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# Effect of Different Vinification Techniques on the Concentration of Volatile Aroma Compounds and Sensory Profile of Malvazija Istarska Wines

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**Abstract:** The majority of chemical compounds that contribute to varietal aroma originate from grape skin. To investigate the differences between volatile aroma compounds when different maceration conditions are applied, a total of six vinification treatments were carried out on Malvazija istarska (*Vitis vinifera* L.) variety, non-maceration control treatment (C), pre-fermentative two days cryomaceration treatment at 8 °C (CRYO), seven days maceration treatment at 16 °C (M7), 14 days maceration treatment at 16 °C (M14), and prolonged post-fermentative maceration treatments at 16 °C for 21 day (M21) and 42 days (M42). Wines were subjected to GC/MS and sensory analysis. Obtained results showed that prolonged post-fermentative maceration treatments contained the highest concentration of total volatile aroma compounds, precisely monoterpenes, alcohols, and other esters. Contrary, C and CRYO wines resulted in highest concentration of ethyl and acetate esters, and fatty acids. In addition, sensory analysis showed that longer maceration treatment wines (M14, M21, M42) were characterized by more aroma complexity, varietal flowery typicity, pronounced fruitiness, with accentuated dried fruit, moderate honey, and herbal notes. Obtained results can provide valuable information to producers when choosing an appropriate vinification technique based on the desired wine style which may lead to a further diversification of white wine market.

**Keywords:** Malvazija istarska wine; maceration treatments; HS-SPME-GC-MS; volatile aroma compounds; aroma profile; QDA sensory analysis; hedonic 100-point O.I.V. method



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## 1. Introduction

The quality of white wines largely depends on its aromatic profiles [1,2]. Knowing the nature and origin of the compounds that contribute the most to wine aroma is of great importance for wine producers who, by properly choosing the harvest date and using adequate vinification technologies, can make the most of the aromatic potential of the variety and thus contribute to the quality of the wine [3]. Despite the large number of volatile aromatic compounds present in wine, only some of them will influence the formation of the final wine "flavor", that is, the synergy of olfactory, gustatory, and tactile sensations of wine [4]. To adequately understand the chemical compounds in wine that confer desirable sensory characteristics, information regarding both the chemical nature and the sensory properties of a wine, or of those components in the wine, is necessary [4]. The information of the two different types of tools, instrumental and sensory data, is very important to establish the quality of wine [4,5].

The health condition of grapes, cultivar, yeast strain, vinification technology, and ripening and storage conditions are factors that determine the final wine aroma [6,7].

According to [4], volatile compounds are released into wine from several sources, directly from the grape berry, during processing and/or storage, through the action of yeast and bacterial metabolism, from wood, and as a result of chemical reactions during wine storage. One of the technological processes during which aroma compounds are released into the must is the maceration process, i.e., the contact of the solid and liquid parts of the berries. Maceration is a technological procedure that can influence the concentration of aromatic and phenolic compounds in wine [8], and implies a period during which the solid parts of the grape (skin, seeds, and stem) are in contact with the grape juice [9]. Most of the primary wine aroma compounds and their precursors are contained in the skin of the berry [10]. In addition to the influence on the aromatics of the wine, maceration leads to the release of phenolic compounds from the skin and seeds of grapes, and, thus, a higher antioxidant activity of the wine [8,11], which can have a positive effect on human health in the case of moderate consumption [3]. According to [12], higher concentrations of higher alcohols, fatty acids, and esters were recorded in wines produced with the maceration process. In addition, [6] state that maceration leads to an increase in the concentration of free and bound monoterpenes, especially linalool and geraniol in the white wine Malvazija istarska wines.

The duration and temperature of maceration play critical roles in influencing the extent and nature of aroma compound extraction from the grape skins. Maceration duration refers to the length of time that the grape skins are in contact with the juice during the maceration process. Shorter maceration durations of a few hours can provide subtle enhancements to the aromatic profile, while longer maceration periods extending up to a couple of days may yield more pronounced effects [13]. The temperature at which maceration is conducted also significantly influences aroma compound extraction. Low temperatures during maceration, typically around 10 °C, tend to favor the extraction of delicate and volatile compounds, such as floral and fruity aromas. Higher maceration temperatures, on the other hand, can facilitate the extraction of more robust and complex aromatic compounds, potentially enhancing the overall aromatic profile of the resulting white wine [14,15]. The choice of maceration duration depends on various factors, including grape variety, desired aroma intensity, and the specific aroma compounds targeted for extraction [13].

Malvazija istarska (*Vitis vinifera* L.) is an autochthonous and the most spread cultivar in all vine-growing areas of Istria, a viticultural region of Croatia [16]. Usually, dry wines with a fruity-flowery aroma are produced from this grape variety, obtained with fast grape processing and must fermentation at low temperatures. Several authors underline that terpenic compounds play a significant role in varietal wine aroma because of their characteristic fruity-flowery odor [6]. Crespo et al. (2022) [17] reported that the aromatic profile of Malvasia wines with different skin-contact time during winemaking shows some relevant conclusions. Volatile components showed mixed behaviors depending on the skin-contact time. Some compounds increased in concentration with time, while others decreased. Longer duration of skin-contact helps to enhance the floral character provided by the terpenols contained in the skin, especially linalool, and major alcohols such as 2-phenylethanol. Additionally, ref. [6] reported the increase in free and bound monoterpenes in wines that were subjected to skin contact and proposed the use of different maceration techniques to enhance the varietal aromatic potential of this variety.

The sensory analysis includes wine tasting, its sensory estimation and appreciation, and its description [18]. Quantitative descriptive analysis is one of the most comprehensive and informative tools used in sensory analysis [19,20]. This technique can provide complete sensory descriptions of a product such as wine [5]. Grouping the aroma compounds with similar descriptors into aroma series, gives an organoleptic profile of the wine. This procedure enables the relation of quantitative information derived by chemical analysis to sensory perceptions, with a view to obtaining an aroma profile for the wine that is more simple and based on more objective criteria [14,21]. On the other hand, the International Organization of Vine and Wine (OIV) 100-point method is the most widely applied sensory

technique to rate wines. The OIV method uses four predefined sensory categories, which are applicable to all types of wines and, thus, are able to differentiate high and low quality [22].

Most previous studies on maceration in the production of white wines have primarily focused on short-term maceration treatments [6,23,24], lasting from a few hours to a few days, whereas this study investigates a longer maceration duration, and prolonged post-fermentative maceration treatments, lasting up to 42 days. In addition, a simultaneous comparison of such maceration techniques and precise information about their influence on volatile aroma compounds and the sensory profile of wines has not been reported up to date. The aim of this study was to investigate the impact of different maceration durations and temperatures on the concentration of volatile aroma compounds and sensory profile of Malvazija istarska white wines. By systematically varying these parameters, we seek to identify the optimal maceration conditions that yield wines with increased aromatic complexity, intensity, and balance. The findings will provide winemakers with valuable insights for refining their maceration practices and producing white wines that showcase enhanced aromatic attributes, thereby meeting the diverse preferences of wine enthusiasts.

## 2. Materials and Methods

### *2.1. Chemicals and Reagents*

Pure standards of individual 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. Setting up the Experiment*

The experiment was performed using a healthy grapes of *cv*. Malvazija istarska (*Vitis vinifera* L.), produced in the experimental vineyard of the Institute of Agriculture and Tourism (Poreč, (Istria), Croatia). Grapes were manually harvested in 2019 at technological maturity (based on the sugar content). The vinification was carried out at the mini-vinification cellar of the Institute of Agriculture and Tourism. A total of six vinification treatments were applied, standard grape processing without maceration which served as a control treatment (C), pre-fermentative two days cryomaceration treatment at 8 °C (CRYO), 7-day maceration treatment at 16 °C (M7), 14-day maceration treatment at 16 °C (M14), and prolonged post-fermentative maceration treatments at 16 °C for 21 days (M21) and 42 days (M42). Each treatment was carried out in three replications in stainless steel 210 L vats.

The C treatment was set up using the traditional winemaking process for white wines. For obtaining the C treatment, the must was separated from grape skins using a closed-type pneumatic press (Letina inox d.o.o., Čakovec, Croatia), immediately after crushing. The crushed grapes were treated with potassium metabisulfite, AEB SPA (Brescia, Italy) at 1 g/hL and Aromax, AEB SPA (Brescia, Italy) of 2 g/hL immediately after crushing. Non-macerated treatment C was pressed immediately after crushing and the level of total $SO_2$ was adjusted to 50 mg/L. Pressed juice was cold settled for 24 h at 12 °C, followed by the addition of pectolytic enzyme at 5 g/hL (Endozym Aromatic, AEB SPA, Brescia, Italy). Maceration treatments (CRYO, M7, M14, M21, and M42) were set up from homogenized mash, immediately after grapes were destemmed and crushed. Mashes were transferred to stainless-steel tanks to obtain each maceration treatment and its replicates. Potassium metabisulfite in a dose of 5 g/hL, 10 g/hL of Aromax, and 5 g/hL of pectolytic enzyme was added to the mashes.

