

Article

Comprehensive Analysis of Teran Red Wine Aroma and Sensory Profiles: Impacts of Maceration Duration, Pre-Fermentation Heating Treatment, and Barrel Aging

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Featured Application: This study provides a comprehensive analysis of the impact of various vinification techniques, including prolonged maceration, pre-fermentation heating, and barrel aging, on the volatile aroma profile and sensory characteristics of Teran red wine. Teran (*Vitis vinifera* L.) is the most widespread red autochthonous variety in Istria, traditionally grown in the north Adriatic area, including the Croatian Istria viticultural subregion. As a key grapevine variety in viticulture across these regions, understanding the impact of the investigated techniques is crucial for enhancing its unique characteristics and market value. These insights can be directly applied by winemakers to optimize and tailor wine production processes, enhancing desirable aroma attributes and sensory qualities to meet specific market demands. The findings offer practical guidelines for producing wines with distinct and appealing aromatic profiles, aiding in product differentiation and improving overall wine quality.



Citation: Rossi, S.; Bestulić, E.; Orbanic, F.; Horvat, I.; Lukić, I.; Ilak Peršurić, A.S.; Bubola, M.; Plavša, T.; Radeka, S. Comprehensive Analysis of Teran Red Wine Aroma and Sensory Profiles: Impacts of Maceration Duration, Pre-Fermentation Heating Treatment, and Barrel Aging. *Appl. Sci.* **2024**, *14*, 8729. <https://doi.org/10.3390/app14198729>

Academic Editors: Anita Pichler and Ivana Ivic

Received: 27 August 2024

Revised: 20 September 2024

Accepted: 25 September 2024

Published: 27 September 2024



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Abstract: This study investigates the effect of prolonged maceration, pre-fermentation heating, and barrel aging on the volatile aroma profile and sensory characteristics of Teran wine. The vinification processes included a control treatment (7-day maceration, TM7-Y; Y—young wine), 10-day maceration (TM10-Y), 21-day post-fermentation maceration (TM21-Y), and 48-h pre-fermentation heating at 45 °C followed by 8-day maceration (TPHT-Y). All wines were then aged in oak barrels for six months, resulting in TM7-A, TM10-A, TM21-A, and TPHT-A wines (A—aged wine). Volatile compounds were extracted using headspace solid-phase microextraction (HS-SPME) and analyzed by gas chromatography/mass spectrometry (GC/MS), while sensory profiles were evaluated using quantitative descriptive analysis (QDA). TPHT-Y and TM21-Y treatments reduced several groups of free volatile compounds while enhancing sensory properties, with TM21-Y wines notably exhibiting pronounced dried fruit notes, likely due to high β -damascenone concentrations. Conversely, TM10-Y and TM7-Y treatments resulted in significantly higher concentrations of the most volatile aroma compounds. Aging in oak barrels significantly increased the levels of particular free volatile compounds like C₁₃-norisoprenoids, volatile phenols, furans, and lactones. It also enhanced sensory quality, with fruity aromas prominent across all treatments, and TM21-A and TPHT-A wines showing strong dried fruit, jam, and liqueur notes. This study offers valuable insights into tailoring wine aromas and sensory attributes through specific vinification techniques, contributing to a more refined approach to optimizing wine production. In conclusion, the findings highlight the importance of maceration and aging techniques in developing complex and desirable wine profiles, offering practical guidance for improving Teran wine quality.

Keywords: Teran red wine; vinification techniques; skin contact; oak wood; volatile aroma compounds; HS-SPME-GC-MS; QDA sensory analysis

1. Introduction

The aroma of wine results from a complex interplay of volatile compounds that significantly influence its overall quality and sensory appeal [1]. These compounds originate from various sources, including grapes, yeast metabolism during fermentation, and transformations during maturation [2]. While several hundred aromatic compounds have been identified in wine [3], only a select few exert a profound impact on its final flavor by integrating perceptions across olfactory, gustatory, and tactile senses [4]. Certain volatile compounds significantly influence the aroma depending on their concentration relative to the olfactory sensitivity threshold [5,6].

Aromatic compounds in wine are broadly classified into varietal (primary), fermentation (secondary), and aging (tertiary) aromas [6]. Varietal aromas, derived directly from grapes, are influenced by factors such as soil, climate, and grape maturity, with key compounds including terpenes, norisoprenoids, methoxypyrazines, and thiols [7,8]. Fermentation aromas arise during grape processing and must fermentation and include mainly higher alcohols, fatty acids, esters, and aldehydes [1,9]. Aging aromas develop during wine aging through chemical reactions, influenced by barrel wood and oxygen diffusion, with compounds like lactones, furans, and volatile phenols contributing to complex aromas [4,10,11]. Understanding the chemical composition and sensory contribution of these compounds is crucial for optimizing vinification approaches [4]. Sensory analysis, including quantitative descriptive analysis, is crucial for objectively evaluating wine aroma profiles [4]. By correlating the results of chemical analyses and sensory attributes, it is possible to precisely characterize the aroma of a particular wine.

Vinification procedures, such as maceration, are fundamental in extracting aromatic compounds from grape skins and seeds, thereby enhancing wine's aromatic complexity [7,12]. The duration and temperature of maceration significantly affect the extraction of aroma compounds, with longer maceration periods and higher temperatures generally leading to a richer and more complex aromatic profile. During maceration, the transfer of free and bound aroma compounds from the grape skins into the must is enhanced, allowing for a more complete utilization of the varietal aroma potential present in grapes [13]. Additionally, the maturation of wine in wooden barrels introduces new aromatic complexities, as volatile compounds from the oak, such as lactones, are extracted into the wine, further enriching its aromatic profile [10]. Recent market trends favor intensely colored red wines with pronounced fruit characteristics [14]. Effective vinification technologies are sought to produce wines with desired sensory attributes, making the study of wine composition, particularly aromatic compounds, crucial for understanding sensory properties and improving wine quality. Identifying compounds impacting aroma remains a key challenge in wine research [4].

The present research investigates the impact of pre-fermentation heat treatment, extended maceration durations, and barrel aging on volatile aroma compound concentrations and the sensory profile of Teran red wines. Teran (*Vitis vinifera* L.) is the most widespread red autochthonous variety in Istria [15], traditionally grown in the north Adriatic area, including the Croatian Istria viticultural subregion [16]. Young Teran wines typically exhibit an intense red color with a purple glow, moderate bitterness and astringency, and a full, rich body, and a pleasant aroma with a characteristic note reminiscent of marasca cherry, alongside high alcoholic strength and higher total acidity [17–20]. Teran wines are also noted for their aging potential, which enhances their complexity and depth. Despite its significance, the scientific literature detailing the specific aromatic profile of Teran wines as well as its response to standard and innovative vinification treatments remains rather limited [19], and comprehensive scientific studies are needed. This especially refers to volatile compounds that constitute its primary aroma, which is expected to be the most affected by the treatments investigated in this study.

The results of this study aim to contribute to existing research by systematically examining the effects of pre-fermentation heat treatment, prolonged maceration techniques, and barrel aging on Teran red wines. Gas chromatography–mass spectrometry (GC–MS) was used to analyze volatile compounds, complemented by quantitative descriptive sensory analysis. The research provides insights into how these factors affect wine quality and offers practical guidance for improving Teran wine production.

2. Materials and Methods

2.1. Chemicals and Reagents

Pure standards of volatile aroma compounds were purchased from Merck (Darmstadt, Germany), Sigma-Aldrich (St. Louis, MO, USA), and Fluka (Buchs, Switzerland). Working standard solutions were prepared by dilution of stock standard solutions in synthetic wine containing 12% of ethanol, 5 g/L of tartaric acid, 50 mg/L of each acetaldehyde, methanol, ethyl acetate, 1-propanol and isobutanol, and 150 mg/L of isoamyl alcohol. Working solutions were adjusted to pH = 3.2 with 0.1 M NaOH.

2.2. Grapes and Winemaking

The experiment was conducted in 2018 with a red grapevine cultivar, Teran (*Vitis vinifera* L.). Grape ripeness was assessed using standard chemical analyses, revealing average harvest metrics of 21.6° Brix, 8.3 g/L total acidity (expressed as tartaric acid), and a pH of 3.20. Grapes were manually harvested on 3 October 2018, from an experimental vineyard at the Institute of Agriculture and Tourism in Poreč, Istria, Croatia. Upon harvesting, the grapes were promptly destemmed and crushed, then evenly transferred to 110-L stainless steel vats located in the experimental “Minivinification” wine cellar of the Institute. To initiate winemaking, the crushed grapes received treatments of potassium metabisulfite (5 g/hL, AEB SPA Brescia, Italy) and Aromax (5 g/hL, AEB SPA). Additionally, the pectolytic enzyme Endozym Rouge (4 g/hL, AEB SPA), a mixture of several enzymes including pectinlyase, pectinesterase, polygalacturonase, and cellulase, was added to all treatments. While this enzyme may have some influence on the aroma profile, its consistent application across all treatments means its effect was uniform and thus not a focus of this study. The grape musts were inoculated with selected dry yeast *Saccharomyces cerevisiae* Fermol Méditerranée (30 g/hL, AEB SPA) rehydrated with Fermol Plus Starter (10 g/hL, AEB SPA Brescia, Italy). Fermol Plus H₂S Free (10 g/hL, AEB SPA) was supplemented on the second and fifth days of fermentation. Four distinct vinification protocols were implemented: a control treatment with 7 days of maceration—TM7 (Y), prolonged 10-day maceration—TM10 (Y), extended 21-day post-fermentation maceration—TM21 (Y), and a 48-h pre-fermentation maceration heating at 45 °C followed by 8 days of maceration—TPHT (Y). The “Y” denotes young wines. In the control (TM7-Y), prolonged maceration (TM10-Y), and extended maceration (TM21-Y) treatments, maceration lasted for 7 days, 10 days, and 21 days, respectively. Yeast (*Saccharomyces cerevisiae* Fermol Méditerranée) was added on the first day in all three treatments, so fermentation and maceration began simultaneously. In all of the treatments, fermentation lasted for 7 days. There was no pre-fermentation maceration (cold soaking); instead, fermentation started immediately with maceration. In the TM21-Y treatment, the 21-day maceration process included both fermentation and post-fermentation maceration, with the lees remaining in the tanks until racking, which was performed at the end of maceration. In the TPHT-Y treatment, the pomace was heated to 45 °C for 48 h as part of a pre-fermentation maceration process. After this heating period, the pomace was cooled to 24 °C, and yeast (*Saccharomyces cerevisiae* Fermol Méditerranée) was added. Following yeast addition, fermentation, which lasted for 7 days as in the other treatments, occurred simultaneously with maceration for an additional 8 days, bringing the total maceration duration to 10 days.

