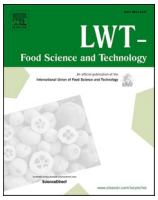
LWT - Food Science and Technology 192 (2024) 115698

Contents lists available at ScienceDirect

LWT

journal homepage: www.elsevier.com/locate/lwt



Influence of prolonged maceration on phenolic compounds, volatile profile and sensory properties of wines from Minutolo and Verdeca, two Apulian white grape varieties

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ARTICLE INFO

Keywords:
Macerated white wine
Orange wine
Amber wine
Free-choice profiling
Terpenes

ABSTRACT

Prolonged maceration was applied to two Apulian white grape cultivars, Minutolo and Verdeca. The impact of prolonged maceration (up to six months) on the quality of the wines was evaluated through characterization of phenolic, volatile and sensory profiles. Macerated wines presented a peak of extraction after 4 months, followed by a decrease probably due to precipitation/reabsorption. Extraction of seed phenols occurred during longer maceration times. Volatile profile was also modified, showing a different behavior at different maceration times. Oxidation and aging compounds appeared during maceration, that are unusual for traditional white wines. Furthermore, maceration successfully enhanced varietal identity of Minutolo. The terpene profile of Minutolo wines changed during maceration, with a maximum of terpenols after 1 month of maceration, while longer maceration added a pattern of oxidized terpenes. Sensory analysis highlighted clearly distinct profiles of wines, according to both cultivar and maceration time. A good attitude was observed of both varieties to undergo maceration. In particular, Verdeca appeared suitable for longer maceration times, with flowery, citrus and apricot notes after 4 months, while enhanced apricot notes were perceived after 6 months. On the other hand, Minutolo wine after 6 months of maceration was characterized by a marked astringency and peach flavor.

1. Introduction

Macerated white wines are defined as ''white wines derived from the alcoholic fermentation of a must in prolonged contact with grape pomace, including skins, pulp, seeds and eventually stems'' (OIV - International Organisation of Vine and Wine, 2020). This technique, that is nowadays increasingly attracting the attention of consumers, producers and researchers, allows to obtain wines that incorporate characteristics of both red and white wines. Sometimes these wines are defined as "orange wines" or "amber wines" (Milat et al., 2019).

There are still few studies available involving macerated white wines and they are mostly focused on their phenolic profile which is found to be richer than in conventional ones (Bestulić et al., 2022; Giriboni et al., 2016; Korenika et al., 2014; Milat et al., 2019; Rustioni et al., 2023). As a result, antioxidant and vasodilator activity are also increased and became similar to those of red wines (Korenika et al., 2014; Milat et al., 2019; Radeka et al., 2022; Salemnia et al., 2019).

Maceration applied during white winemaking seems to induce a faster onset of fermentation, probably due to the extraction of readily assimilable nitrogen and co-factors necessary for the correct development of the cells (Sancho-Galán et al., 2021; Townshend, 2018). It also implies increased potassium extraction, that leads to a higher pH value, a decrease in total acidity, and a rise in volatile acidity (Darias-Martín et al., 2000; Sancho-Galán et al., 2021). Moreover, maceration affects sensory profile due to increased extraction of phenols, which results in higher antioxidant activity, but can also lead to increased sensation of astringency and bitterness as well as to the development of orange/-amber colour that is typical in these wines (Bestulić et al., 2022). Furthermore, long maceration can have a negative impact on esters, β-damascenone, hotrienol. It can also increase sulfur compounds

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https://doi.org/10.1016/j.lwt.2023.115698
Received 26 July 2023; Received in revised form 7 December 2023; Accepted 25 December 2023
Available online 29 December 2023

(particularly dimethyl sulphide) (Buican et al., 2023), which could be related to defects in sensorial properties. Nevertheless, as a consequence of higher extraction of aromatic volatile compounds and aromatic precursors, macerated white wines can be, depending on a series of factors (es. cv, percentages of skins, maceration time and temperature, etc), appreciated for their high aromatic intensity and complexity (Bestulić et al., 2022; Buican et al., 2023; Darias-Martín et al., 2000; Salemnia et al., 2019; Townshend, 2018). In fact, sensory profile of long macerated white wines is influenced by compounds that are typical of aging process (ethyl acetate, ethyl 3-metylbutyrate, isoamyl alcohol, TDN, ethyl lactate) that give rise to floral and fruity notes but also sweet sensations. Higher concentration of volatile benzoic compounds can determine honey, dried figs and tobacco notes. Anyway, aroma pattern cahnges during aging, with a decline in fruity and floral aromas, that can be replaced by spicy ones (Buican et al., 2023).