Each of the six treatments was inoculated with 30 g/hL of selected dry yeast (Fermol Arome Plus, Saccharomyces cerevisiaeAEB SPA (Brescia, Italy) and yeast starter (Fermoplus Starter, AEB SPA (Brescia, Italy) was added at the rehydration process in a dose of 15 g/hL. The fermentation/maceration temperature was set to 16 °C, except for CRYO treatment

that was subjected to 8 °C during two days maceration period and was later set to 16 °C as well, for the onset of the fermentation process.

The level of free $SO_2$ was adjusted to 20 mg/L after pressing each maceration treatment. Treatments pH was adjusted from initial 3.6 to 3.4. A mixture of tartaric, malic, and lactic acid (TLM MIX Acid, AEB SPA, Brescia, Italy), was used in a dose of 1 mL/L to adjust the acidity of the treatments to 5.5 g/L expressed as tartaric acid. Punch-downs were performed on the maceration treatments three times a day during the fermentation period. Yeast supplements (Fermoplus Floral, AEB SPA, Brescia, Italy) were added 4 days after yeast starter addition in a dose of 20 g/hL, and again during the last third of the fermentation process, considering the reducing sugar content, in a dose of 10 g/hL. Each treatment was racked two times during the winemaking process, after which the free $SO_2$ level was adjusted to 30 mg/L. All treatment wines were racked from their lees to a clean 220 L stainless steel vat and infused with nitrogen gas. The level of free and bound $SO_2$ was monitored throughout the whole process and was corrected to 30 mg/L of $SO_2$ after fermentation, before and after racking, and before sampling. Until the analysis, wines were stored in 750 mL dark green glass bottles, sealed with a cork stopper, and kept at the wine cellar temperature (15–17 °C). Approximately 6 months after bottling wines were subjected to standard physico-chemical analysis, volatile aromatic compounds analysis, and sensory analysis.

### *2.3. Standard Physico-Chemical Analysis*

According OIV methods [25], following parameters in wine were analyzed: alcoholic strength by volume (%), reducing sugars (g/L), total dry extract (g/L), total dry extract without reducing sugars (g/L), total acidity (g/L), volatile acidity (g/L), and pH.

### *2.4. Analysis of Volatile Aroma Compounds*

Volatile aroma compounds were isolated from wine samples according to the method previously described by [26], using a headspace solid-phase microextraction (HS-SPME) technique. SPME fiber holder and 50/30 nm divinylbenzene-carboxen-polydimethylsiloxane (DVB-CAR-PDMS) fibers were purchased from Supelco (Bellafonte, PA, USA). Wine sample (1 mL), 50 µL of internal standards solution (2-octanol at 0.84 mg/L, 1-nonanol at 0.82 mg/L, and heptanoic acid at 2.57 mg/L), and distilled water (2.95 mL) were added into the glass vial of 10 mL volume, previously filled with 1.0 g of ammonium sulphate. A sealed vial with a Teflon-faced septum cap, sample preparation was preconditioned at 40 °C for 15 min with stirring (800 rpm) in a heating oven (100–800, Memmert GmbH+Co.KG, Schwabach, Germany). Microextraction was performed on 40 °C for 40 min, again with the stirring. After the extraction, the fiber was inserted into the GC/MS injector port at 248 °C for 10 min, with the first 3 min in the splitless mode.

Identification and quantification of volatile aroma compounds was performed using Varian 3900 gas chromatograph (GC) coupled to a Varian Saturn 2100T ion trap mass spectrometer (MS) (Varian Inc., Harbour City, CA, USA), as previously described by [26,27]. The initial column temperature was 40 °C, then it was increased from 2 °C/min to 240 °C, and it remained at this temperature for the next 10 min. The carrier gas was helium with a 1.2 mL/min flow rate. Electron ionization mode (EI, 70 eV) in the range of 20–350 m/z was used to acquire mass spectra. Identification was performed by comparing retention times and mass spectra with those of the pure standards and with those available in the NIST05 library. Spectra reverse match numbers RM > 800 were considered satisfactory. In the cases of RM < 800, the identification was based on the similarity of the intensities of a quantifier ion and other major ions in the spectra to those in the reference spectra. A solution containing C10 to C28 n-alkanes was injected under the same chromatographic conditions, the linear retention indices were calculated, and the identity of volatile compounds was additionally confirmed by comparison with the retention indices reported in the literature. Standard solutions were also injected, and the calibration curves were constructed with $r^2 > 0.99$ in all cases. Internal standards were used for normalization before quantification

by using calibration curves. The compounds present in high concentrations were quantified based on total ion current peak area, while quantifier ions were used to quantify others. Compounds for which the authentic standards were not available were semi-quantified as equivalents of the corresponding internal standards.

### *2.5. Sensory Analysis*

To obtain a quality evaluation and comprehensive wine aroma assessment six months after bottling sensory analysis of wine were conducted by the accredited Sensory panel of Institute of Agriculture and Tourism, as previously described by [28,29]. The panel is comprised of seven trained wine tasters, members of Croatian Viticultural and Enological Society, certified and authorized by the Croatian Ministry of Agriculture for official commercial wine sensory analysis in placing wines on the Croatian market, hence they were especially trained in Malvazija istarska 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" [30] for organoleptic (sensory) testing of wines using the method prescribed by the Ordinance on wine and fruit wine sensory testing "Official Gazette" [31] N. N. 106/04 with all amendments concluding with N.N. 1/15.

The sensory analysis was performed on 18 wine samples (6 wines × 3 replicates) by both quantitative descriptive analysis (QDA) and hedonic 100-point O.I.V./U.I.O.E. (Organisation Internationale de la Vigne et du Vin/Union Internationale des Oenologues) methods. Wine assessment took place at the Institute of Agriculture and Tourism, in a room constructed in accordance with ISO standards [32]. as Additionally, a standard 200 mL wine tasting glasses [33] were used, in which 50 mL of appropriately cooled sample, at 12 °C, was poured according to hidden schedule assigned by the head of the panel. At the beginning of the sensory analysis, the tasters attuned their criteria for wine aroma by tasting two Malvazija istarska wine samples, one obtained from macerated and the other one from non-macerated grapes.

Quantitative descriptive analysis (QDA) was used to estimate the intensity of perceived individual aroma attributes for the overall impression of each aroma group, the overall typicity, and overall wine impression. The QDA tasting sheet, for white wines, was composed of 57 aroma attributes (descriptors) arranged in 9 groups and of a 10-point structured scale (0 = attribute not perceptible, 10 = attribute strongly perceptible). The aroma attributes (descriptors) sorted into groups in the tasting sheet as follows, floral aroma group (acacia flower, whitethorn flower, carnation, elderflower, linden tree flower, jasmine, lilac, rose, violet, almond flower, orange tree flower, chamomile); fruit aroma group (apricot, peach, apple, banana, plum, quince, lemon, kiwi, pineapple, papaya); dried fruit aroma group (raisins, dried figs, prunes, dried apricots); nutty aroma group (walnut, hazelnut, almond); herbal aroma group (grass, hay, tea, tobacco, dry leaves); spicy and aromatic herb aroma group (heather, laurel, mint, pepper, anise, fennel); sauvignon-like aroma group (volatile thiols derived group: passion fruit, grapefruit, melon, rush broom; methoxypyrazine derived group: green pepper, tomato leaf, urine like odor); muscat-like aroma group (rose, citrus fruits, lily, camphor); and several unsorted descriptors (toasted bread, honey, wax, butter, carob, coniferous resin).

The O.I.V./U.I.O.E. 100-point evaluation method [34] was used to evaluate the visual, nose and taste category and overall judgement, i.e., harmony of the wine with adequate number of points. When evaluating the wine visual category, tasters evaluated limpidity by providing a maximum of 5 points, and aspects other than limpidity by 10 points, hence 15 points in total. Regarding the nose category, genuineness could be rated with a maximum of 6 points, positive intensity could be rated with 8 points, and the quality of aroma was rated with 16 points, therefore 30 point in total. Within the taste category, genuineness could be assessed with a maximum of 6 points, positive intensity and harmonious persistence could be assessed with 8 points, quality could be assessed with 22 points, totaling 44 points. Tasters also ranked the harmony-overall judgement category with 11 points at most and provided a total score that includes the sum of each category scores.