Each treatment was replicated three times, with fermentations maintained at 24 °C and the cap punched down three times daily during maceration. After complete maceration, the wines underwent pressing using a closed-type pneumatic press (Letina Inox d.o.o., Čakovec, Croatia) set at pressures of 3×0.3 bars and 1×0.5 bars. Once alcoholic fermentation reached completion (reducing sugars <2 g/L), the wines were stored under cellar conditions (12–15 °C) for 3 months. Subsequently, they were racked and sampled into 0.75-L bottles for analysis. Throughout the process, levels of free and bound SO₂ were closely monitored and adjusted as necessary. Following the second rack, the young wines from each treatment were aged for six months in 100-L oak barrels (LT-light toasted) (Bačvarija Golub, Jastrebarsko, Croatia) to evaluate the maturation differences between young and aged Teran wines produced via different vinification techniques. Malolactic fermentation was not actively controlled during the winemaking processes. The aged wines from each treatment, TM7 (A), TM10 (A), TM21 (A), and TPHT (A), where “A” indicates aged wines (six months in oak barrels), were sulfited with potassium metabisulfite (5 g/hL) and bottled. Approximately three months post-bottling, wines underwent standard physico-chemical analysis, volatile aromatic compound analysis, and sensory evaluation. The aged wines were assessed after this period to account for their additional six-month maturation in oak barrels. The results of the standard physico-chemical analysis of the wine have been previously published in our earlier work [15].

2.3. Analysis of Free Volatile Aroma Compounds

Volatile aroma compounds were isolated from wine samples using solid-phase microextraction (SPME) with a divinylbenzene-carboxen-polydimethylsiloxane (DVB-CAR-PDMS) fiber. The method was detailed in previous research [21]. A 4 mL wine sample was prepared in a 10 mL glass vial. The sample was diluted fourfold with the addition of 1 g of (NH₄)₂SO₄ (Kemika d.d., Zagreb, Croatia) and 50 µL of internal standard solution (2-octanol at a concentration of 0.84 mg/L of wine for monoterpenes, C₁₃-norisoprenoids, and alcohols; methyl nonanoate at a concentration of 0.82 mg/L of wine for esters; and heptanoic acid at a concentration of 2.57 mg/L for fatty acids). After conditioning for 15 min at 40 °C, headspace extraction was performed using a DVB-CAR-PMS fiber (Stable-Flex, 50/30 µm, 1 cm; Supelco, Bellefonte, PA, USA) at the same temperature for 40 min with continuous stirring using a magnetic stirrer (800 rpm). Following extraction, the SPME fiber was placed in the gas chromatograph injector for the desorption of volatile compounds at 248 °C for 5 min (3 min splitless mode). Identification and quantification were conducted using a gas chromatograph–mass spectrometer (GC–MS) system consisting of a Varian 3900 gas chromatograph coupled to a Varian Saturn 2100 T mass spectrometer with an ion trap analyzer (Varian Inc., Harbor City, CA, USA) and the associated Star Chromatography Workstation software, version 6.6 (Varian, Inc.). A capillary Rtx-WAX column (60 m \times 0.25 mm i.d. \times 0.25 µm d.f.) (Restek, Bellefonte, PA, USA) was used. The oven temperature program was as follows: the initial temperature was set at 40 °C, then increased by 2 °C per minute to 240 °C, where it was held for 10 min. The injector temperature was set to 245 °C, the transfer line to 80 °C, and the ion trap to 120 °C. Mass spectra were obtained by electron ionization (EI, 70 eV) with 1 s/scan, and the detection range was set for ions with a mass-to-charge ratio of 30–450. Helium was used as the carrier gas with a flow rate of 1.2 mL/min. Calibration curves were prepared based on the analysis of standard solutions. Linear retention indices (relative to retention indices of n-alkanes from C10 to C28) were calculated and compared with the literature values. Compound identification was performed by comparing the retention times and mass spectra with those of pure standards when available, and with mass spectra from the NIST05 spectral library. For other compounds, semi-quantitative determination was conducted, and concentrations were expressed as equivalents of compounds with similar chemical structures for which standards were available, assuming a response factor of 1.

2.4. Sensory Analysis

To obtain a quality evaluation and comprehensive wine aroma assessment three months after bottling, a sensory analysis of the wines was conducted by the accredited sensory panel of the Institute of Agriculture and Tourism, as previously described [15,22]. The panel was composed of five trained wine tasters, members of the Croatian Viticultural and Enological Society, certified and authorized by the Croatian Ministry of Agriculture for official commercial wine sensory analysis for placing wines on the Croatian market. They were specially trained in Teran wine assessment. The sensory panel is accredited according to the EN ISO/IEC 17025:2017 standard, “General requirements for the competence of testing and calibration laboratories” [23] for organoleptic (sensory) testing of wines using the method prescribed by the Ordinance on Wine and Fruit Wine Sensory Testing “Official Gazette” [24] N.N. 106/04, with all amendments concluding with N.N. 1/15, which was valid at the time when investigation was performed. The sensory analysis was conducted on 24 wine samples using the quantitative descriptive analysis (QDA) method. This included 4 young wines, each with 3 replicates, and 4 aged wines, each with 3 replicates, analyzed separately. The young wines were evaluated first, followed by the aged wines after six months of maturation. The wine assessment took place at the Institute of Agriculture and Tourism, in a room constructed in accordance with the ISO standard [25]. Standard 200 mL wine-tasting glasses [26] were used, with 50 mL of sample appropriately cooled at 18 °C, poured according to a randomized and coded schedule assigned by the head of the panel. At the beginning of the sensory analysis, the tasters aligned their criteria by tasting two Teran wine samples. Quantitative descriptive analysis (QDA) was applied using a tasting sheet for red wines comprising 57 aroma attributes (descriptors) arranged into 11 groups and the intensities of the particular attributes and groups were evaluated using a 10-point structured scale (0 = attribute not perceptible, 10 = attribute strongly perceptible). The aroma attributes (descriptors) were sorted into groups as follows: floral aroma group (violet, rose, lemon balm, lilac); fruit aroma group (raspberry, blackberry, strawberry, black currant, red currant, cherry, sour cherry, gooseberry, plum, blueberry); dried fruit aroma group (raisin, fig, sour cherry, cherry, plum); jammy aroma group (cherry, sour cherry, plum, fig); nutty aroma group (walnut, hazelnut, almond, bitter almond); herbal aroma group (grass, hay, tea, tobacco, dry leaf); spicy and aromatic herb aroma group (clove, laurel, mint, pepper, anise, fennel, oregano, wormwood); methoxypyrazine-derived group (green pepper, tomato leaf); liqueur aroma group (cherry, sour cherry, chocolate); toasted aroma group (caramel, chocolate, toasted bread, toasted walnut, toasted almond, toasted hazelnut, coffee); and several unsorted descriptors (honey, wax, butter, milk, carob).

2.5. Statistical Data Analysis

All experiments were performed in triplicate and mean values were used in further data analysis. To determine the statistical difference between treatments, one-way analysis of variance (ANOVA) and Fischer’s least significant difference test (LSD) were used to compare the mean values (at the level of significance of $p < 0.05$.) of volatile aroma compounds concentrations and the sensory attribute scores of analyzed wines. Statistical analysis was performed using Statistica v.13.2 software (Stat-Soft Inc., Tulsa, OK, USA).

3. Results and Discussion

3.1. Evaluation of Volatile Aroma Compounds

Table 1 summarizes the results of the analysis of volatile aroma compounds in Teran wines. The study identified 58 volatile aroma compounds, categorized into monoterpenes, C₁₃-norisoprenoids, alcohols, fatty acids, ethyl esters, acetate esters, other esters, volatile phenols, benzenoids, furans, and lactones.

Table 1. Concentration ($\mu\text{g/L}$) of volatile aroma compounds (means \pm standard deviations) in Teran wines produced by different vinification treatments.

