet, regardless of the percentage of aromatic distillate added. The fatty series exhibited a total intensity ranging between 9 and 21, while the floral and green series showed lower total intensities. Significant differences were found between the total intensity of aromatic series determined in CW and wines with added aromatic distillate, but regardless of the differences between AW1, AW2, and AW3, no significant differences were found among AW2 and AW3.

Table 4. Odor descriptor, aromatic series, odor threshold (µg/L), and odor activity values (OAVs) of free volatile compounds of the control dealcoholized wine (CW) and dealcoholized wines with added aromatic distillate in concentrations of 0.5 (AW1), 1 (AW2), and 1.5 (AW3) % *v*/*v*.

Compound	Aromatic Descriptor	Odor Threshold (µg/L)	Aromatic Series *	OAV CW	OAV AW1	OAV AW2	OAV AW3
β-Damascenone	Sweet, fruity	0.05 [a]	1, 4	862	918	998	960
Ethyl octanoate	Caramel, fruity	5.00 [c]	1, 4	5.05	546	1232	1582
Ethyl hexanoate	Green apple	14.0 [c]	1	0.00	65.4	160	237
Isoamyl acetate	Banana	30.0 [c]	1	1.61	14.9	29.2	41.5
Ethyl butyrate	Fruity	20.0 [c]	1	0.00	5.94	26.3	31.9
Ethyl decanoate	Caramel, fruity	200 [c]	1, 4	0.00	4.56	12.23	14.6
Octanoic acid	Sweat, cheese	500 [c]	6	3.63	6.41	9.40	9.60
Hexanoic acid	Sweat	420 [b]	6	4.70	6.19	7.85	7.74
Decanoic acid	Rancid fat	1000 [b]	6	0.16	0.91	1.64	2.11
2-Phenylethanol	Floral, rose	10,000 [a]	2	1.24	1.25	1.34	1.32
(*Z*)-3-hexen-1-ol	Green, cut grass	400 [c]	3	0.06	0.67	1.23	1.30
Geraniol	Rose, geranium	30.0 [a]	2	0.00	0.00	0.00	1.13
Linalool	Floral	15.0 [a]	2	0.00	0.35	0.67	1.08
Isovaleric acid	Acid, rancid	33.0 [c]	4, 6	0.00	0.26	0.32	0.96
1-hexanol	Green	8000 [c]	2, 3	0.01	0.18	0.36	0.46
Butyric acid	Rancid, cheese	173 [c]	6	0.28	0.29	0.37	0.39
β-citronellol	Floral	40.0 [a]	2	0.00	0.21	0.24	0.30
2-phenethyl acetate	Floral	250 [a]	2	0.04	0.09	0.16	0.22
3-metilthio-1-propanol	Cooked vegetables	1000 [a]	6	0.20	0.18	0.19	0.19

[a] Guth, 1997 [24]. [b] Etiévant, 1991 [22]. [c] Ferreira et al., 2000 [23]. * 1: fruity; 2: floral; 3: green; 4: sweet; 6: fatty.

Table 5. Intensity of aromatic series calculated with the summatory of mean OAVs of each analyzed sample ± standard deviation. CW: control dealcoholized wine; AW1: dealcoholized wine with added aromatic distillate in concentration of 0.5% *v*/*v*; AW2: dealcoholized wine with added aromatic distillate in concentration of 1% *v*/*v*; AW3: dealcoholized wine with added aromatic distillate in concentration of 1.5% *v*/*v*.

		CW	AW1	AW2	AW3
1	Fruity	869 [c] ± 1	1556 [b] ± 4	2659 [a] ± 6	2867 [a] ± 7
4	Sweet	867 [c] ± 1	1470 [b] ± 3	2444 [a] ± 6	2558 [a] ± 7
6	Fatty	8.97 [c] ± 0.25	14.2 [b] ± 2.3	19.9 [a] ± 1.1	21.0 [a] ± 1.3
2	Floral	1.24 [d] ± 0.20	1.99 [c] ± 0.47	2.77 [b] ± 0.53	4.51 [a] ± 0.65
3	Green	0.00	0.85 [b] ± 0.21	1.59 [a] ± 0.14	1.85 [a] ± 0.26

[a,b,c,d]: different superindexes in the same row indicate significant differences at a 0.05 level according to Student–Newman–Keuls statistical test ($p \leq 0.05$) between CW, AW1, AW2, and AW3.