### 2.6. Statistical Data Analysis

All experiments were performed in triplicate, and mean values were used in further data analysis. To determine 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 concentration and sensory attributes scores of analyzed wines.

To obtain a further visualization of the data, principal component analysis (PCA) was applied on the dataset. All statistical analysis was performed using Statistica v.13.2 software (Stat-Soft Inc., Tulsa, OK, USA).

## 3. Results and Discussion

### 3.1. Standard Physico-Chemical Analysis

The results of standard physico-chemical analysis are presented in Table 1. Alcohol content decreased in maceration treatments M7, M14, M21, and M42 in comparison to C and CRYO treatment. As described in our previous work [28], the possible explanation of ethanol decrease could be oxidation or esterification reactions. The total dry extract content showed an increasing trend in maceration treatments, especially longer maceration treatments, in relation to C treatment. The highest total extract content was noted in M7 treatment. According to [35], longer skin contact can enhance the extraction of minerals and organic matter that consequently affects the concentration of total dry extract in wine. Regarding ash content, some authors [24,35,36] reported an increase in ash content when maceration duration increased, and such results are explained by the higher extraction of inorganic matter located in grape skin. In this study, ash content significantly increased in maceration treatments M7, M14, and M42 in relation to control treatment. pH values increased in all maceration treatments in relation to C treatments which is in accordance with previous papers [37,38].

**Table 1.** Standard physico-chemical analysis parameters (means ± standard deviations) of Malvazija istarska wines produced by different vinification techniques.

| Standard Physico-Chemical Parameters | Treatment | | | | | |
|---|---|---|---|---|---|---|
| | **C** | **CRYO** | **M7** | **M14** | **M21** | **M42** |
| Alcohol (vol%) | 12.69 ± 0.07 $^{a}$ | 12.65 ± 0.03 $^{a}$ | 11.74 ± 0.08 $^{b}$ | 11.69 ± 0.08 $^{b}$ | 11.68 ± 0.08 $^{b}$ | 11.63 ± 0.05 $^{b}$ |
| Total dry extract (g/L) | 19.9 ± 0.10 $^{d}$ | 20.04 ± 0.35 $^{d}$ | 22.6 ± 0.00 $^{a}$ | 21.83 ± 0.25 $^{b}$ | 20.73 ± 0.12 $^{c}$ | 21.67 ± 0.12 $^{b}$ |
| Reducing sugars (g/L) | 1.77 ± 0.06 $^{e}$ | 2.20 ± 0.00 $^{cd}$ | 2.67 ± 0.06 $^{a}$ | 2.43 ± 0.06 $^{b}$ | 2.13 ± 0.06 $^{d}$ | 2.23 ± 0.06 $^{c}$ |
| Extract without reducing sugars (g/L) | 17.13 ± 0.06 $^{d}$ | 16.84 ± 0.35 $^{d}$ | 18.93 ± 0.06 $^{a}$ | 18.40 ± 0.30 $^{b}$ | 17.6 ± 0.10 $^{c}$ | 18.43 ± 0.06 $^{b}$ |
| Ash (g/L) | 2.71 ± 0.05 $^{c}$ | 2.84 ± 0.06 $^{bc}$ | 3.11 ± 0.08 $^{a}$ | 3.13 ± 0.01 $^{a}$ | 2.88 ± 0.21 $^{bc}$ | 2.91 ± 0.03 $^{b}$ |
| pH | 3.48 ± 0.01 $^{c}$ | 3.60 ± 0.07 $^{a}$ | 3.55 ± 0.02 $^{b}$ | 3.62 ± 0.01 $^{a}$ | 3.62 ± 0.02 $^{a}$ | 3.63 ± 0.00 $^{a}$ |
| Total acidity [1] (g/L) | 5.00 ± 0.00 $^{ab}$ | 4.40 ± 0.36 $^{c}$ | 5.37 ± 0.12 $^{a}$ | 4.43 ± 0.06 $^{c}$ | 4.57 ± 0.29 $^{bc}$ | 4.97 ± 0.40 $^{ab}$ |
| Volatile acidity [2] (g/L) | 0.44 ± 0.04 $^{b}$ | 0.42 ± 0.10 $^{b}$ | 0.53 ± 0.25 $^{ab}$ | 0.48 ± 0.11 $^{b}$ | 0.60 ± 0.21 $^{ab}$ | 0.85 ± 0.33 $^{a}$ |

[1] as tartaric acid; [2] as acetic acid; C—control treatment, CRYO—pre-fermentative two days cryomaceration treatment, M7—seven days maceration treatment, M14—14 days maceration treatment, M21—prolonged post-fermentative 21-day maceration treatment, M42—prolonged post-fermentative 42-day maceration treatment. Different lowercase superscript letters represent statistically significant differences between treatments at $p < 0.05$ obtained by one-way ANOVA and least significant difference (LSD) test.

Total acidity usually decreases with maceration due to higher potassium extraction from skin and further potassium bi-tartrate precipitation [23,39]. In the present study, treatments CRYO, M14, and M21 showed lower acidity values than C treatments, while other treatments (M7 and M42) did not statistically differed from control treatment. Volatile acidity differed between M42 treatments and C, CRYO, and M14 treatments. Although M42 treatment showed the highest concentration of volatile acidity, such values did not exceed the maximum allowed values for volatile acidity in white wines, according to Croatian Wine Law, NN 32/2019 and Commission Regulation (EC) No 606/2009 [40].

### *3.2. Evaluation of Volatile Aroma Compounds*

The concentrations of volatile aroma compounds are reported in Table 2. In total, 53 volatiles were identified in analyzed wines and grouped according to their chemical structure as follows, monoterpenes, $C_{13}$-norisoprenoides, alcohols, fatty acids, ethyl esters, acetates, other esters, volatile phenols, benzenoids, and lactones. The concentration sum of all identified volatile compounds for each treatment shows that post-fermentative maceration treatments M21 and M42 have significantly the highest concentration of volatile aroma compounds in relation to all other maceration treatments.

**Table 2.** Concentration (μg/L) of volatile aroma compounds (means ± standard deviations) in different Malvazija istarska wines.