Volatile Compounds	Young/Aged Wine	Treatments			
		TM7	TM10	TM21	TPHT
Monoterpenes					
Limonene	Y	1.03 \pm 0.12 ^{a*}	0.98 \pm 0.04 ^{a*}	0.23 \pm 0.02 ^c	0.37 \pm 0.06 ^{b*}
	A	0.45 \pm 0.05 ^a	0.18 \pm 0.02 ^b	0.62 \pm 0.14 ^{a*}	0.17 \pm 0.10 ^b
β -pinene	Y	2.17 \pm 0.24 ^{a*}	2.17 \pm 0.16 ^{a*}	0.42 \pm 0.03 ^b	0.34 \pm 0.04 ^{b*}
	A	0.62 \pm 0.03 ^b	0.26 \pm 0.03 ^c	1.37 \pm 0.20 ^{a*}	0.11 \pm 0.07 ^c
Linalool	Y	25.28 \pm 1.18 ^{a*}	24.01 \pm 0.95 ^{a*}	5.22 \pm 0.76 ^b	4.94 \pm 0.73 ^b
	A	18.24 \pm 0.45 ^a	3.56 \pm 0.25 ^c	14.08 \pm 1.04 ^{b*}	14.09 \pm 0.62 ^{b*}
4-Terpineol	Y	5.07 \pm 0.18 ^{a*}	4.36 \pm 0.8 ^a	0.97 \pm 0.11 ^b	1.07 \pm 0.17 ^b
	A	4.16 \pm 0.10 ^a	4.40 \pm 0.17 ^a	2.84 \pm 0.38 ^{b*}	1.34 \pm 0.15 ^c
α -Terpineol	Y	28.32 \pm 2.66 ^{a*}	28.15 \pm 2.46 ^{a*}	4.64 \pm 0.42 ^b	6.10 \pm 0.39 ^b
	A	8.88 \pm 0.98 ^b	9.12 \pm 0.52 ^b	20.41 \pm 1.41 ^{a*}	9.86 \pm 1.08 ^{b*}
Citronellol	Y	16.02 \pm 0.76 ^{a*}	13.96 \pm 1.45 ^{b*}	7.07 \pm 0.46 ^c	4.99 \pm 0.60 ^{d*}
	A	7.70 \pm 0.10 ^a	3.24 \pm 0.08 ^b	7.64 \pm 0.76 ^a	3.02 \pm 0.13 ^b
Geraniol	Y	3.47 \pm 0.33 ^a	1.62 \pm 0.26 ^c	2.59 \pm 0.28 ^b	1.58 \pm 0.33 ^c
	A	3.49 \pm 0.23 ^b	2.12 \pm 0.18 ^c	7.46 \pm 0.77 ^{a*}	1.64 \pm 0.43 ^c
Geranyl acetone	Y	6.80 \pm 0.51 ^{a*}	4.58 \pm 0.49 ^{b*}	0.99 \pm 0.19 ^c	1.07 \pm 0.15 ^{c*}
	A	0.98 \pm 0.14 ^b	0.80 \pm 0.18 ^b	2.44 \pm 0.48 ^{a*}	0.56 \pm 0.17 ^b
<i>trans</i> -Nerolidol	Y	2.23 \pm 0.17 ^{a*}	1.42 \pm 0.09 ^{b*}	0.41 \pm 0.06 ^c	0.48 \pm 0.10 ^{c*}
	A	0.54 \pm 0.05 ^b	0.45 \pm 0.03 ^b	1.37 \pm 0.27 ^{a*}	0.12 \pm 0.09 ^c
Eucalyptol	Y	1.65 \pm 0.16 ^{a*}	1.12 \pm 0.05 ^{b*}	0.19 \pm 0.03 ^d	0.52 \pm 0.07 ^{c*}
	A	0.90 \pm 0.02 ^a	0.40 \pm 0.04 ^c	0.74 \pm 0.01 ^{b*}	0.13 \pm 0.10 ^d
Menthol	Y	62.96 \pm 3.86 ^{a*}	41.79 \pm 2.11 ^{b*}	8.37 \pm 0.73 ^c	10.68 \pm 1.73 ^{c*}
	A	11.55 \pm 1.28 ^b	8.65 \pm 1.06 ^b	23.89 \pm 3.05 ^{a*}	2.20 \pm 1.63 ^c
<i>trans</i> -Rose oxide	Y	4.05 \pm 0.70 ^{a*}	3.98 \pm 0.76 ^{a*}	0.57 \pm 0.07 ^b	0.87 \pm 0.14 ^{b*}
	A	2.28 \pm 0.11 ^a	0.81 \pm 0.07 ^c	1.04 \pm 0.07 ^{b*}	0.19 \pm 0.12 ^d
Total monoterpenes	Y	159.1 \pm 9.3 ^{a*}	128.2 \pm 6.4 ^{b*}	31.68 \pm 2.70 ^c	33.01 \pm 4.24 ^c
	A	59.80 \pm 1.60 ^b	34.00 \pm 2.29 ^c	83.89 \pm 7.96 ^{a*}	33.43 \pm 3.08 ^c
C ₁₃ -norisoprenoids					
Vitispirane I	Y	8.43 \pm 0.42 ^{a*}	6.12 \pm 0.45 ^{b*}	1.96 \pm 0.15 ^d	2.59 \pm 0.16 ^{c*}
	A	2.30 \pm 0.23 ^b	0.96 \pm 0.07 ^c	3.74 \pm 1.12 ^a	0.39 \pm 0.25 ^c
Vitispirane II	Y	2.98 \pm 0.58 ^{b*}	4.32 \pm 0.18 ^{a*}	1.25 \pm 0.19 ^c	1.49 \pm 0.12 ^{c*}
	A	0.05 \pm 0.01 ^b	0.09 \pm 0.07 ^b	1.86 \pm 1.46 ^a	0.24 \pm 0.17 ^b
Actinidol ethyl ether I	Y	n.d.	n.d.	n.d.	n.d.
	A	80.49 \pm 0.96 ^a	77.05 \pm 4.35 ^{ab}	68.80 \pm 5.68 ^b	55.32 \pm 7.51 ^c
Actinidol ethyl ether II	Y	n.d.	n.d.	n.d.	n.d.
	A	47.56 \pm 0.33 ^a	45.68 \pm 2.36 ^a	41.78 \pm 4.05 ^a	31.90 \pm 5.16 ^b
TDN	Y	n.d.	n.d.	n.d.	n.d.
	A	1.42 \pm 0.03 ^b	1.41 \pm 0.08 ^b	1.85 \pm 0.29 ^a	1.15 \pm 0.21 ^b
β - Damascenone	Y	8.33 \pm 0.86 ^{b*}	8.31 \pm 0.81 ^{b*}	10.27 \pm 0.12 ^{a*}	1.65 \pm 0.35 ^{c*}
	A	1.66 \pm 0.13 ^b	1.41 \pm 0.15 ^b	3.73 \pm 0.48 ^a	0.45 \pm 0.33 ^c
β - Ionone	Y	4.69 \pm 1.08 ^{a*}	3.00 \pm 0.60 ^{b*}	0.47 \pm 0.13 ^c	0.63 \pm 0.07 ^c
	A	0.84 \pm 0.07 ^b	0.58 \pm 0.11 ^{bc}	1.72 \pm 0.24 ^{a*}	0.31 \pm 0.22 ^c
TPB	Y	n.d.	n.d.	n.d.	n.d.
	A	1.27 \pm 0.08 ^b	2.39 \pm 0.13 ^a	2.83 \pm 0.41 ^a	1.69 \pm 0.22 ^b
Actinidol I	Y	n.d.	n.d.	n.d.	n.d.
	A	14.21 \pm 0.25	16.7 \pm 1.42	18.65 \pm 2.68	16.72 \pm 3.71
Actinidol II	Y	n.d.	n.d.	n.d.	n.d.
	A	27.00 \pm 0.46	25.1 \pm 1.78	27.99 \pm 4.06	24.29 \pm 4.85
Total C ₁₃ -norisoprenoides	Y	24.43 \pm 1.93 ^a	21.75 \pm 0.91 ^b	13.95 \pm 0.47 ^c	6.35 \pm 0.61 ^d
	A	176.8 \pm 1.4 ^{a*}	171.4 \pm 10.0 ^{a*}	172.9 \pm 18.3 ^{a*}	132.5 \pm 22.4 ^{b*}

Table 1. Cont.

Volatile Compounds	Young/Aged Wine	Treatments			
		TM7	TM10	TM21	TPHT
Alcohols					
1-Hexanol	Y	4245 ± 149 ^{b*}	4593 ± 292 ^{a*}	1372 ± 87 ^c	1473 ± 79 ^c
	A	1578 ± 38 ^b	1617 ± 18 ^b	2211 ± 100 ^{a*}	1236 ± 43 ^c
<i>trans</i> -3-Hexen-1-ol	Y	43.67 ± 2.89 ^{b*}	52.16 ± 2.05 ^{a*}	17.55 ± 1.83 ^c	16.29 ± 1.13 ^c
	A	11.29 ± 4.71 ^c	19.18 ± 0.61 ^b	24.69 ± 2.43 ^a	16.9 ± 0.33 ^b
<i>cis</i> -3-Hexen-1-ol	Y	97.88 ± 3.45 ^b	102.7 ± 1.80 ^{a*}	25.09 ± 2.38 ^c	26.26 ± 1.18 ^c
	A	14.98 ± 0.37 ^b	23.59 ± 2.21 ^b	45.23 ± 8.06 ^a	24.99 ± 5.83 ^b
Benzyl alcohol	Y	2.03 ± 0.49 ^a	1.53 ± 0.15 ^b	1.34 ± 0.03 ^{bc}	1.02 ± 0.04 ^c
	A	2.05 ± 0.07 ^a	2.01 ± 0.09 ^{ab*}	1.80 ± 0.12 ^{bc*}	1.69 ± 0.19 ^{c*}
2-Phenylethyl Alcohol	Y	152,349 ± 5115 ^{a*}	131,289 ± 6406 ^{b*}	70,524 ± 5296 ^c	67,290 ± 2106 ^c
	A	122,741 ± 2219 ^a	60,919 ± 2600 ^c	122,662 ± 8255 ^{a*}	73,310 ± 1367 ^{b*}
Total alcohols	Y	156,739 ± 5051 ^{a*}	136,039 ± 6350 ^{b*}	71,940 ± 5385 ^c	68,807 ± 2119.28 ^c
	A	124,347 ± 2222 ^a	62,581 ± 2607 ^c	124,945 ± 8161 ^{a*}	74,590 ± 1326 ^{b*}
Fatty acids					
Butanoic acid	Y	1544 ± 77 ^a	1642 ± 97 ^a	702.7 ± 86.4 ^b	741.9 ± 46.6 ^b
	A	1663 ± 25 ^a	1655 ± 57 ^a	1072 ± 54 ^{b*}	655.3 ± 51 ^c
Hexanoic acid	Y	1022 ± 96 ^{ab}	1100 ± 147 ^a	862.2 ± 46.9 ^{bc}	746.2 ± 62.1 ^c
	A	1200 ± 11 ^{b*}	1092 ± 17 ^b	1399 ± 72 ^{a*}	941.0 ± 95.6 ^{c*}
Octanoic Acid	Y	385.6 ± 53.2 ^a	272.5 ± 60.0 ^b	386.7 ± 35.1 ^a	402.8 ± 29.2 ^{a*}
	A	329.4 ± 18.7 ^b	260.2 ± 22.4 ^b	471.4 ± 71.7 ^{a*}	257.3 ± 19.4 ^b
Nonanoic acid	Y	281.3 ± 70.6 ^{ab}	311.4 ± 57.3 ^{a*}	198.0 ± 16.3 ^b	61.93 ± 13.46 ^c
	A	264.5 ± 19.5 ^a	175.9 ± 18.7 ^b	199.5 ± 2.8 ^b	71.76 ± 21.78 ^c
Decanoic acid	Y	177.3 ± 20.3 ^{a*}	149.9 ± 18.3 ^{a*}	146.7 ± 17.2 ^{ab}	111.9 ± 22.6 ^{b*}
	A	84.45 ± 4.5 ^b	84.49 ± 9.22 ^b	185.3 ± 13.3 ^{a*}	36.4 ± 0.6 ^c
Total fatty acids	Y	3411 ± 147 ^a	3476 ± 242 ^a	2296 ± 150 ^b	2064 ± 78 ^b
	A	3542 ± 24 ^a	3268 ± 105 ^b	3328 ± 82 ^{b*}	1961 ± 88 ^c
Ethyl esters					
Ethyl butanoate	Y	147.4 ± 1.9 ^{b*}	192.1 ± 3.8 ^{a*}	83.20 ± 4.60 ^d	94.78 ± 3.38 ^{c*}
	A	73.60 ± 1.49 ^b	72.71 ± 2.10 ^b	117.4 ± 6.4 ^{a*}	73.81 ± 6.25 ^b
Ethyl 2-methylbutanoate	Y	26.11 ± 2.30 ^b	36.06 ± 0.79 ^{a*}	17.62 ± 1.27 ^c	23.62 ± 1.26 ^b
	A	25.13 ± 0.81 ^c	25.85 ± 1.28 ^{bc}	35.30 ± 3.27 ^{a*}	30.38 ± 3.69 ^{b*}
Ethyl 3-methylbutanoate	Y	59.73 ± 4.62 ^b	77.26 ± 4.54 ^{a*}	29.53 ± 1.92 ^d	43.31 ± 1.01 ^c
	A	48.43 ± 1.09 ^b	50.97 ± 2.24 ^{ab}	56.74 ± 1.97 ^{a*}	52.65 ± 7.96 ^{ab}
Ethyl pentanoate	Y	13.38 ± 1.22 ^{a*}	14.86 ± 1.58 ^{a*}	6.46 ± 1.14 ^c	3.65 ± 0.20 ^b
	A	7.72 ± 0.42 ^a	3.83 ± 1.45 ^b	7.02 ± 0.58 ^a	6.26 ± 0.44 ^{a*}
Ethyl hexanoate	Y	156.9 ± 6.2 ^{b*}	177.1 ± 1.9 ^{a*}	120.1 ± 4.5 ^c	181.0 ± 8.4 ^{a*}
	A	114.5 ± 1.7 ^b	105.2 ± 1.2 ^c	137.2 ± 1.3 ^{a*}	70.5 ± 3.5 ^d
Ethyl octanoate	Y	492.4 ± 26.6 ^{a*}	201.7 ± 44.6 ^{b*}	199.8 ± 21.7 ^{b*}	151.6 ± 24.9 ^{b*}
	A	11.92 ± 2.09 ^b	13.69 ± 1.85 ^b	32.01 ± 3.04 ^a	12.09 ± 0.71 ^b
Total ethyl esters	Y	896.0 ± 4.6 ^{a*}	699.1 ± 5.0 ^{b*}	456.7 ± 3.4 ^{d*}	498.0 ± 4.9 ^{c*}
	A	281.3 ± 1.3 ^b	272.3 ± 2.2 ^c	385.7 ± 1.7 ^a	245.7 ± 5.0 ^d
Acetate esters					
Butyl acetate	Y	0.59 ± 0.05 ^a	0.38 ± 0.22 ^a	0.09 ± 0.02 ^b	0.11 ± 0.01 ^b
	A	0.53 ± 0.07 ^a	0.10 ± 0.00 ^c	0.13 ± 0.02 ^c	0.21 ± 0.02 ^{b*}
Isoamyl acetate	Y	1217 ± 264 ^a	1178 ± 98 ^{a*}	613.9 ± 69.9 ^b	262.3 ± 17.8 ^b
	A	1317 ± 83 ^a	849.9 ± 31.1 ^c	1031 ± 117 ^{b*}	751.5 ± 51.7 ^{c*}
Hexyl acetate	Y	6.36 ± 0.46 ^a	2.44 ± 0.31 ^b	0.88 ± 0.08 ^c	2.36 ± 0.09 ^b
	A	6.34 ± 0.21 ^a	5.70 ± 0.52 ^{b*}	3.50 ± 0.12 ^{c*}	2.54 ± 0.11 ^d
2-Phenethyl acetate	Y	123.2 ± 8.1 ^a	42.81 ± 1.79 ^b	30.31 ± 3.27 ^c	36.59 ± 5.62 ^{bc}
	A	126.1 ± 4.9 ^a	46.57 ± 3.12 ^c	72.56 ± 3.43 ^{b*}	50.80 ± 0.21 ^{c*}
Total acetate esters	Y	1347 ± 259 ^a	1224 ± 99 ^{a*}	645.2 ± 73.2 ^b	301.3 ± 19.5 ^c
	A	1450 ± 88 ^a	902.3 ± 28.6 ^c	1107 ± 116 ^{b*}	805.0 ± 52.0 ^{c*}