Prolonged maceration is a valid technique to enhance white wines' quality and differentiate production. However, macerated white wines characteristics cannot be generalized, as they depend on many factors (grape variety, ripening state, winemaking parameters etc.). The recent review by Buican et al. (2023) highlighted some aspects requiring further research, in order to exploit this winemaking technique outside of its traditional areas. In particular, an in-depth study is required to improve the technique, adapt it to different cultivars and assess the optimal duration of maceration. Such research could support industry on the way of the exploitation of local biodiversity and of the adaptation to new market trends and environmental changes.

The present research was aimed at applying this ancient winemaking technique to preserve and valorise Apulian cultivars by producing wines which are different from conventional ones in sensory properties and chemical profile. To this aim, prolonged maceration from 1 to 6 months was applied to two native Apulian white grape varieties: the first one, Minutolo is a terpenic variety (Paradiso et al., 2022) while, the other, Verdeca, is more neutral. The impact of prolonged maceration on the quality of the wines obtained from these cultivars has been evaluated throughout physicochemical analysis and characterization of phenolic, volatile and sensory profiles.

2. Materials and methods

2.1. Wine samples

Grapes from *Vitis vinifera* cv. Minutolo and Verdeca were harvested at technological maturity and phytosanitary health 100%, placed in boxes, transported to winery and immediately processed. At harvest, grapes had a potential alcohol content of 13% vol and a pH of 3.3. After crushing and destemming, potassium metabisulphite (6 g 100 kg^{-1}) was added to grapes, together with ascorbic acid (2 g 100 kg^{-1}), oenological tannin (3 g 100 kg^{-1}, Galitan, Enolife s.r.l., Montemesola, Italy) and pectolytic enzymes for white grapes (1 g 100 kg^{-1}, Lisofine, Enolife s.r.l., Montemesola, Italy). For each cv. crushed grapes were divided into two batches: one batch was directly pressed into a pneumatic press and transferred to a 25 hL steel tank for conventional white winemaking (V-CT and M-CT); the other was transferred into three 50 L steel tanks and submitted to winemaking with maceration. Fermentation was carried out for both control and macerated samples adding 20 g hL^{-1} yeast (*Saccharomyces cerevisiae* LF 13 V, Enolife s.r.l., Montemesola, Italy). Both batches were submitted to the same nitrogen supplementation protocol. Fermentation temperature was kept at 15–16 °C with a final rise at 20 °C. During maceration, push down occurred three times a day until the first half of fermentation, two times a day until ¾ of fermentation and then once a day till the end of fermentation. At the end of fermentation (16 days after inoculation) macerated samples from each 50 L tank were transferred into four 1 L airtight glass jars and kept, to obtain 1 month (M-1, V-1), two months (M-2, V-2), four months (M-4, V-4) and six months (M-6, V-6) macerated samples. Overall, each macerated wine was obtained in three experimental replicates. Maceration was carried out at room temperature (20 ± 3 °C). Both control and macerated wines were bottled in glass 0.75 L bottles and rapidly analyzed.

2.2. Chemical analysis

Main oenological parameters were evaluated using the FTIR technique (OenoFoss, Foss, Hillerod, DK). Total phenols (TP), total flavonoids (TF), proanthocyanidins (PA) and flavans reactive with vanillin (VRF) were determined according to Gambacorta et al., 2011, using an UV–visible spectrophotometer. For flavonoids and anthocyanins determination, wine samples were diluted 25 times with ethanol-hydrochloric acid solution. An adsorbance spectrum in the range of 230–700 nm was recorded. The flavonoids content was calculated according to the following formula:

$$F = E_{280} * df * 82.4$$

E_{280} = Specific extinction coefficient at 280 nm assessed with the graphic method (absorbance corresponding to the segment, parallel to the y-axis, starting from the peak medium at 280 nm and finishing on the tangent joining the points of minimum at the left and the right of the peak).

df = dilution factor

82.4 = value determined considering the ratio between the concentration (expressed as mg L^{-1}) and the corresponding E_{280} of pure (+)-catechin.

The anthocyanins content was determined according to the following formula:

$$F = E_{maximum} * df * 26.6$$

$E_{maximum}$ = specific extinction coefficient at the maximum of visible region (~520 nm) assessed with the graphic method as described before.

df = dilution factor.

26.6 = value determined considering the molar extinction coefficient of an anthocyanins mixture deriving from grapes (Ɛ = 18800, MW medium = 500). The A content was determined using a calibration curve for malvidin-3-glucoside and expressed as mg L^{-1} of malvidin-3-glucoside.