| Volatile Compounds | Treatments | | | | | | |
|---|---|---|---|---|---|---|---|
| | C | CRYO | M7 | M14 | M21 | M42 | |
| Monoterpenes | | | | | | | |
| Limonene | 2.50 ± 0.53 | 4.04 ± 0.33 | 4.60 ± 0.30 | 3.79 ± 2.69 | 3.11 ± 3.40 | 4.98 ± 0.68 | n.s. |
| Eucalyptol | 0.20 ± 0.17 $^{b}$ | 1.63 ± 2.34 $^{a}$ | 0.48 ± 0.17 $^{b}$ | 0.50 ± 0.07 $^{b}$ | 4.25 ± 2.95 $^{a}$ | 2.34 ± 2.22 $^{ab}$ | |
| β-pinene | 2.83 ± 2.09 $^{b}$ | 5.55 ± 4.65 $^{ab}$ | 10.15 ± 0.49 $^{ab}$ | 12.42 ± 1.61 $^{a}$ | 10.28 ± 7.34 $^{ab}$ | 9.35 ± 6.93 $^{ab}$ | |
| Linalool | 45.06 ± 2.73 $^{c}$ | 73.72 ± 6.91 $^{b}$ | 69.90 ± 0.80 $^{b}$ | 78.89 ± 4.55 $^{b}$ | 113.91 ± 9.33 $^{a}$ | 111.65 ± 20.50 $^{a}$ | |
| 4-Terpineol | 0.43 ± 0.04 $^{d}$ | 0.57 ± 0.04 $^{bc}$ | 0.89 ± 0.05 $^{a}$ | 0.44 ± 0.04 $^{cd}$ | 0.66 ± 0.15 $^{b}$ | 0.67 ± 0.07 $^{b}$ | |
| Menthol | 13.69 ± 3.54 $^{b}$ | 6.94 ± 0.26 $^{c}$ | 18.98 ± 1.66 $^{a}$ | 6.78 ± 0.59 $^{c}$ | 11.18 ± 1.99 $^{b}$ | 12.28 ± 1.16 $^{b}$ | |
| α-Terpineol | 33.12 ± 0.5 $^{b}$ | 33.45 ± 1.69 $^{b}$ | 39.81 ± 2.84 $^{b}$ | 46.52 ± 1.92 $^{b}$ | 76.68 ± 13.77 $^{a}$ | 68.46 ± 12.17 $^{a}$ | |
| Citronellol | 6.05 ± 1.44 $^{cd}$ | 4.62 ± 0.26 $^{d}$ | 8.86 ± 0.97 $^{bc}$ | 11.52 ± 0.27 $^{b}$ | 19.53 ± 2.32 $^{a}$ | 18.54 ± 2.68 $^{a}$ | |
| Geraniol | 37.68 ± 7.32 $^{b}$ | 71.66 ± 5.92 $^{a}$ | 44.21 ± 13.58 $^{b}$ | 46.62 ± 2.95 $^{b}$ | 40.22 ± 12.90 $^{b}$ | 44.79 ± 5.36 $^{b}$ | |
| Geranyl acetone | 2.90 ± 1.37 $^{a}$ | 2.18 ± 0.15 $^{ab}$ | 1.99 ± 0.37 $^{ab}$ | 0.96 ± 0.18 $^{b}$ | 2.06 ± 0.17 $^{ab}$ | 2.80 ± 1.47 $^{a}$ | |
| *trans*-Nerolidol | 7.26 ± 1.16 $^{a}$ | 5.95 ± 1.18 $^{a}$ | 2.87 ± 0.44 $^{b}$ | 2.47 ± 0.75 $^{b}$ | 2.98 ± 0.24 $^{b}$ | 1.87 ± 0.67 $^{b}$ | |
| *trans*-Rose oxide | 0.58 ± 0.02 $^{d}$ | 0.58 ± 0.04 $^{d}$ | 0.99 ± 0.04 $^{c}$ | 0.96 ± 0.07 $^{c}$ | 1.34 ± 0.05 $^{a}$ | 1.16 ± 0.19 $^{b}$ | |
| Total monoterpenes | 152.31 ± 12.60 $^{c}$ | 210.89 ± 13.92 $^{b}$ | 203.73 ± 20.67 $^{b}$ | 211.89 ± 11.19 $^{b}$ | 286.18 ± 47.82 $^{a}$ | 278.87 ± 42.35 $^{a}$ | |
| C13-norisoprenoides | | | | | | | |
| Vitispirane I | 2.92 ± 0.05 $^{d}$ | 4.04 ± 0.67 $^{cd}$ | 5.26 ± 0.17 $^{bc}$ | 5.39 ± 0.37 $^{b}$ | 6.38 ± 0.49 $^{ab}$ | 6.78 ± 1.53 $^{a}$ | |
| Vitispirane II | 2.25 ± 0.12 $^{c}$ | 2.73 ± 0.46 $^{bc}$ | 3.17 ± 0.26 $^{bc}$ | 3.27 ± 0.35 $^{bc}$ | 5.58 ± 1.21 $^{a}$ | 3.92 ± 0.92 $^{b}$ | |
| β-Damascenone | 18.76 ± 1.78 $^{ab}$ | 23.22 ± 0.61 $^{a}$ | 15.89 ± 4.05 $^{bc}$ | 12.07 ± 3.18 $^{c}$ | 19.27 ± 5.39 $^{ab}$ | 11.08 ± 1.42 $^{c}$ | |
| β-Ionone | 2.41 ± 1.27 | 1.18 ± 0.64 | 3.43 ± 0.61 | 1.73 ± 0.24 | 2.94 ± 0.49 | 3.28 ± 20.89 | |
| α-Isomethyl ionone | 6.78 ± 6.50 | 2.94 ± 0.71 | 3.65 ± 1.05 | 2.88 ± 1.22 | 1.98 ± 0.71 | 2.76 ± 1.38 | n.s. |
| Total C13-norisoprenoides | 33.12 ± 8.21 $^{ab}$ | 34.11 ± 2.84 $^{ab}$ | 31.40 ± 5.42 $^{ab}$ | 25.34 ± 1.84 $^{b}$ | 36.15 ± 5.06 $^{ab}$ | 27.83 ± 4.92 $^{ab}$ | |
| Alcohols | | | | | | | |
| 1-Hexanol | 3006 ± 79 $^{b}$ | 1646 ± 26 $^{d}$ | 2123 ± 110 $^{cd}$ | 2221 ± 41 $^{c}$ | 4329 ± 195 $^{a}$ | 4752 ± 746 $^{a}$ | |
| *trans*-3-Hexen-1-ol | 207.04 ± 6.33 $^{a}$ | 65.76 ± 3.48 $^{c}$ | 51.58 ± 0.22 $^{d}$ | 53.45 ± 1.23 $^{cd}$ | 97.61 ± 3.45 $^{b}$ | 89.28 ± 16.21 $^{b}$ | |
| *cis*-3-Hexen-1-ol | 169.67 ± 10.77 $^{a}$ | 84.38 ± 5.69 $^{b}$ | 46.74 ± 2.08 $^{d}$ | 38.55 ± 1.15 $^{d}$ | 75.17 ± 11.11 $^{bc}$ | 69.91 ± 10.26 c | |
| 2-Phenylethyl Alcohol | 51,271 ± 1578 $^{b}$ | 28,495 ± 1114 $^{c}$ | 38,950 ± 1747 $^{bc}$ | 41,021 ± 951 $^{bc}$ | 77,248 ± 5958 $^{a}$ | 79,674 ± 17,382 $^{a}$ | |
| Total alcohols | 54,654 ± 1631 $^{b}$ | 30,291 ± 1091 $^{c}$ | 41,172 ± 1652 $^{bc}$ | 43,334 ± 927 $^{bc}$ | 81,750 ± 5817 $^{a}$ | 84,585 ± 18,133 $^{a}$ | |
| Fatty acids | | | | | | | |
| Butanoic acid | 4015 ± 324 $^{a}$ | 2425 ± 42 $^{b}$ | 1421 ± 98 $^{d}$ | 1062 ± 28 $^{d}$ | 1854 ± 152 $^{c}$ | 1818 ± 339 $^{c}$ | |
| Hexanoic acid | 12,527 ± 1491 $^{a}$ | 5956 ± 615 $^{abc}$ | 2138 ± 144 $^{bx}$ | 1285 ± 115 $^{c}$ | 3820 ± 1595 $^{bc}$ | 9834 ± 11,287 $^{ab}$ | |
| Octanoic Acid | 11,821 ± 1317 $^{a}$ | 9532 ± 407 $^{b}$ | 2460 ± 117 $^{cd}$ | 1550 ± 73 $^{d}$ | 2703 ± 169 $^{c}$ | 2824 ± 110 $^{c}$ | |
| Nonanoic acid | 84.36 ± 104.91 | 64.33 ± 101.50 | 24.61 ± 19.54 | 19.81 ± 3.12 | 46.85 ± 65.00 | 51.31 ± 51.91 | n.s. |
| n-Decanoic acid | 3558 ± 644 $^{a}$ | 3212 ± 405 $^{a}$ | 526 ± 4 $^{b}$ | 271 ± 21 $^{b}$ | 441 ± 111 $^{b}$ | 339 ± 59 $^{b}$ | |
| Total fatty acids | 32,006 ± 3466 $^{a}$ | 21,189 ± 538 $^{b}$ | 6569 ± 282 $^{cd}$ | 4188 ± 194 $^{d}$ | 8865 ± 1458 $^{cd}$ | 14,866 ± 11,098 $^{bc}$ | |
| Ethyl esters | | | | | | | |
| Ethyl butanoate | 853.76 ± 59.49 $^{a}$ | 561.38 ± 10.47 $^{b}$ | 232.87 ± 22.28 $^{cd}$ | 174.34 ± 4.82 $^{d}$ | 273.98 ± 34.40 $^{c}$ | 242.07 ± 38.89 $^{c}$ | |
| Ethyl 2-methylbutanoate | 26.13 ± 2.29 $^{a}$ | 14.20 ± 1.17 $^{c}$ | 19.72 ± 2.01 $^{bc}$ | 16.86 ± 0.42 $^{c}$ | 28.70 ± 4.85 $^{a}$ | 25.17 ± 5.05 $^{ab}$ | |
| Ethyl 3-methylbutanoate | 53.08 ± 6.90 $^{a}$ | 28.58 ± 3.00 $^{b}$ | 34.47 ± 3.01 $^{b}$ | 30.40 ± 1.90 $^{b}$ | 53.14 ± 9.96 $^{a}$ | 46.74 ± 9.09 $^{a}$ | |
| Ethyl pentanoate | 2.27 ± 0.14 $^{c}$ | 4.93 ± 0.31 $^{a}$ | 2.16 ± 0.21 $^{c}$ | 2.56 ± 0.07 $^{c}$ | 3.65 ± 0.44 $^{b}$ | 3.85 ± 0.48 $^{b}$ | |
| Ethyl hexanoate | 1566 ± 110 $^{a}$ | 1463 ± 10 $^{a}$ | 482 ± 45 $^{b}$ | 340 ± 8 $^{c}$ | 481 ± 44 $^{b}$ | 467 ± 87 b | |
| Ethyl octanoate | 4348 ± 555 $^{a}$ | 3908 ± 239 $^{a}$ | 773 ± 70 $^{b}$ | 505 ± 41 $^{b}$ | 871 ± 108 $^{b}$ | 732 ± 187 b | |
| Ethyl 3-furoate | 188.41 ± 20.3 $^{a}$ | 115.19 ± 12.48 $^{b}$ | 77.12 ± 10.64 $^{c}$ | 63.81 ± 6.06 $^{c}$ | 90.33 ± 18.34 $^{bc}$ | 78.93 ± 19.00 $^{c}$ | |
| Ethyl hex-4-enoate | 9.3 ± 1.55 $^{a}$ | 5.86 ± 1.0 $^{b}$ | 2.78 ± 0.22 $^{d}$ | 3.28 ± 0.08 $^{cd}$ | 6.38 ± 2.1 $^{b}$ | 5.22 ± 0.96 $^{bc}$ | |
| Ethyl 2-hexenoate | 232.1 ± 11.36 $^{a}$ | 64.5 ± 1.65 $^{c}$ | 49.84 ± 4.18 $^{d}$ | 50.2 ± 0.13 $^{d}$ | 82.05 ± 8.18 $^{b}$ | 70.55 ± 12.6 $^{bc}$ | |
| Ethyl cinnamate | 10.02 ± 0.83 $^{ab}$ | 11.42 ± 2 $^{a}$ | 3.92 ± 6.32 $^{ab}$ | 2.81 ± 4.02 $^{b}$ | 8.88 ± 6.54 $^{ab}$ | 7.04 ± 3.42 $^{ab}$ | |
| Total ethyl esters | 7290 ± 724 $^{a}$ | 6177 ± 227 $^{b}$ | 1678 ± 128 $^{cd}$ | 1189 ± 49 $^{d}$ | 1899 ± 196 $^{c}$ | 1679 ± 356 $^{cd}$ | |
| Acetate esters | | | | | | | |
| Butyl acetate | 0.12 ± 0.03 $^{ab}$ | 0.08 ± 0.02 $^{b}$ | 0.15 ± 0.01 $^{a}$ | 0.07 ± 0.01 $^{b}$ | 0.14 ± 0.06 $^{a}$ | 0.16 ± 0.02 $^{a}$ | |
| Isoamyl acetate | 2870 ± 258 $^{a}$ | 1452 ± 404 $^{b}$ | 617 ± 317 $^{c}$ | 440 ± 25 $^{c}$ | 1062 ± 528 $^{bc}$ | 1030 ± 522 $^{bc}$ | |
| Hexyl acetate | 91.88 ± 14.37 $^{a}$ | 39.81 ± 36.17 $^{b}$ | 3.78 ± 2.12 $^{c}$ | 3.49 ± 0.32 $^{c}$ | 6.35 ± 3.63 $^{c}$ | 8.07 ± 4.05 $^{c}$ | |
| 2-Phenethyl acetate | 164.6 ± 21.99 $^{a}$ | 83.41 ± 29.13 $^{b}$ | 30.79 ± 8.60 $^{c}$ | 25.31 ± 0.60 $^{c}$ | 43.27 ± 8.58 $^{c}$ | 47.89 ± 15.84 $^{c}$ | |
| Isobornyl acetate | 14.14 ± 10.90 $^{a}$ | 6.45 ± 0.26 $^{ab}$ | 13.48 ± 1.12 $^{ab}$ | 5.69 ± 0.50 $^{b}$ | 6.41 ± 1.35 $^{ab}$ | 5.90 ± 1.01 $^{b}$ | |
| Total acetate esters | 3141 ± 294 $^{a}$ | 1582 ± 466 $^{b}$ | 667 ± 328 $^{c}$ | 475 ± 25 $^{c}$ | 1118 ± 542 $^{bc}$ | 1092 ± 542 $^{bc}$ | |
| Other esters | | | | | | | |
| Ethyl lactate | 54,251 ± 1501 $^{c}$ | 50,485 ± 32,073 $^{c}$ | 43,406 ± 2542 $^{c}$ | 97,682 ± 90 $^{b}$ | 160,331 ± 2903 $^{a}$ | 152,140 ± 23,248 $^{a}$ | |
| Diethyl succinate | 10,966 ± 469 $^{c}$ | 4609 ± 101 $^{d}$ | 4608 ± 442 $^{d}$ | 5398 ± 83 $^{cd}$ | 18,615 ± 5155 $^{b}$ | 36,258 ± 5934 $^{a}$ | |
| Isoamyl propanoate | 0.11 ± 0.01 $^{c}$ | 0.08 ± 0.02 $^{d}$ | 0.21 ± 0.03 $^{ab}$ | 0.17 ± 0.00 $^{b}$ | 0.23 ± 0.02 $^{a}$ | 0.17 ± 0.04 $^{b}$ | |
| Isoamyl lactate | 2340 ± 95 $^{c}$ | 2047 ± 1469 $^{c}$ | 2571 ± 100 $^{c}$ | 9588 ± 232 $^{b}$ | 16,057 ± 1824 $^{a}$ | 15,660 ± 1916 $^{a}$ | |
| n-Hexyl salicylate | 14.54 ± 4.37 | 17.64 ± 2.50 | 18.40 ± 4.94 | 18.26 ± 3.62 | 11.10 ± 0.01 | 15.95 ± 10.56 | n.s. |
| Total other esters | 67,573 ± 1156 $^{c}$ | 57,159 ± 33,450 $^{c}$ | 50,603 ± 2206 $^{c}$ | 112,686 ± 337 $^{b}$ | 195,015 ± 5677 $^{a}$ | 204,075 ± 30,942 $^{a}$ | |
| Volatile phenols | | | | | | | |