Table 1. Cont.

Volatile Compounds	Young/Aged Wine	Treatments			
		TM7	TM10	TM21	TPHT
Other esters					
Ethyl lactate	Y	142,057 ± 2847 ^b	137,925 ± 6529 ^b	162,549 ± 10,898 ^a	55,354 ± 6695 ^c
	A	153,191 ± 2942 ^{bc*}	171,138 ± 5786 ^{b*}	235,716 ± 26,846 ^{a*}	131,284 ± 6124 ^{c*}
Diethyl succinate	Y	8079 ± 397 ^a	7363 ± 238 ^b	2265 ± 186 ^d	2773 ± 151 ^c
	A	11,982 ± 536 ^{b*}	9060 ± 593 ^{c*}	17,668 ± 1157 ^{a*}	6882 ± 461 ^{d*}
Total other esters	Y	150,136 ± 2574 ^b	145,288 ± 6434 ^b	164,815 ± 11,083 ^a	58,127 ± 6646 ^c
	A	165,173 ± 3473 ^{b*}	180,198 ± 5298 ^{b*}	253,384 ± 26,412 ^{a*}	138,166 ± 5687 ^{c*}
Volatile phenols					
Guaiacol	Y	n.d.	n.d.	n.d.	n.d.
	A	2.28 ± 0.34 ^{bc}	2.20 ± 0.25 ^c	3.82 ± 0.58 ^a	3.03 ± 0.36 ^b
Eugenol	Y	n.d.	n.d.	n.d.	n.d.
	A	3.66 ± 0.21 ^b	6.47 ± 2.42 ^b	19.73 ± 8.04 ^a	2.03 ± 1.09 ^b
4-Ethylguaiacol	Y	n.d.	n.d.	n.d.	n.d.
	A	531.4 ± 34.2 ^a	345.8 ± 29.3 ^b	233.3 ± 29.5 ^c	326.7 ± 56.3 ^b
4-Ethylphenol	Y	17.77 ± 1.54 ^a	7.79 ± 0.23 ^b	1.78 ± 0.32 ^c	2.93 ± 0.64 ^c
	A	352.7 ± 6 ^{a*}	376.6 ± 27.8 ^{a*}	75.55 ± 8.16 ^{b*}	356.2 ± 49.6 ^{a*}
4-Vinylguaiacol	Y	24.82 ± 0.96 ^b	19.91 ± 1.13 ^c	28.25 ± 1.95 ^a	23.59 ± 1.67 ^b
	A	25.44 ± 0.98 ^b	23.32 ± 1.24 ^{b*}	41.29 ± 4.88 ^{a*}	21.30 ± 3.70 ^b
Total volatile phenols	Y	42.59 ± 2.40 ^a	27.70 ± 1.25 ^b	30.02 ± 2.12 ^b	26.52 ± 2.07 ^b
	A	915.5 ± 29.1 ^{a*}	754.4 ± 59.9 ^{b*}	373.7 ± 49.3 ^{c*}	709.2 ± 110.1 ^{b*}
Benzenoids					
Benzaldehyde	Y	2.58 ± 0.30	2.49 ± 0.19	2.18 ± 0.05	2.97 ± 0.1
	A	2.32 ± 0.24 ^b	2.23 ± 0.14 ^b	2.89 ± 0.36 ^{b*}	7.02 ± 0.65 ^{a*}
Furans					
Furfuryl ether	Y	n.d.	n.d.	n.d.	n.d.
	A	104.2 ± 2.7 ^c	104.8 ± 3.0 ^c	126.4 ± 5.7 ^b	166.6 ± 11.1 ^a
Furfural	Y	n.d.	n.d.	n.d.	n.d.
	A	16.88 ± 0.33 ^b	17.81 ± 0.77 ^b	17.91 ± 0.37 ^b	20.08 ± 2.17 ^a
5-Methylfurfural	Y	n.d.	n.d.	n.d.	n.d.
	A	5.70 ± 0.23 ^b	9.88 ± 0.48 ^a	5.99 ± 0.78 ^b	4.71 ± 0.23 ^c
Ethyl-3-furoate	Y	153.0 ± 4.7 ^{a*}	139.3 ± 2.5 ^{b*}	34.62 ± 1.57 ^d	44.94 ± 4.42 ^c
	A	83.84 ± 4.21 ^a	81.83 ± 7.07 ^a	91.05 ± 8.03 ^{a*}	59.03 ± 6.58 ^{b*}
Total furans	Y	153.0 ± 4.7 ^a	139.3 ± 2.5 ^b	34.62 ± 1.57 ^d	44.94 ± 4.42 ^c
	A	210.6 ± 6.1 ^{c*}	214.3 ± 4.5 ^{c*}	241.3 ± 3.6 ^{b*}	250.4 ± 2.3 ^{a*}
Lactones					
trans-oak lactone	Y	n.d.	n.d.	n.d.	n.d.
	A	88.74 ± 1.08 ^b	88.00 ± 6.78 ^b	146.4 ± 14.7 ^a	109.3 ± 18.8 ^b
cis- oak lactone	Y	n.d.	n.d.	n.d.	n.d.
	A	108.9 ± 1.3 ^c	152.3 ± 5.0 ^b	197.4 ± 16.5 ^a	173.4 ± 22.7 ^b
γ- Nonalactone	Y	31.08 ± 1.68	31.75 ± 2.61	28.58 ± 0.92	28.21 ± 2.08
	A	33.23 ± 0.31	33.04 ± 1.45	32.84 ± 2.99	29.82 ± 3.50
γ- Decalactone	Y	n.d.	n.d.	n.d.	n.d.
	A	14.49 ± 0.24 ^b	16.41 ± 1.15 ^b	38.56 ± 3.18 ^a	16.33 ± 2.93 ^b
Total lactones	Y	31.08 ± 1.68	31.75 ± 2.61	28.58 ± 0.92	28.21 ± 2.08
	A	245.3 ± 0.6 ^{c*}	289.7 ± 14.0 ^{bc*}	415.2 ± 36.0 ^{a*}	328.8 ± 47.8 ^{b*}

Abbreviations: TM7—a control treatment with 7 days of maceration; TM10—prolonged 10-day maceration; TM21—extended 21-day post-fermentation maceration; TPHT—48-h pre-fermentation maceration heating at 45 °C followed by 8 days of maceration; Y = young wines; A= aged wines (after aging in wooden barrels). Different letters next to values within a row indicate statistically significant differences between treatments at a significance level of $p < 0.05$ for young (Y) and aged (A) samples separately (using one-way ANOVA and the LSD test). Statistically significant differences between young and aged samples of the same treatment at a significance level of $p < 0.05$ are marked with * (higher value is indicated). n.d. = not detected.

3.1.1. Monoterpenes

Among young wines, TM7-Y had the highest monoterpene concentrations (Table 1). Aging generally reduced monoterpene levels, except in TM21-A wines, which showed an

increase and the highest post-aging concentrations. Monoterpene changes involve acid and enzyme-catalyzed hydrolysis, isomerization, and cyclization [27], along with yeast activity during maceration and fermentation. Their concentrations are influenced by skin extraction [28], hydrolysis of bound forms, yeast conversion, and adsorption by solids [29]. Grape skins, which contain more free and glycosylated monoterpenes than the flesh/juice [10], are typically favored for extraction. In this study, TM21-Y wines showed lower monoterpene concentrations compared to control TM7-Y, possibly due to the losses during fermentation and extended maceration, attributed to adsorption onto yeast and solids, followed by precipitation. In previous research, high negative correlations were found between linalool, nerol, citronellol, and geraniol concentrations, and the duration of maturation in macerated wines [30]. The pre-fermentation heating treatment TPHT-Y also significantly reduced monoterpene concentrations, which was consistent with previous findings on their thermal degradation at 70 °C for 3 h [31]. Significant changes in monoterpenes also occur during aging [8], where the cleavage of glycosidic bonds can increase and oxidation can decrease their concentration. A larger increase in the concentration of several monoterpenes after aging noted in TM21-A in comparison to other wines was possibly a consequence of the increased extraction of glycosides during a long 21-day post-fermentation maceration period and their subsequent hydrolysis into free forms during aging. An increase in α -terpineol during aging was also observed in TM7-A and TM10-A wines, consistent with its formation through monoterpene oxidation [32,33]. A decrease in the concentration of particular and total monoterpenes in TM7-A and TM10-A wines compared to the corresponding young wines was probably a result of oxidation and other conversions. Most monoterpene alcohols convert to terpene oxides over time, having significantly higher sensory detection thresholds, and therefore, a lower impact on wine quality.