3.4. Influence of the Addition of Aromatic Distillate on Aroma Sensory Profile

In order to research the influence of the addition of different quantities of the aromatic distillate on the sensory profile of dealcoholized Tempranillo rosé wines, a descriptive sensory analysis was performed with the control wine and wines with the aromatic distillate added in a proportion of 0.5, 1.0, and 1.5% *v*/*v* (AW1, AW2, and AW3, respectively) to one liter of dealcoholized Tempranillo rosé. Table 6 shows the average wine aroma intensity attributes scores and the standard deviation of the wines studied. According to the results, the aroma profile of the dealcoholized Tempranillo rosé wines was characterized by raspberry, strawberry, green, fresh, and fresh fruit attributes, with sweet and citric notes. As can be seen, the dealcoholization process reduced the intensity of the most important attributes of the traditional Tempranillo rosé wine (Table S3) in agreement with investigations about dealcoholized wine [16]. The addition of dealcoholized distillate independent of the quantity added enhanced the intensity of the principal attributes of the dealcoholized wines. Nevertheless, the addition of 1.0 and 1.5% *v*/*v* (AW2 and AW3, respectively) of the aromatic distillate to one liter of dealcoholized Tempranillo rosé produced a significant change in the aroma intensity attributes between the control and AW1 wines, which was especially remarkable in the raspberry, strawberry, sweet, green, fresh, and fresh fruit aroma attributes. These results are in agreement with the analytical results that show a higher concentration of esters, β-damascenone, benzenic, and C_6 compounds related to fruity, sweet, and fresh/green aromas.

Table 6. Principal sensory attributes obtained by descriptive sensory analysis. Mean intensity ± standard deviation (n = 2). CW: control dealcoholized wine; AW1: dealcoholized wine with added aromatic distillate in concentration of 0.5% *v*/*v*; AW2: dealcoholized wine with added aromatic distillate in concentration of 1% *v*/*v*; AW3: dealcoholized wine with added aromatic distillate in concentration of 1.5% *v*/*v*.

	CW		**AW1**		**AW2**		**AW3**	
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**
Strawberry	5.64^{a}	±0.43	5.84^{a}	±0.33	7.64^{b}	±0.43	7.79^{b}	±0.53
Raspberry	5.23^{a}	±0.12	5.93^{a}	±0.42	6.23^{b}	±0.12	6.54^{b}	±0.32
Fresh Fruit	4.49^{a}	±0.23	5.00^{a}	±0.13	5.49^{b}	±0.23	5.38^{b}	±0.53
Green/Fresh	4.63^{a}	±0.52	5.63^{b}	±0.52	6.63^{c}	±0.72	7.63^{d}	±0.42
Sweet	3.64^{a}	±0.63	3.94^{a}	±0.46	4.74^{b}	±0.24	4.97^{b}	±0.63
Citric	2.60^{a}	±0.25	2.57^{a}	±0.15	3.04^{b}	±0.45	3.15^{b}	±0.75
Floral	n.d.		n.d.		n.d.		n.d.	

^{a,b,c,d}: different superindexes in the same row indicate significant differences at a 0.05 level according to Student–Newman–Keuls statistical test ($p \leq 0.05$) between CW, AW1, AW2, and AW3. n.d.: not detected.

4. Conclusions

The addition of the aromatic distillate in assayed quantities to the dealcoholized Tempranillo rosé wine improves the aroma of wines. The addition of the distillate increased the concentration of various volatile compounds, including C_6 compounds, terpenic compounds, benzene compounds, and esters, especially in AW3 wines. The samples with higher distillate concentrations also exhibited higher OAVs for compounds associated with fruity and sweet aromas, and the total intensity of these aromatic series increased. While the addition of the aromatic distillate improved the aroma profile, it also led to an increase in alcohol content, exceeding the legal limit for dealcoholized wines in AW3. Therefore, the resulting AW3 wine would be classified as wine with a reduced ethanol content rather than a dealcoholized wine and exhibits the aroma typicity of La Mancha Tempranillo grape variety. In this sense, according to the results, the addition of 1% *v*/*v* of the aromatic distillate permits the enhancement of the aromatic typicity of La Mancha Tempranillo rosé wines, and they can be classified as dealcoholized wines as per the regulations.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/beverages10040123/s1, Table S1: Retention index (RI) and source of the identified free volatile compounds; Table S2: Mean concentration (μg/L) and relative standard deviation (n = 2) of free volatile compounds identified in aromatic distillate (AD), control dealcoholized wine (CW), and dealcoholized wines added with aromatic distillate in concentration of 0.5 (AW1), 1 (AW2) and 1.5 (AW3) % v/v. and Table S3: Mean concentration (µg/L) and relative standard deviation (n = 2) of volatile compounds formed during alcoholic fermentation identified in aromatic distillate (AD), control dealcoholized wine (CW), and dealcoholized wines added with aromatic distillate in concentration of 5 (AW1), 10 (AW2) and 15 (AW3) % v/v.

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