**Table 2.** *Cont.*

| Volatile Compounds | Treatments | | | | | | |
|---|---|---|---|---|---|---|---|
| | C | CRYO | M7 | M14 | M21 | M42 | |
| 4-Ethylguaiacol | 2.38 ± 1.88 $^{c}$ | 1.25 ± 0.43 $^{c}$ | 2.22 ± 0.10 $^{c}$ | 75.76 ± 13.71 $^{a}$ | 40.90 ± 42.06 $^{b}$ | 5.79 ± 0.88 $^{c}$ | |
| Eugenol | 0.81 ± 0.05 | 0.65 ± 0.40 | 1.22 ± 1.17 | 1.48 ± 0.36 | 0.82 ± 0.80 | 1.71 ± 2.23 | n.s. |
| 4-Ethylphenol | 13.21 ± 1.28 | 14.51 ± 2.40 | 14.13 ± 2.88 | 17.14 ± 6.22 | 12.74 ± 2.83 | 11.83 ± 0.66 | n.s. |
| 4-Vinylguaiacol | 23.36 ± 2.50 $^{ab}$ | 26.31 ± 4.29 $^{ab}$ | 19.54 ± 14.28 $^{ab}$ | 29.79 ± 5.21 $^{a}$ | 15.01 ± 11.88 $^{b}$ | 21.8 ± 0.74 $^{ab}$ | |
| Total volatile phenols | 39.76 ± 2.15 $^{bc}$ | 42.72 ± 6.76 $^{bc}$ | 37.11 ± 18.42 $^{c}$ | 124.18 ± 14.68 $^{a}$ | 69.47 ± 36.07 $^{b}$ | 41.14 ± 2.74 $^{bc}$ | |
| Benzenoids | | | | | | | |
| Benzaldehyde | 1.99 ± 0.14 $^{c}$ | 3.74 ± 0.84 $^{c}$ | 11.99 ± 1.04 $^{b}$ | 20.64 ± 1.12 $^{a}$ | 11.46 ± 2.30 $^{b}$ | 19.86 ± 3.96 $^{a}$ | |
| Lactones | | | | | | | |
| γ -Nonalactone | 24.57 ± 3.24 | 21.69 ± 1.47 | 26.31 ± 5.20 | 25.82 ± 3.45 | 24.86 ± 8.35 | 27.80 ± 1.84 | n.s. |
| Total volatile compounds | 164,915 ± 3351 $^{b}$ | 116,711 ± 31,492 $^{c}$ | 101,000 ± 989 $^{c}$ | 162,279 ± 1431 $^{b}$ | 289,075 ± 9415 $^{a}$ | 306,692 ± 39,715 $^{a}$ | |

n.s.—not significant; C—control treatment, CRYO—pre-fermentative two days cryomaceration treatment, M7—seven days maceration treatment, M14—14-day maceration treatment, M21—prolonged post-fermentative 21-day maceration treatment, M42—prolonged post-fermentative 42-day maceration treatment. Different lowercase superscript letters represent statistically significant differences between treatments at $p < 0.05$ obtained by one-way ANOVA and least significant difference (LSD) test.