3.1.2. C₁₃-Norisoprenoids

The highest concentration of β -damascenone, a key C₁₃-norisoprenoid, was observed in TM21-Y wine (Table 1), likely due to enhanced extraction of its carotenoid precursors. β -Damascenone, along with α - and β -ionone, are prominent C₁₃-norisoprenoids known for their floral and fruity aromas [10]. β -Damascenone, in particular, plays a crucial role in wine aroma due to its potent influence even at low concentrations because of its rather low odor perception threshold of 0.05 µg/L. Its scent is often compared to stewed apples, plums, honey, dried fruit, and prunes [30]. These aroma characteristics are essential in shaping the sensory profile of Teran red wine, adding to its unique aromatic complexity. Grape skins contain high levels of C₁₃-norisoprenoid precursors (79–94% of total content in grapes) [34], but the levels of other norisoprenoids did not significantly increase after extended maceration in this study. In previous research, the highest concentrations were observed after prolonged maceration and maturation [30], but no significant changes were found after vinification with pre-fermentation heating [19]. The lowest β -damascenone, β -ionone, and total norisoprenoid concentrations were observed in TPHT-Y wine, suggesting that the heating process favored their degradation or resulted in the denaturation of enzymes responsible for their release. Similarly, a significant decrease in C₁₃-norisoprenoids after a pre-fermentation heating treatment was reported [31], which aligns with our findings. Aging generally decreased the levels of norisoprenoids found in young wines, as a result of oxidation and various conversions into other forms, with the lowest total level found in TPHT-Y. The concentration of β -damascenone decreased in aged wines of all the treatments, likely due to rapid release from its precursors and reaction with sulfur dioxide [35]. A similar sequence of events happened to β -ionone, although its concentration increased in TM21-A. In fact, TM21-A contained the highest β -damascenone and β -ionone concentrations among aged wines, suggesting prolonged maceration provided the highest reserves. During aging, other C₁₃-norisoprenoids were formed, such as actinidol derivatives, TDN, and TPB. It was noted previously that TDN forms from carotenoid breakdown during wine aging [36] and that increased TDN was observed in ‘Cabernet Sauvignon’ wine aged in oak barrels [37]. Positive correlations between vitispirane, TDN, and actinidol concentrations

and aging time have been reported [11,32]. Increased actinidol levels during aging were likely due to the transformation of norisoprenoid precursors [33,38].

3.1.3. Alcohols

TM10-Y had the highest C₆-alcohol levels, TM7-Y contained the most benzyl and 2-phenylethyl alcohol, while TM21-Y and TPHT-Y had the lowest concentrations of alcohols in general among young wines (Table 1). C₆-alcohols are formed from long-chain fatty acids by enzymes such as lipoxygenase (LOX), hydroperoxide lyase (HPL), (3Z)-(2E)-enal isomerase, and alcohol dehydrogenase (ADH) released during grape crushing [39,40]. Higher 1-hexanol concentrations were found in wines with shorter maceration durations (five days) compared to longer durations (10 and 15 days). Additionally, *trans*- and *cis*-3-hexenol were not detected in wines macerated for 5 or 10 days but were present in those macerated for 15 days [41]. This trend was also observed in this study. Interactions between phenolic and volatile aroma compounds might have also occurred, potentially influencing C₆- and other alcohol levels [42]. Decreases in alcohols can be attributed to their fixation onto macromolecules or berry skins, which can be beneficial as high C₆-alcohol levels might negatively impact aroma [43]. The hydroperoxide lyase enzyme, optimal at 15 °C, is inhibited by higher temperatures [44], explaining the reduced enzyme activity and lower C₆-alcohol concentrations in TPHT-Y treatment wine. Decreased C₆-alcohol levels after pre-fermentation heating during winemaking have been observed in other studies as well, likely due to lipoxygenase inactivation [19,31,37,45]. Lower levels of 2-phenylethyl alcohol in TPHT-Y wine with respect to control possibly resulted from the reduction of its amino acid or glycosidic precursors by heating. Previous research has reported significant decreases in some C₆-alcohols after wine aging [37], which aligns with the trends observed in this study, although not all C₆-alcohols followed this pattern. Aging reduced the concentrations of certain alcohols in TM7-A and TM10-A wines but increased them in TM21-A and TPHT-A wines, which altered the ratios of young to aged wine concentrations among the treatments. It was assumed that TM21 and TPHT treatments may have extracted more alcohol precursors compared to the other treatments. These precursors could have undergone hydrolysis and other transformations during aging, leading to higher concentrations of volatile alcohols in TM21-A and TPHT-A wines. This aligns with the previous observations (30), which suggest that differences in alcohol concentrations could similarly result from the extraction and subsequent chemical changes in alcohol precursors during aging.

3.1.4. Fatty Acids

Significant differences in fatty acid concentrations were observed in young wines, with TM7-Y and TM10-Y having higher levels than TM21-Y and TPHT-Y wines, with the exception of octanoic acid found in lower concentration in TM10-Y than in other wines (Table 1). During maceration and fermentation, yeasts utilize longer-chain fatty acids as a carbon source, potentially reducing their concentrations. Previous research observed that longer macerations decreased fatty acid concentrations, possibly due to ester formation [46]. It was also noted that grape solids inhibit the biosynthesis of volatile fatty acids, leading to lower concentrations in wine [19,47]. However, other studies found a positive correlation between grape solids and volatile fatty acid concentrations in wine [48]. In this study, reduced hexanoic, octanoic, and decanoic acid concentrations were observed in TM21-Y and TPHT-Y treatments with some exceptions, which was consistent with previous findings showing that macerations lasting 10 to 15 days decreased the level of these acids [41]. Reduced medium-chain acids are generally considered beneficial as higher concentrations can lead to off-flavors like fats, cheese, and rancidity [33]. In this study, TPHT-Y treatment led to the most significant decrease in most fatty acids compared to the control, aligning with research noting a decrease with heating treatments, though not statistically significant compared to the control [19]. Fatty acid concentrations were found to increase with aging, as seen for butanoic acid [49], which was confirmed by this study for hexanoic and octanoic

acid in wines of particular treatments, although decreases were also observed, e.g., for decanoic acid. It was assumed that the changes after a 6-month aging period were partly a result of two contrasting phenomena, ethyl ester hydrolysis and adsorption, by the wood of the barrel [37,50].

3.1.5. Ethyl Esters

Among young wines, the concentrations of particular ethyl esters, such as ethyl butanoate, ethyl 2-methylbutanoate, and ethyl 3-methylbutanoate, were the highest in TM10-Y, while the total concentration of ethyl esters, predominantly driven by ethyl octanoate, was the highest in TM7-Y. In contrast, TM21-Y and TPHT-Y wines had lower concentrations of most individual ethyl esters and total ethyl esters compared to the other treatments (Table 1). Ethyl esters and volatile fatty acids share precursors and typically correlate in fermentation [49]. This study found higher ethyl ester concentrations in treatments with shorter maceration times, which was consistent with previous findings [21,41,51]. As suggested [21], it is possible that a longer period of fermentation with skins inhibited the formation of ethyl esters by providing either competitive substrates or inhibitors of the yeast enzymes and/or simply resulted in their adsorption onto skin phenols or other compounds and subsequent removal by precipitation. Grape solids can inhibit fatty acid synthesis in yeast cells during fermentation. This inhibition reduces the production of ethyl esters, as fatty acids are key precursors for their formation. Consequently, this leads to lower concentrations of ethyl esters in wine, as these compounds are less available for synthesis due to the decreased precursor levels in the must [47]. The decrease in ethyl esters in TM21-Y was also likely due to non-enzymatic hydrolysis [50]. It was previously noted that heating reduces ethyl ester concentrations [48], while other studies observed no significant changes [19,31]. In this study, heating reduced the levels of ethyl butanoate, ethyl 3-methylbutanoate, and ethyl octanoate and increased the level of ethyl hexanoate (Table 1). The decrease in major medium-chain ethyl esters during aging is known to be due to a shift in the balance between esterification and hydrolysis toward the latter [32]. Despite the protection provided by sulfur dioxide, ester hydrolysis during barrel aging still occurs. Ethyl ester concentrations were shown to decrease with longer barrel aging, while the losses were lower during aging in stainless steel tanks [52,53]. The results of this study were mostly in line with previous findings, with reduced concentrations of the majority of medium-chain ethyl esters after a 6-month aging period (Table 1). The levels of ethyl esters of short-branched-chain fatty acids, such as ethyl 2-methylbutanoate and ethyl 3-methylbutanoate, increased in TM21-A and TPHT-A wines. This increase was likely due to esterification processes reaching equilibrium concentrations, as suggested by previous studies on esterification during maturation [30].

3.1.6. Acetate Esters

TM7-Y and TM10-Y wines generally had higher acetate ester concentrations than the other remaining young wines, with TM7-Y containing the highest concentrations of hexyl and 2-phenethyl acetate (Table 1). TPHT-Y wine had the lowest concentration of total acetates, mostly due to a significantly reduced isoamyl acetate level. Acetate ester concentrations result from a balance between acyl transferase enzymes, which promote their synthesis, and esterase enzymes, which facilitate hydrolysis [54]. In this study, prolonged maceration led to decreased acetate ester concentrations. The absence of mechanical actions reduces ester evaporation [45], while berry skins can also reduce acetate ester formation by extracting enzyme inhibitors or adsorbing the esters [51]. Another possible reason for the decrease is non-enzymatic acetate esters hydrolysis [50]. Acetate esters from acetyl-CoA and higher alcohols [49] and acetyl-CoA accumulation during fermentation affect acetate ester formation. Higher fermentation temperatures and oxygen availability also impact their synthesis. Heat accelerates chemical reactions, so acetate esters like isoamyl and hexyl acetate, which are stable at 0 °C, hydrolyze quickly above 30 °C [7]. This may explain a significant decrease in acetate esters in TPHT-Y treatment wine. Another reason

is the possible negative effect of heating on acetyl-CoA formation by enzyme inhibition or precursor degradation. It has been found that pre-fermentation heating followed by traditional maceration does not significantly affect acetate ester concentrations, while pre-fermentation heating followed by pressing significantly increases their levels [19]. This was attributed to the absence of solids and high Yeast Assimilable Nitrogen (YAN) extraction during thermal treatment [31]. The inactivation of transferase and esterase enzymes during thermal treatment could have also had an impact on acetate ester production. Although previous research indicated that acetate ester concentrations decrease with barrel aging due to adsorption by wood and chemical hydrolysis [3,33], in this study, no significant changes were observed in treatment TM7-A after aging. However, in the treatment TM10-A, the concentration decreased, while in treatments TM21-A and TPHT-A, the concentrations significantly increased with barrel aging (Table 1).