Total volatile aroma compounds in prolonged post-fermentative maceration treatments M21 and M42, increased for 75% and 86%, respectively, in relation to control treatment.

When observing only the total monoterpenes content, the same trend was observed, meaning that the highest concentrations of total monoterpenes were found in M21 and M42 treatments. Such results were expected given that a notable amount of monoterpenes are contained in grape skin [41] and released during the maceration process. According to [42], the longer the period of maceration, the higher the extraction of linalool into the wine, while, according to [15], the presence of the skins during the fermentation leads to a decrease in terpene concentration. In the present study, monoterpene alcohols, linalool, α-terpineol, and citronellol, which represent the most significant monoterpene compounds in Malvazija istarska wines, reached the highest concentrations in the longest maceration treatments M21 and M42, while significantly the highest concentration of geraniol was recorded in CRYO treatment. Significantly the highest concentration of geraniol in CRYO treatment wine is a result of short pre-fermentative maceration that due to lower temperatures favors the extraction of terpenic compounds into the must and wine as reported by [6].

Norisoprenoids are caroten-derived aroma compounds [43]. A significant difference in the concentration of total C13-norisoprenoids was determined only in the case of M21 treatment, having the highest concentration, and treatment M14, with the lowest concentration of total C13-norisoprenoids. The most abundant C13-norisoprenoid was β-damascenone, responsible for fruity-flowery, exotic fruit, rose-like, honey-like, dried plum, and stewed apple odors [44], with the highest concentrations found in CRYO and M21 and C treatment wines, which did not differ significantly from M7 treatment in the concentration of β-damascenone. Norisoprenoides, β-damascenone, and β-ionone which is considered to be responsible for its characteristic violet aroma [44], showed a decreasing trend after 14 days, and again after 42 days of maceration in the case of β-damascenone. On the other hand, both vitispirane I and II showed an increase in prolonged post-fermentative treatments. It is possible that over time, from bottling to analysis, a decrease in β-damascenone and β-ionone occurred, as well as the appearance of other norisoprenoid compounds, such as vitispiran, that is associated with more evolved structures and aromas [45].

Volatile alcohols, including C-6 alcohols, originate from enzymatic and chemical oxidation of fatty acids precursors extracted from grape skins [15,46]. Those compounds are responsible for herbaceous and vegetal notes [2] even though some alcohols, such as 2-phenylethyl alcohol, have a rose-like [27]. When observing the sum of alcohols, it is evident that they increased only in prolonged post-fermentative treatments, when compared to C treatment. Selli et al. (2006) [47] in their work noted that after 12 h of maceration the highest level of C-6 alcohols was obtained although no significant increase was observed.

Among all the identified alcohols, the most abundant compounds are 2-phenylethyl alcohol and 1-hexenol, respectively, with the highest concentrations of both compounds found in M21 and M42 treatment wines. According to [24], total alcohol concentration

increased in skin contact wine in relation to control wine, which is in agreement with the results from the present study.

Total fatty acids reached the highest values in the control treatment wine (C) and such results are in agreement with those of [15]. Those compounds are produced in the lipid metabolism of yeast and are usually related with fatty, cheese, and rancid attributes [2,48]. The most abundant fatty acids were hexanoic and octanoic acids which is in accordance with [12].

The majority of esters found in wine are secondary metabolites produced by yeast during alcoholic fermentation [49]. In wine, two main ester classes can be formed, the ethyl esters, or ethanol and fatty acids esters, and the acetate esters, higher alcohols and acetic acid esters [50]. Ethyl esters play an essential role in wine fruity aromas. They are usually found in high concentrations and have low detection thresholds [2]. The highest concentration of total ethyl esters was recorded in the C, followed by CRYO treatment wine. Such results are in agreement with those obtained from [51] who noted lower concentration of ethyl esters in pre-fermentative maceration in relation to control wine. Additionally, [15] found that ethyl hexanoate (green apple, banana, violets, and strawberry) and 2-phenyl-ethyl acetate (rose, honey, and tobacco) resulted in significantly higher concentrations in control, non-maceration treatment in relation to both pre-fermentative maceration treatment as well as maceration during fermentation treatment. Such results are in agreement with the ones from the present study.

According to [49], acetate esters are produced from the reaction of acetyl-coA with higher alcohols that are formed by degradation or amino acids or carbohydrates. When observing the total acetate esters, it is evident that the highest concentrations were recorded in the control wine, while longer and prolonged maceration treatments resulted in lowest concentrations.

Regarding other esters, ethyl lactate, diethyl succinate, isoamyl propanoate, and isoamyl lactate n-hexyl salicylate were identified in wines. The highest concentration of total other esters was observed in M21 and M42 treatment wines. Ethyl lactate was the most dominant ester among this group and the highest concentration was found in M21 and M42. Ref. [14] reported fruity and butter aromas associated with ethyl lactate. While [52] did not report any differences in wine maceration treatments in regard to both ethyl lacatate and ethyl succinate, in a present study both esters increased in prolonged post-fermentative treatments. Such results are in accordance with [15], who reported that, of all esters, only ethyl lactate and diethyl succinate had significantly higher levels in the macerated wines compared to other treatments. As reported by [53], diethyl succinate and ethyl lactate are compounds formed principally during malolactic fermentation, hence given that, in this study, elevated concentrations of those compounds were observed, we presumed that spontaneous malolactic fermentation took place. On the other hand, lower concentration of diethyl succinate in cold macerated wines in relation to control, non-maceration wines reported by [54], is in accordance with the results from this study. Regarding the concentration of isoamyl lactate, it is evident that significantly the highest concentration of this ester was observed in M21 and M42 treatment wines. According to [55], isoamyl lactate was associated with cream and nutty aromas in wine.

The significantly highest concentrations of volatile phenols were recorded in treatment M14, with 4-vinylguaiacol, associated with clove and curry notes, being the most abundant of all identified volatile phenols. These compounds are related to pharmaceutical odors, as reported by [2].

Of benzenoids, only benzaldehyde was identified, and the significantly highest concentrations were observed in treatment M14 and M42, while γ-nonalactone was the only lactone identified in all wines, however, the concentrations between the treatments did not differ significantly. As reported by [55], benzaldehyde is responsible for roasted and almond scents in wine.

According to [15], maceration could affect wine volatile aroma composition by influencing the availability of different aroma-precursor amino acids. Authors also reported that

a decrease in some volatile compounds could have been associated with their adsorption by certain macromolecules and skin components.

For a further visualization of differences between treatments according to their volatile aroma content, obtained data were subjected to unsupervised statistical analysis by PCA (Figure 1). The first two principal components, PC1 and PC2, explained 72% of the total variance, enabling a good separation of wine treatments. Prolonged post-fermentative maceration treatments M21 and M42 were clearly separated from C and CRYO treatments along the first principal component which explained 48% of the variation, while the second principal component (PC2) explained 24% of the total variance. According to the obtained plot, M21 and M42 treatment wines highly correlated with total monoterpenes, alcohols, other esters, as well as total volatile aroma compounds. Such results are in concordance with those from Table 1. Total C13-norisoprenoides correlated highly with all treatments, except M14, that was placed on the lower left side of the Cartesian system, indicating a weaker connection of this treatment with C-13 norisoprenoides. Treatment M14, highly and mostly correlated with total volatile phenols and benzenoids, particularly benzaldehyde. Treatments C and CRYO were entirely placed on the right side, correlating highly with total fatty acids, and total ethyl and acetate esters. On the other hand, treatment M7 was placed on the interception of the two axes, indicating a weaker connection with all other wine volatile aroma groups.

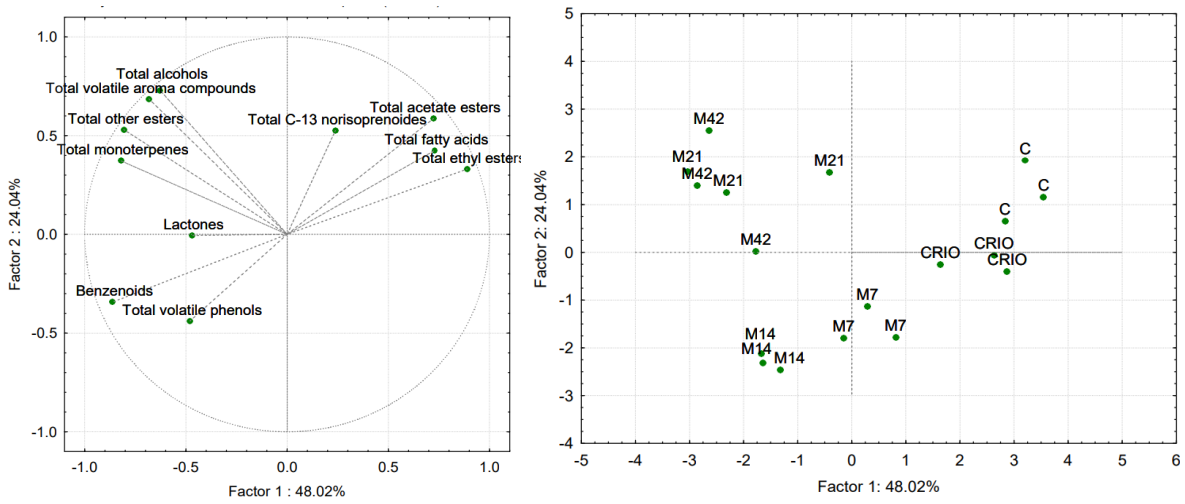


**Figure 1.** Separation of Malvazija istarska wines produced by different maceration treatments presented in three replications in two-dimensional space defined by the first two principal components (PC1 and PC2) and separation of volatile aroma compounds as obtained by gas chromatography: PC—principal components; C—control treatment, CRYO—pre-fermentative two days cryomaceration treatment, M7—seven days maceration treatment, M14—14-day maceration treatment, M21—prolonged post-fermentative 21-day maceration treatment, M42—prolonged post-fermentative 42-day maceration treatment.

### *3.3. Sensory Analysis*

Using the QDA sensory method 49 aroma attributes were identified in investigated Malvazija istarska wine. The points obtained for impression of aroma group in total were shown in Figure 2, and the results for the best rated aroma attributes, that determined particular aroma group were shown in Figure 3.

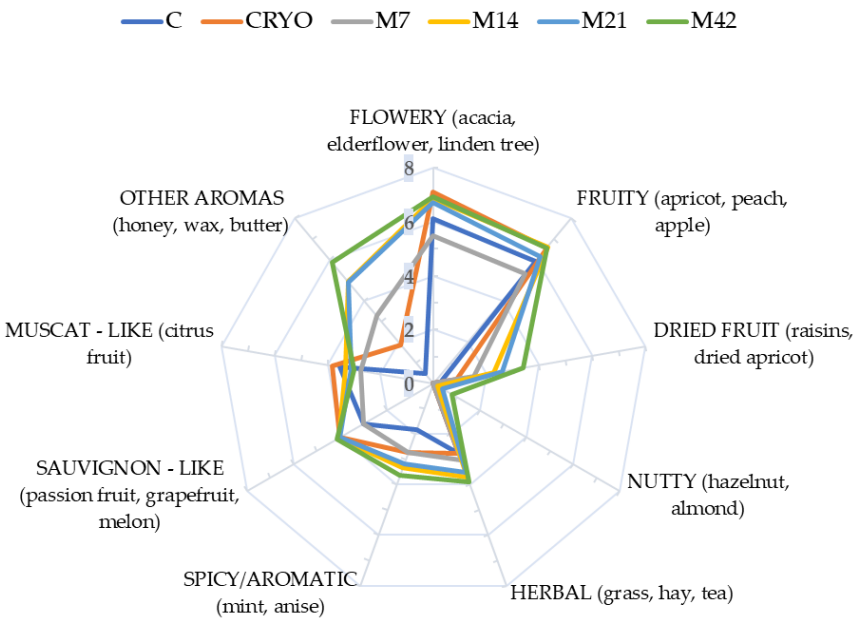


**Figure 2.** Perception of aroma group intensity obtained by QDA of Malvazija istarska wines produced by different vinification treatments: QDA—quantitative descriptive analysis; C—control treatment, CRYO—pre-fermentative two days cryomaceration treatment, M7—seven days maceration treatment, M14—14-day maceration treatment, M21—prolonged post-fermentative 21-day maceration treatment, M42—prolonged post-fermentative 42-day maceration treatment.

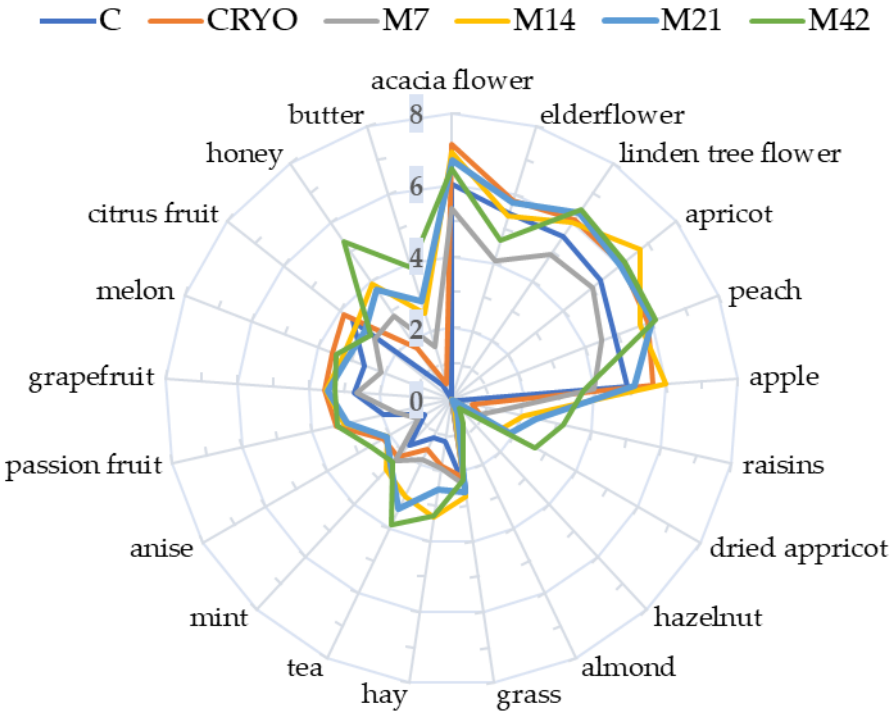


**Figure 3.** Perception of individual aroma attribute intensity, that determined particular aroma group obtained with QDA of Malvazija istarska wines produced by different vinification treatments: QDA—quantitative descriptive analysis; C—control treatment, CRYO—pre-fermentative two days cryomaceration treatment, M7—seven days maceration treatment, M14—14-day maceration treatment, M21—prolonged post-fermentative 21-day maceration treatment, M42—prolonged post-fermentative 42-day maceration treatment.

Perception of flowery and fruity aroma attributes, mostly associated with monoterpenes, that play a significant role in varietal wine aroma [17,56] was significantly higher in treatments submitted to cryomaceration (CRYO), and to M14 and M42 maceration treatments comparing to control treatment (C) and M7. These results correlated with total content of monoterpenes since an increase in monoterpenes content was evident in all treatments in comparison to C treatment. Also, the perception of those aroma groups was rated with the highest points, on a scale from 0 to 10, in comparison to other aroma groups, which is in accordance with results obtained in our previous study on Malvazija istarska wine [57]. The most represented flowery aroma attributes were those that remind of acacia flower, elderflower, and linden tree flower, classifying Malvazija istarska to a cultivar with significant flowery aromatic potential which was assumed to be partially a result of increased extraction of monoterpenes, varietal aroma compounds contained in grape berry skins [6]. The most important monoterpenoids in wine are linalool, *trans*-hotrienol, citronellol, geraniol, nerol, (−)-cis-rose oxide, and α-terpineol [58], and except for flowery aromas they are also responsible for muscat-like scents, in particular citrus fruit attributes [7,59]. This agreed with our findings since muscat-like aroma group was mostly associated with citrus aromas, the highest intensities of which were noted in CRYO treatment wine, possibly due to highest geraniol concentration also found in this treatment.

In addition, [60] reported that short cold pre-fermentation maceration applied to several Croatian native varieties had the significant influence on increasing of primary aroma compounds, i.e., terpenes. On the other hand, the most dominant fruity odors were described as apricots, peaches, and apples, as was reported in our earlier studies [44,61], and they were results not only of monoterpenes but ethyl esters, which were essential in imparting a fruity character to wine [4,15].

Furthermore, intensity of dried fruit aromas increased proportionally along with maceration duration, with significantly the highest score detected in treatment submitted to the longest maceration of 42 days. The highest intensity of dried fruit aromas was expressed, such as raisins and dried apricot odors, which was suggested to be due to the formation of β-damascenone already in the grapes [15]. In our case, β-damascenone content was significantly the highest in CRYO treatment, and this was not in correspondence with intensity of dried fruit aroma. Ref. [15] who also investigated sensory profile of macerated and non-macerated wines, reported that the presence of grape skin throughout the fermentation decreased the tropical and fruity notes but significantly increased sweet-associated aromas, such as marmalade, raisins, honey, and dry grass, which supports the findings from our study. These results could be further explained by the increase in the concentration of volatile alcohols, that are associated with herbaceous odor, and that increase under maceration conditions [47].