3.1.7. Other Esters

Among young wines, the highest concentration of ethyl lactate was found in TM21-Y wine, although not drastically different from those in TM7-Y and T10-Y wines (Table 1). TPHT-Y wine contained a significantly reduced level. The applied treatments influenced the concentration of diethyl succinate with significant differences between all the treatments. The highest concentration was determined in control TM7-Y wine, followed, in decreasing order, by TM10-Y, TPHT-Y, and TM21-Y wine. Prolonged maceration significantly reduced the diethyl succinate concentration. Previous research found no significant differences in diethyl succinate levels between 5 and 15 days of maceration but noted a lower concentration after 10 days of maceration [41]. The same authors observed that ethyl lactate concentrations decreased with longer maceration. A significant increase in ethyl lactate and diethyl succinate was observed after aging in wooden barrels in wines of all the treatments (Table 1). Certain amounts of ethyl lactate are formed in alcoholic fermentation, but malolactic fermentation can produce much higher concentrations [53]. A strong positive correlation between prolonged maceration and aging with increased levels of these esters was found previously [30,55]. Other studies confirmed this increase, with higher concentrations observed during a one-year bottle aging [33,37,56,57]. The increase in ethyl lactate and diethyl succinate concentrations during aging likely results from spontaneous esterification to balance precursor concentrations [10,58].

3.1.8. Volatile Phenols

TM7-Y wine had the highest concentration of 4-ethylphenol and total volatile phenols, while the lowest levels of the former compound were observed in TM21-Y and TPHT-Y wines (Table 1). 4-Vinylguaiacol was the most abundant in TM21-Y and the least abundant in TM10-Y wine. Volatile phenols can originate from grapes or be formed during fermentation from phenolic acids and later be transformed by *Brettanomyces/Dekkera* contamination [49]. *Saccharomyces cerevisiae* yeasts produce minimal amounts of volatile phenols due to a relatively low degree of decarboxylation of hydroxycinnamic acids [59]. This aligns with the findings of this study and low volatile phenol concentrations in young wines. Previous research found volatile phenols only in wines macerated for 5 days in contrast to 10 and 15 days [41], similar to this study showing the lowest concentrations of 4-ethylphenol in wine of the maceration treatment with the longest duration. It was noted that heating at 70 °C promotes guaiacol development through thermal decarboxylation of hydroxycinnamic acids [38], but in this study, guaiacol was not detected in young wines obtained by pre-fermentation heating. Increased volatile phenol concentrations with heating have also been observed [29]. Lower 4-ethylphenol concentration in TPHT-Y wine was likely due to thermal degradation, as reported previously [60]. Aging increased volatile phenol levels across all treatments, with the exception of 4-vinylguaiacol in TM7-A and TPHT-A. Aging in wood resulted in the occurrence of guaiacol, eugenol, and 4-ethylguaiacol. The most abundant phenols after aging were 4-ethylguaiacol and 4-ethylphenol. Volatile phenols can also be extracted from oak barrel wood, but increased concentrations in matured wines are

likely due to *Brettanomyces/Dekkera* yeasts, which thrive in aerobic conditions during oak aging [12].

3.1.9. Benzenoids

No significant differences in benzaldehyde concentration were observed in young wines (Table 1). Significant increases in benzaldehyde with longer maceration durations have been observed previously [13,61], while short macerations had no notable effect [62]. Enzymatic hydrolysis and the formation of vinylphenol via cinnamate esterase activity also contribute to increased benzenoid concentrations [63]. Benzaldehyde levels are linked to benzyl alcohol and may result from yeast oxidation of amino acids or from precursors like glycosides or phenolic compounds [64]. Consistent with this study, no significant differences in benzaldehyde concentrations with pre-fermentation heating treatments were observed previously [19]. Conversely, a decrease in benzaldehyde with pre-fermentation heating has been reported [31]. The increase in benzaldehyde after aging is likely due to oxidation processes [65]. Benzaldehyde concentrations increased after aging in TM21-A and TPHT-A treatment wines, with the highest value in the latter. Higher benzaldehyde levels in wines aged in oak barrels compared to stainless steel tanks have been observed previously [53], which was attributed to lignin decomposition [66]. However, no significant changes in benzaldehyde concentrations after 12 months of maturation in oak barrels have also been reported [37].

3.1.10. Furans

Only ethyl-3-furoate was detected in young wines, with the highest concentration in TM7-Y, followed by, in decreasing order, TM10-Y, TPHT-Y, and TM21-Y wines (Table 1). Aging introduced furfuryl ether, furfural, and 5-methylfurfural, with increased ethyl-3-furoate levels. This increase and the formation of additional furans altered the concentration ratios compared to young wines, with TPHT-A treatment showing the highest levels. Furfural concentrations increase with oxygen exposure, as observed previously [32], which is consistent with the findings of this study. Furanic compounds from wooden barrels can be transferred into wine [67], and significant increases in furfural and 5-methylfurfural during aging have been noted [68]. It has been reported that furan levels increase during aging, particularly in wines from prolonged maceration [30]. This increase has been linked to sediment and oxygen presence [69]. Furanic compounds, like furfural, undergo various chemical reactions in wine, including reduction to alcohol by yeasts, reactions with H₂S to form thiols, and interactions with anthocyanins and other phenols [63]. It was found that furfural and 5-methylfurfural are initially extracted from wood, followed by a reduction to alcohols [70].

3.1.11. Lactones

In young wines, only γ -nonalactone was detected with even amounts across the treatments (Table 1). Aging introduced *trans*-oak lactone, *cis*-oak lactone, and γ -decalactone, with increased γ -nonalactone levels altering the lactone ratios between the treatments. This also resulted in changes in lactone ratios compared to young wines. TM21-A treatment wine had the highest lactone concentrations after maturation, mostly attributed to increased oak lactone levels. TM10-A and especially TM7-A wine had lower concentrations than the remaining treatments. Particular lactones can originate from grapes [7] or be produced by yeasts during fermentation [38]. The oak lactones, which significantly impact wine sensory qualities, originate exclusively from the wood of the barrel. Formation can occur through cyclization or enzymatic processes, with glutamic acid often serving as a precursor [49]. The extraction of lactones from oak barrels depends on factors such as the oak origin, toasting level, and drying method, as well as wine's chemical composition [71]. It was found that total acidity affects lactone extraction, with higher acidity leading to lower concentrations of *trans*- and *cis*-oak lactones and vice versa [72]. This observation aligns with the findings of this study, where TM21-Y wines, which had the lowest total acidity,

exhibited the highest levels of *trans*- and *cis*-oak lactones. The lower total acidity in TM21-Y may have facilitated the extraction and stability of these lactones. Although specific acidity data are not shown in a table, the trend observed supports the significant impact of acidity on lactone concentrations.

Increased lactone concentrations after aging, especially with prolonged maceration, have been observed [30]. Lactones have been identified as markers of oak aging [12]. In this study, *trans*-oak lactone concentrations were lower than those of *cis*-oak lactone, consistent with previous findings [70].

3.2. Sensory Evaluation of Wine

In total, 57 distinct aroma attributes were identified and used in the Quantitative Descriptive Analysis (QDA) of Teran wine sensory profiles. The scores for the aroma groups and their key descriptors are illustrated in Figure 1 for young Teran wines and in Figure 2 for aged Teran wines. A comprehensive overview of all aroma descriptors, along with statistical analysis, is provided in Table 2.

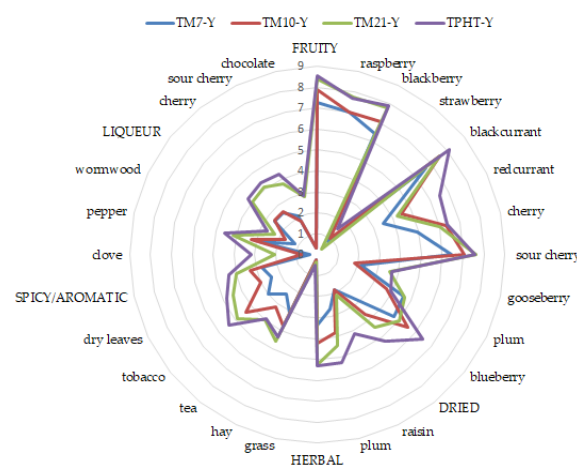


Figure 1. Perception of aroma group intensities obtained by the QDA of Teran young wines produced by different vinification treatments: QDA—quantitative descriptive analysis; TM7—a control treatment with 7 days of maceration; TM10—prolonged 10-day maceration; TM21—extended 21-day post-fermentation maceration; TPHT—48-h pre-fermentation maceration heating at 45 °C followed by 8 days of maceration.

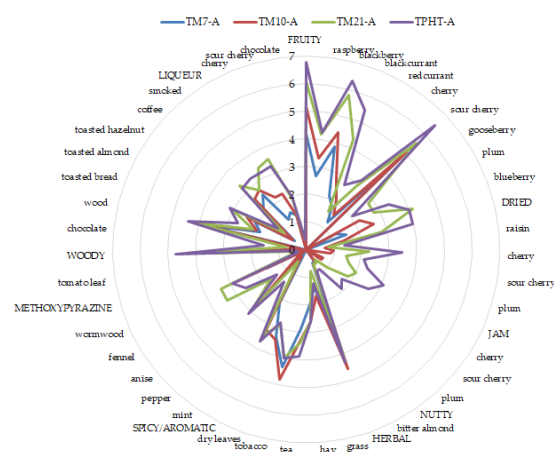


Figure 2. Perception of aroma group intensities obtained by the QDA of Teran aged wines produced by different vinification treatments: QDA—quantitative descriptive analysis; TM7—a control treatment with 7 days of maceration; TM10—prolonged 10-day maceration; TM21—extended 21-day post-fermentation maceration; TPHT—48-h pre-fermentation maceration heating at 45 °C followed by 8 days of maceration.

Table 2. Results of the quantitative descriptive sensory analysis (means \pm standard deviations of the intensities) of Teran wines produced by different vinification treatments.