Following the previous finding, in perception of herbal group aroma, the most grass, hay and tea attributes, the impact of extended skin-contact was evident because treatments M14, M21, and M42 were statistically greater, regardless of maceration duration length, in relation to control (C), CRYO, and M7 treatment.

The nutty aroma group described by almond and hazelnut notes was present only in treatments exposed to longer (M14) and prolonged maceration (M21 and M42) with the highest score achieved in M21 and M42. These results agreed with the content of isoamyl lactate, the concentration of which was also the highest in those treatments. Isoamyl lactate is a compound associated with cream and nutty aromas in wine [55]. In addition, intensity of almond odor in our study was in accordance with concentration of benzaldehyde compound, responsible for roasted and almond scents in wine [55].

The intensity of spicy and aromatic herb notes, such as mint and anise, was significantly higher in all treatments in comparison to the control treatment (C), increasing along with maceration duration, and showing the strongest intensity in M42 treatment. These findings were directly related to the concentration of vitispirane I, C13-norisoprenoid compound associated with woody and spicy aromas [62].

The overall impression of sauvignon-like aromas was significantly higher in CRYO, M14, M21, and M42 treatments in comparison to control treatment (C), with grapefruit, passion fruit, and melon nuances prevailing, derived from volatile thiol compounds. In earlier investigations of the Malvazija istarska aroma profile [57], sauvignon-like odors were ordinarily identified by sensory analysis but thiol compounds, of which the majority sauvignon-like aromas originated [7,59], were not analyzed in this study.

Among other aroma group attributes, honey-like odor was notably perceived in all treatments, with a significantly higher intensity value in prolonged post-fermentative maceration treatment (M42) in comparison to C treatment and other maceration treatments, meaning that longer maceration duration had a positive effect on this aroma attribute. Also, this result was in correspondence with content of 2-phenylethyl alcohol aroma compound (Table 2), which is among other few compounds (β-damascenone, 2-phenyl-ethyl acetate), responsible for honey-like odor [4,27,44,62]. Furthermore, butter odor was also detected in all treatments except C treatment, with an increase in intensity in relation to maceration duration, and, significantly, the highest value was found in M42. Such a result nearly coincided with the concentration of ethyl lactate, a compound directly associated with butter attributes [14].

Although the overall composition of most grape varieties is very similar, there are clear and distinct aroma and flavor differences between most cultivars. These differences can mostly be attributed to relatively minor variations in the ratios of the compounds that constitute the aroma profile of a grape [7]. Among the compounds that determine the free varietal aroma, determined by volatile substances linked to the aromatic typicity of the variety, two chemical families are distinguished, pyrazines and terpenes [18,63]. In the case of Malvazija istarska, monoterpenes are regarded as typical grape varietal odorants with an accentuated acacia flower odor with fruity nuances [44,50]. In this study, the overall typicity score in treatments CRYO, M14, M21, and M42 was statistically higher compared to C and M7, supporting the fact that in white wine vinification, skin contact treatment is a process often applied to increase the wine's varietal character [14,15,47].

Regarding hedonic 100-point O.I.V./U.I.O.E. method a total score was taken into account. Obtained results showed (Figure 4) that wines submitted to longer (M14) and prolonged post-fermentative maceration (M14, M21, and M42) achieved the highest scores with no significant difference among them, i.e., no difference between periods of maceration duration was evident. Total scores in those wines ranged from 89.27 in M21, 89.33 in M14 to the greatest 90.6 point in M42. Therefore, CRYO treatment was statistically equal to M14 and M21 wines. Conversely, control treatment (C) and M7 received the lowest scores statistically. Similar results were obtained by QDA-wine overall impression where M14, M21 and M42 wine also obtained the most points, while control wine (C) showed the lowest intensity of QDA-overall impression by a significant margin.

Therefore, these results can be correlated with the concentration sum of all identified volatile compounds (Table 2) where the greatest content was obtained in treatments exposed to prolonged maceration (M21, M42), meaning that numerous volatile compounds, i.e., complex aroma profile have a noted impact on overall impression of wine. Those wines obtained with longer (M14) and prolonged skin-contact treatments (M21, M42) were distinguished with aroma complexity, varietal flowery typicity, pronounced fruitiness, with accentuated dried fruit, moderate honey and herbal notes, and a hint of spicy touch.

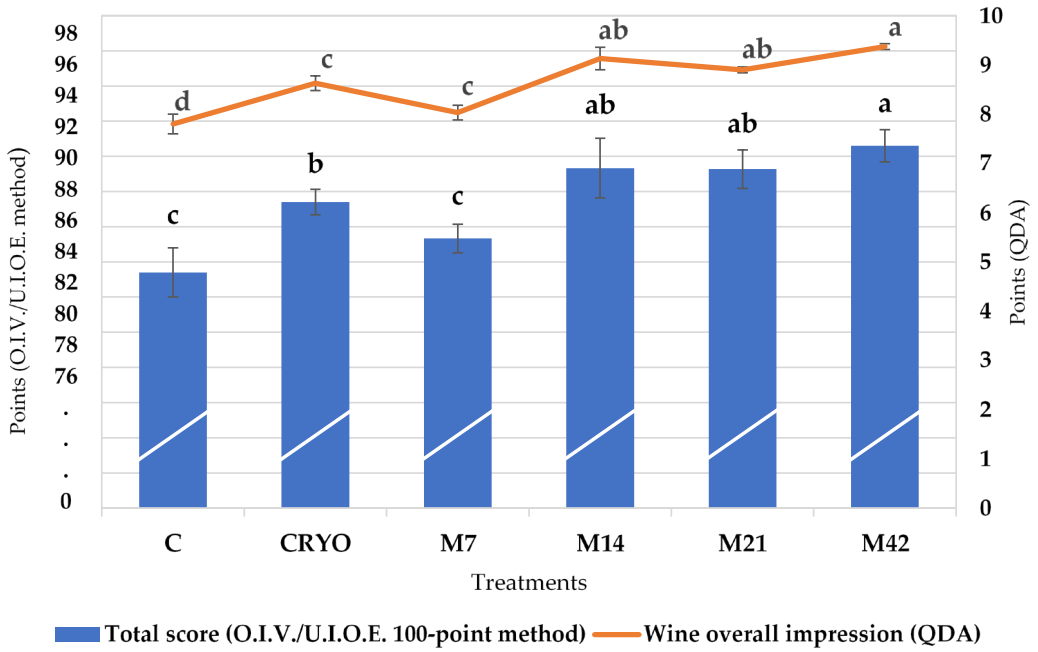


**Figure 4.** Total score of sensory analysis of wine obtained by O.I.V./U.I.O.E. The 100-point method and intensity values of the overall impression obtained by QDA of Malvazija istarska wines produced by different vinification treatments: QDA—quantitative descriptive analysis; C—control treatment, CRYO—pre-fermentative two days cryomaceration treatment, M7—7-day maceration treatment, M14—14-day maceration treatment, M21—prolonged post-fermentative 21-day maceration treatment, M42—prolonged post-fermentative 42-day maceration treatment. Different lowercase letters represent statistically significant differences between treatments at $p < 0.05$ obtained by one-way ANOVA and least significant difference (LSD) test.

## 4. Conclusions

The present investigation provides valuable insights into the impact of different maceration treatments on the aroma profile and sensory characteristics of wines. The results demonstrated that prolonged post-fermentative maceration treatments were particularly effective in significantly enhancing the presence of volatile aroma compounds, specifically monoterpenes, alcohols, other esters, as well as total volatile aroma compounds. On the other hand, wines produced through the C and CRYO methods exhibited highest levels of acetate and ethyl esters, as well as fatty acids.

Furthermore, the sensory analysis revealed that wines subjected to 14-day maceration (M14) and prolonged maceration treatments (M21, M42) exhibited a greater complexity in aroma, with a distinctive varietal flowery typicity and pronounced fruitiness. These wines also demonstrated a notable dried fruits odor, accompanied by moderate herbal and honey notes.

These results highlight the importance of maceration duration in shaping the aromatic profile and sensory attributes of wines. Winemakers can utilize this knowledge to tailor their production methods and optimize the desired flavor and aroma characteristics based on consumer preferences. Further research in this area is necessary to explore the underlying mechanisms and potential of different grape varieties according to applied winemaking technics, thereby advancing the understanding of wine production and sensory experiences.

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