Aroma Descriptors	Young/Aged Wine	Treatments			
		TM7	TM10	TM21	TPHT
Fruity	Y	7.25 \pm 0.22 ^{c*}	7.88 \pm 0.13 ^{b*}	8.42 \pm 0.19 ^{a*}	8.54 \pm 0.19 ^{a*}
	A	4.17 \pm 0.21 ^d	5.17 \pm 0.21 ^c	6.07 \pm 0.21 ^b	6.77 \pm 0.12 ^a
raspberry	Y	6.96 \pm 0.26 ^{b*}	7.00 \pm 0.38 ^{b*}	7.71 \pm 0.26 ^{a*}	7.67 \pm 0.14 ^{a*}
	A	2.67 \pm 0.23 ^b	3.33 \pm 0.50 ^{ab}	4.20 \pm 1.25 ^{ab}	4.27 \pm 0.90 ^a
blackberry	Y	6.42 \pm 0.47 ^{b*}	7.04 \pm 0.31 ^{ab*}	7.79 \pm 0.38 ^{ab*}	7.88 \pm 0.94 ^a
	A	3.87 \pm 0.23 ^b	4.40 \pm 0.35 ^b	5.80 \pm 0.60 ^a	6.33 \pm 0.31 ^a
strawberry	Y	0.83 \pm 0.72	1.00 \pm 0.87	0.33 \pm 0.58	1.58 \pm 1.77
	A	n.d.	n.d.	n.d.	n.d.
blackcurrant	Y	6.67 \pm 0.29 [*]	7.54 \pm 0.31 [*]	7.54 \pm 1.68 [*]	8.08 \pm 0.29 [*]
	A	2.13 \pm 1.01 ^c	2.80 \pm 1.51 ^{bc}	4.33 \pm 0.50 ^{ab}	5.47 \pm 0.31 ^a
redcurrant	Y	3.50 \pm 0.90 ^b	4.50 \pm 1.32 ^b	4.25 \pm 1.25 ^{b*}	6.50 \pm 1.21 ^{a*}
	A	1.80 \pm 0.60	2.13 \pm 1.14	1.60 \pm 0.69	2.73 \pm 0.12
cherry	Y	4.92 \pm 0.38 [*]	6.33 \pm 0.69 [*]	6.04 \pm 1.77 [*]	6.42 \pm 0.72 [*]
	A	1.27 \pm 0.31 ^b	1.60 \pm 0.60 ^{ab}	2.93 \pm 1.62 ^{ab}	3.20 \pm 0.53 ^a
cherry sour	Y	6.42 \pm 0.52 ^{b*}	7.04 \pm 0.38 ^{ab*}	7.63 \pm 0.50 ^{a*}	7.58 \pm 0.63 ^{a*}
	A	4.83 \pm 0.32 ^c	5.07 \pm 0.31 ^{bc}	5.53 \pm 0.42 ^b	6.47 \pm 0.12 ^a
gooseberry	Y	2.17 \pm 1.89	1.83 \pm 1.71	3.58 \pm 0.14	3.67 \pm 0.38 [*]
	A	n.d.	n.d.	2.80 \pm 0.72	2.07 \pm 0.81
plum	Y	4.58 \pm 0.38 [*]	3.71 \pm 1.70	4.67 \pm 1.01	3.92 \pm 1.18
	A	1.20 \pm 0.20 ^c	2.20 \pm 0.20 ^b	2.80 \pm 0.72 ^{ab}	3.40 \pm 0.35 ^a
blueberry	Y	4.71 \pm 0.85 ^{b*}	5.58 \pm 0.29 ^{ab*}	5.04 \pm 0.26 ^b	6.46 \pm 0.64 ^{a*}
	A	1.53 \pm 0.83 ^c	2.60 \pm 0.92 ^{bc}	4.13 \pm 0.64 ^a	4.00 \pm 0.53 ^{ab}
Dried Fruit	Y	2.58 \pm 0.31 ^d	3.67 \pm 0.07 ^{c*}	4.42 \pm 0.19 ^{b*}	5.25 \pm 0.45 ^{a*}
	A	n.d.	1.40 \pm 0.35 ^c	2.80 \pm 0.20 ^b	3.97 \pm 0.25 ^a
raisin	Y	1.88 \pm 0.82	1.88 \pm 1.72	2.17 \pm 1.61	4.17 \pm 0.63 [*]
	A	n.d.	0.67 \pm 0.46 ^b	0.80 \pm 0.20 ^{ab}	1.40 \pm 0.53 ^a
sour cherry	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	1.00 \pm 0.20 ^c	2.27 \pm 0.42 ^b	3.47 \pm 0.64 ^a
cherry	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	0.87 \pm 0.12 ^c	1.47 \pm 0.23 ^b	2.13 \pm 0.50 ^a
plum	Y	2.67 \pm 0.19 ^d	3.83 \pm 0.26 ^c	4.42 \pm 0.31 ^{b*}	5.29 \pm 0.36 ^{a*}
	A	n.d.	n.d.	1.60 \pm 0.40 ^b	2.33 \pm 0.31 ^a
Jam	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	0.87 \pm 0.12 ^c	2.00 \pm 0.53 ^b	3.07 \pm 0.15 ^a
sour cherry	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	0.67 \pm 0.23 ^c	1.80 \pm 0.40 ^b	2.67 \pm 0.50 ^a
cherry	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	n.d.	1.00 \pm 0.35 ^b	1.67 \pm 0.46 ^a
plum	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	n.d.	0.47 \pm 0.42 ^b	1.93 \pm 0.58 ^a
Nutty	Y	n.d.	n.d.	n.d.	n.d.
	A	0.53 \pm 0.50	0.60 \pm 0.60	0.60 \pm 0.00	0.87 \pm 0.46
bitter almond (marzipan)	Y	n.d.	n.d.	n.d.	n.d.
	A	0.53 \pm 0.50	0.60 \pm 0.60	0.60 \pm 0.00	0.87 \pm 0.46
Herbal	Y	3.33 \pm 0.59 ^c	4.25 \pm 0.13 ^b	5.25 \pm 0.22 ^{a*}	5.29 \pm 0.36 ^{a*}
	A	3.53 \pm 0.49 ^b	4.56 \pm 0.48 ^a	4.56 \pm 0.48 ^a	4.56 \pm 0.48 ^a
grass	Y	0.25 \pm 0.43	0.25 \pm 0.43	0.33 \pm 0.58	0.50 \pm 0.43
	A	1.38 \pm 0.56 [*]	1.72 \pm 0.93	1.20 \pm 1.13	1.27 \pm 0.61
hay	Y	3.08 \pm 0.75 ^c	3.79 \pm 0.26 ^{bc}	4.58 \pm 0.14 ^{a*}	4.33 \pm 0.14 ^{ab*}
	A	2.00 \pm 0.53	2.63 \pm 1.10	2.60 \pm 0.35	2.60 \pm 0.72
tea	Y	2.42 \pm 0.58 ^b	3.17 \pm 0.29 ^{ab}	4.00 \pm 0.87 ^{ab}	3.92 \pm 0.72 ^a
	A	2.87 \pm 0.42 ^b	3.33 \pm 0.31 ^{ab}	3.40 \pm 0.60 ^{ab}	3.87 \pm 0.61 ^a

Table 2. Cont.

Aroma Descriptors	Young/Aged Wine	Treatments			
		TM7	TM10	TM21	TPHT
tobacco	Y	3.00 ± 0.66 ^c	4.38 ± 0.22 ^b	4.92 ± 0.38 ^{ab}	5.42 ± 0.36 ^{a*}
	A	4.33 ± 0.12 [*]	4.81 ± 0.72	4.00 ± 0.53	4.00 ± 0.53
dry leaves	Y	2.42 ± 0.72 ^c	3.00 ± 1.39 ^{bc}	4.50 ± 0.66 ^{ab*}	4.83 ± 0.29 ^{a*}
	A	3.33 ± 0.81	3.43 ± 0.97	2.87 ± 0.64	2.80 ± 0.69
Spicy/Aromatic	Y	2.75 ± 0.38 ^b	3.29 ± 0.47 ^{ab}	3.96 ± 0.19 ^a	4.33 ± 0.52 ^a
	A	2.17 ± 0.29 ^b	3.23 ± 0.25 ^a	3.50 ± 0.26 ^a	3.70 ± 0.26 ^a
clove	Y	0.33 ± 0.29 ^b	0.79 ± 0.75 ^b	2.04 ± 1.82 ^{ab}	3.17 ± 0.63 ^a
	A	n.d.	n.d.	n.d.	n.d.
mint	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	0.27 ± 0.46 ^b	0.53 ± 0.12 ^b	1.40 ± 0.35 ^a
pepper	Y	2.83 ± 0.29 ^{b*}	3.25 ± 0.38 ^{b*}	4.13 ± 0.45 ^{a*}	4.54 ± 0.47 ^{a*}
	A	1.20 ± 0.20 ^c	2.20 ± 0.20 ^b	2.87 ± 0.46 ^a	3.13 ± 0.42 ^a
anis	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	0.40 ± 0.35 ^b	1.67 ± 0.31 ^a	1.40 ± 0.00 ^a
fennel	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	n.d.	3.40 ± 0.00 ^a	2.60 ± 0.35 ^b
wormwood	Y	1.21 ± 0.19 ^c	1.71 ± 0.40 ^{bc}	2.25 ± 0.25 ^{ab}	2.67 ± 0.38 ^a
	A	1.60 ± 0.35 ^b	2.93 ± 0.58 ^{a*}	3.40 ± 0.35 ^{a*}	2.93 ± 0.31 ^a
Methoxypyrazine	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	n.d.	0.47 ± 0.23	0.33 ± 0.42
tomato leaf	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	n.d.	0.47 ± 0.23	0.33 ± 0.42
Woody	Y	n.d.	n.d.	n.d.	n.d.
	A	3.63 ± 0.25 ^c	4.07 ± 0.31 ^{bc}	4.30 ± 0.40 ^{ab}	4.73 ± 0.15 ^a
chocolate	Y	n.d.	n.d.	n.d.	n.d.
	A	n.d.	0.20 ± 0.35 ^b	0.73 ± 0.42 ^{ab}	1.53 ± 0.70 ^a
wood	Y	n.d.	n.d.	n.d.	n.d.
	A	3.80 ± 0.35	4.13 ± 0.23	4.07 ± 0.31	4.40 ± 0.69
toasted bread	Y	n.d.	n.d.	n.d.	n.d.
	A	1.80 ± 0.20 ^b	1.93 ± 0.42 ^b	2.07 ± 0.46 ^{ab}	2.67 ± 0.12 ^a
toasted almond	Y	n.d.	n.d.	n.d.	n.d.
	A	2.13 ± 0.70	2.47 ± 0.95	3.07 ± 0.42	3.13 ± 0.12
toasted hazelnut	Y	n.d.	n.d.	n.d.	n.d.
	A	0.53 ± 0.23 ^b	0.60 ± 0.20 ^b	1.00 ± 0.20 ^a	1.27 ± 0.12 ^a
coffee	Y	n.d.	n.d.	n.d.	n.d.
	A	2.13 ± 0.23 ^b	2.60 ± 0.72 ^{ab}	3.33 ± 0.50 ^{ab}	3.20 ± 0.40 ^a
smoked	Y	n.d.	n.d.	n.d.	n.d.
	A	2.53 ± 0.46	2.73 ± 0.12	2.73 ± 0.61	3.27 ± 0.31
Liqueur	Y	2.58 ± 0.26 ^{b*}	2.63 ± 0.13 ^b	4.00 ± 0.38 ^a	4.25 ± 0.50 ^a
	A	1.27 ± 0.31 ^b	2.20 ± 0.87 ^b	3.43 ± 0.35 ^a	3.23 ± 0.40 ^a
sour cherry	Y	2.58 ± 0.38 ^{b*}	2.63 ± 0.13 ^b	4.13 ± 0.54 ^a	4.38 ± 0.38 ^{a*}
	A	1.47 ± 0.42 ^b	2.20 ± 0.87 ^b	3.53 ± 0.23 ^a	3.27 ± 0.46 ^a
cherry	Y	2.08 ± 0.76 ^b	1.79 ± 0.64 ^b	3.75 ± 0.5 ^{a*}	4.29 ± 0.51 ^{a*}
	A	1.30 ± 0.17 ^b	1.33 ± 0.23 ^b	1.87 ± 0.12 ^a	2.00 ± 0.20 ^a
chocolate	Y	0.33 ± 0.29 ^b	0.33 ± 0.38 ^b	2.83 ± 1.01 ^{a*}	2.88 ± 0.70 ^{a*}
	A	n.d.	n.d.	0.33 ± 0.31	0.33 ± 0.31

Abbreviations: TM7—control treatment with 7 days of maceration; TM10—prolonged 10-day maceration; TM21—extended 21-day post-fermentation maceration; TPHT—48-h pre-fermentation maceration heating at 45 °C followed by 8 days of maceration; Y = young wines; A = aged wines (after aging in wooden barrels). Different letters next to values within a row indicate statistically significant differences between treatments at a significance level of $p < 0.05$ for young (Y) and aged (A) samples separately (using one-way ANOVA and LSD tests). Statistically significant differences between young and aged samples of the same treatment at a significance level of $p < 0.05$ are marked with * (higher value is indicated). n.d. = not detected.

The intensities of fruity aromas in young wines were comparable between the TM21-Y and TPHT-Y treatments, both of which exhibited significantly higher levels than the other two treatments (Table 2 and Figure 1). The predominant fruity aromas in these young wines included raspberry, blackberry, blackcurrant, and sour cherry. Previous research has established that the length of maceration significantly influences the fruity aroma of wine [43]. Similarly, esters were identified as the primary source of fruit aromas in wine [73], while other authors observed that ethyl esters of fatty acids contribute substantially to the fruity notes of red wines [74]. The elevated levels of ethyl-hexanoate, which were many times higher than its sensitivity threshold of 0.014 mg/L [56], likely contributed to the observed differences in fruity aroma intensity. Previous research highlights the positive impact of higher alcohols on the fruity aroma of red wines [75]. The influence of heating treatments on enhancing fruit aroma intensity has been documented in earlier studies [76–78]. It was also observed that pre-fermentation heating treatments significantly boost the intensity of fruitiness compared to control treatments [14]. The observed decrease in ethyl ester concentrations during wine aging in this study resulted in a reduced intensity of fruity aromas. β -Damascenone, a C_{13} -norisoprenoid whose odor is often compared to fruits such as stewed apples and plums, as well as honey [10,59], dried fruit [79], and prunes [30], was found in significantly higher concentrations in young wines from the TM21-Y treatment and in aged wines from the TM21-A treatment compared to the extremely low sensitivity threshold of 0.05 μ g/L [80]. It was hypothesized that the higher concentration of β -damascenone in these wines might have compensated for the lack of impact of volatile esters in these wines. The lower intensity of dried fruit aroma in the control TM7 treatment wines, as well as in TM10 wines (Table 2, Figures 1 and 2), could be due to the masking effect of higher concentrations of 4-ethylphenol in these treatments, both in young and aged wines [81]. Additionally, a weaker intensity of dried fruit aromas observed in this study across all treatments after aging, despite significantly higher concentrations of C_{13} -norisoprenoids, may be related to a notable increase in the concentration of 4-ethylphenol.

In young wines, the jam odor group was not detected (Table 2 and Figure 1). However, in aged wines, the jam odor group was found in wines of all treatments except TM7-A, with significantly higher intensities observed in TM21-A and TPHT-A treatments (Table 2 and Figure 2). The odor of ethyl isobutanoate and ethyl hexanoate has been compared to that of jam [82], which aligns with the significantly higher concentrations of ethyl hexanoate found in TPHT-Y treatment wine in this study. The aroma of wine subjected to pre-fermentation heating treatment followed by maceration has been compared to overripe fruit and jam [83]. Additionally, γ -nonalactone has been noted to contribute to the jammy odor of wine [84,85], and the significantly higher concentrations of γ -nonalactone in aged wines observed in this study support this finding. One of the main groups of volatile compounds synthesized as a result of yeast activity during fermentation is higher alcohols, which are known to carry odors resembling marzipan [9]. Specifically, amyl alcohol or isoamyl alcohol has been identified as having the greatest influence on the marzipan aroma [6]. Aldehydes are also recognized for contributing to nutty odors [9], with benzaldehyde being significant in contributing to the almond-like aroma of wine [12]. The increase in benzaldehyde concentration observed in almost all wines after maturation in this research, particularly in the TPHT-Y treatment, can be associated with the nutty aroma note.

The intensity of herbal/aromatic herb odors was equally strong in TM10-Y, TM21-Y, and TPHT-Y treatment wines (Table 2 and Figure 1). Among these young wines, pepper notes were most prominent within the spices/aromatic herbs category. The duration of maceration has been found to affect the intensity of herbal/aromatic properties [86], and thermovinification has been shown to increase it as well [87]. Aging wine in wood significantly impacts the release of spicy aromas from the wood into the wine [6], with eugenol concentration being associated with them [88], which aligns with the findings of this study. Additionally, the increase in benzaldehyde concentration after aging, particularly in TPHT-

A treatment wine, may contribute not only to the nutty aroma previously mentioned but also to a greater intensity of herbal/aromatic herb odors (Table 2 and Figure 2).

Prolonged maceration has been reported to increase the extraction of methoxypyrazines, although higher concentrations can negatively affect wine quality [7]. Since methoxypyrazines are primarily found in the skin of berries [59], it is possible that the TM21-Y treatment in this study led to greater extraction and thus a more noticeable methoxypyrazine odor in that treatment. During wine maturation, the concentration of methoxypyrazines tends to decrease due to complex formation with phenolic compounds, with a reduction observed in red wines that are richer in phenolic compounds [89]. Given that the intensity of methoxypyrazine odor in this study was very low, it can be concluded that this property did not negatively impact the wine in this case.

In aged wines, the wood aroma attribute was most intensely pronounced in the TM21-A and TPHT-A treatments (Table 2 and Figure 2). Among these aged wines, the predominant wood aromas included notes of wood, toasted almonds, and coffee. According to some authors [90], key compounds contributing to these woody and nutty aromas are lactones, which impact woody and coconut notes, guaiacol and vanillin, which add woody nuances, and furans, which contribute to almond aromas. Previous research [91] also noted that the perception of wood aroma positively correlates with the concentrations of guaiacol, eugenol, and lactones, with increased wood-wine contact enhancing the woody aroma perception. This study observed a similar positive relationship between these compounds and specific wood aromas. Phenolic aldehydes like vanillin play a significant role in wine sensory characteristics, providing aromas reminiscent of vanilla, coffee, dark chocolate, smoke, and woody. Although furfural and 5-methylfurfural do not have a major direct impact, furfural significantly affects the perception of oak lactones, enhancing the aromas of wood, caramel, and vanilla [92]. The sensitivity threshold for *cis*-oak lactone, identified in wines treated with extended post-fermentation maceration for 21 days and found in significantly high concentrations, was previously determined to be 87 µg/L [59]. This suggests that *cis*-oak lactone contributed directly to the aroma of wines of all treatments after aging, particularly TM21-A and TPHT-A wines. Furthermore, some authors [61] have linked the liqueur aroma to ethyl octanoate, while others [93] have associated it with octanoic and nonanoic acids and their ethyl esters. In this study, the concentration of octanoic acid in wines subjected to prolonged post-fermentation maceration and pre-fermentation heating treatments corresponds with these findings, suggesting that the higher concentration of octanoic acid may be associated with a more intense liqueur aroma in these wines.

4. Conclusions

This study offers an in-depth analysis of the impact of maceration duration, pre-fermentation heating, and barrel aging on the aroma and sensory profiles of Teran red wine. The results reveal that pre-fermentation heating at 45 °C followed by 8 days of maceration and extended 21-day post-fermentation maceration significantly reduced the concentrations of most free volatile compounds compared to the control, with the exception of particular tertiary aromas. Despite these reductions, wines from these treatments were characterized by enhanced sensory attributes. Notably, wines from the extended 21-day post-fermentation maceration were characterized by pronounced notes of dried fruit, herbs, and aromatic herbs, with a marked increase in fruity characteristics. This effect is likely due to high concentrations of impactful volatile compounds, such as β -damascenone. The influence of other unidentified compounds cannot be excluded. A similar effect was observed in wine of the pre-fermentation heating treatment, where high ethyl hexanoate concentrations might have played a decisive role. Conversely, shorter maceration treatments of 7 and 10 days resulted in higher concentrations of the majority of free volatile aroma compounds, but with lower intensities of some sensory attributes. It seems that volatile compounds that commonly dominant in white wine aromas, such as ethyl octanoate and isoamyl acetate, did not exhibit the same dominance in shaping the sensory profile of a red wine such as

Teran. The corresponding descriptors often associated with these compounds, such as apple, peach, pear, banana, etc., were not selected as the characteristic Teran attributes in sensory analysis, suggesting that the typical odors produced by these compounds were significantly modulated by interaction with other aromas. The impact of six months of oak barrel aging was substantial, significantly enhancing the concentration of free volatile aroma compounds, particularly enhancing those from the groups of C₁₃-norisoprenoids, volatile phenols, furans, and lactones. Following aging, all treatments exhibited prominent fruity aromas. Notably, treatments combining extended post-fermentation maceration or pre-fermentation heating with oak aging resulted in wines with intensified notes of dried fruit, jam, and liqueur. These findings emphasize the critical roles of maceration duration, pre-fermentation heating, and barrel aging in shaping the aromatic profile and sensory characteristics of Teran wines. The insights gained are essential in refining vinification techniques and optimizing the quality of Teran wines, thus maximizing the potential of this unique variety.

Author Contributions: Conceptualization, S.R. (Sara Rossi) and S.R. (Sanja Radeka); methodology, S.R. (Sara Rossi), S.R. (Sanja Radeka) and I.L.; formal analysis, S.R. (Sara Rossi), E.B., F.O., and I.H.; investigation, S.R. (Sara Rossi) and S.R. (Sanja Radeka); resources, S.R. (Sara Rossi) and S.R. (Sanja Radeka); data curation, S.R. (Sara Rossi) and S.R. (Sanja Radeka); writing—original draft preparation, S.R. (Sara Rossi); writing—review and editing, E.B., F.O., I.H., I.L., A.S.I.P., M.B., T.P. and S.R. (Sanja Radeka); visualization, S.R. (Sara Rossi) and S.R. (Sanja Radeka); supervision, S.R. (Sanja Radeka); project administration, S.R. (Sanja Radeka); funding acquisition, S.R. (Sanja Radeka). All authors have read and agreed to the published version of the manuscript.

Funding: Croatian Science Foundation, Research project “Influence of different vinification technologies on the qualitative characteristics of wines from Croatian autochthonous varieties: The role of wine in human diet”—VINUM SANUM (IP-2018-01-5049) and the project “Young Researchers’ Career Development Project—Training New Doctoral Students” HRZZ-DOK-2018-09-5004).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The raw data supporting the conclusions of this article will be made available by the authors without undue reservation.

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

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