The total polyphenols content was measured at 700 nm using the Folin-Ciocalteu reactive and quantified by the following formula:

$$TP = E_{700}\ V^{-1} * 186.5$$

E_{700} = absorbance at 700 nm.

V = wine volume (1 mL)

186.5 = value determined considering the ratio between the concentration (expressed as mg L^{-1}) and the corresponding E_{700} of pure (+)-catechin.

In the case of flavans reacting with vanillin, absorbance at 500 nm was measured and content was evaluated according to the following formula:

$$FRV = \Delta E * df * 290.8$$

$C = E1 - E0$ (difference between the absorbance assessed at 500 nm of the sample with and without vanillin).

df = dilution factor.

290.8 = value determined considering the ratio between the concentration (expressed as mg L^{-1}) and the corresponding E_{500} of pure (+)-catechin.

The proanthocyanidins content was measured at 532 nm and calculated according to the following formula:

$P = \Delta E\ V^{-1} * 1162.5$

$\Delta E = E1 - E0$ (difference between the specific extinction coefficient assessed at 532 nm with the graphic method of the sample after and before acid hydrolysis by heat).

V = wine volume (1 mL).

1126.5 = value determined considering the ratio between the concentration (expressed as mg L^{-1}) and the corresponding ΔE of pure cyanidin chloride.

Three experimental replicates were analyzed for each wine.

2.3. Analysis of volatile aroma compounds

The volatile compounds were extracted by the solid-phase microextraction (SPME) technique, according to Paradiso et al. (2022). The samples were weighed (1 ± 0.05 mL) into 20 mL vials containing 0.2 g mL^{-1} of NaCl (to increase the ionic strength), closed with a silicone/PTFE septum and an aluminium cap. Semi-quantitation was performed adding internal standard (2-octanol). A mother solution, obtained from the pure standard (Sigma Aldrich, Milan, Italy), was diluted to reach a final concentration of 8.2 mg L^{-1}, then 10 μL of this final dilution was added to the sample. Samples were loaded into an autosampler Triplus RSH (ThermoFisher Scientific, Rodano, Italy). Before extraction, stabilization of the headspace in the vial was obtained by equilibration for 10 min at 50 °C. The extraction was carried out using a divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) 50/30 mm SPME fiber assembly (Supelco, Bellefonte, PA, USA) at 50 °C for 30 min. The fiber was desorbed at 200 °C for 2 min in the injection port of the gas chromatograph, operating in split-less mode. The GC-MS analyses were performed using a Trace1300 gas chromatograph equipped with a mass spectrometer ISQ Series 3.2 SP1. The compounds were separated in a Thermo capillary column VF-WAX MS (60 m, 0.25 mm, 0.25 μm), under the following conditions: injection port temperature, 200 °C; oven temperatures, 40 °C for 0.5 min then 3 °C min^{-1} to 210 °C with a final isothermal for 2 min. Mass detector was set at the following conditions: detector voltage, 1700 V; source temperature, 250 °C; ionization energy, 70 eV; scan range, 33–150 amu. Tentative identification of the peaks was done by means of Xcalibur v2.0 software, in particular, Qual Browser, by matching their spectra with the reference mass spectra of NIST library. Semi-quantitation of the compounds was done by the internal standard method, and the amounts were expressed as μg of 2-octanol equivalents $Liter^{-1}$.

2.4. Sensory analysis

A panel composed of 6 judges (4 males, 2 females; age 35–58), winemakers and professionals, participated at wine evaluation sessions. All judges were experienced in wine tasting and were familiar with Apulian cultivars. One training session was carried out to familiarize with the sensory methodology. The adopted methodology did not require further training, being based on free description (Souza Gonzaga et al., 2019, 2021). In order to provide robustness to the analysis and compensate for the number of panelists, lower than 10–15 as suggested by Souza Gonzaga et al. (2021), three experimental replicates were analyzed for each wine. Samples were coded with 3-digit random numbers and presented in random order at serving temperature (13 ± 2 °C) in glasses complying with the requirements of the ISO 3591 (2) standard (ISO, 1977). Panelists individually evaluated each wine in an open-plan sensory facility and took a 1-min forced break between each wine, while having access to water and plain crackers as palate cleansers. Two sensorial sessions were carried out. In each session two batches of 4 or 5 samples were analyzed, with an interval between batches for rinsing and defatiguing mouth (Jackson, 2017).

Due to the need of exploring the sensory space covered by these wines (Barbe et al., 2021), they were characterized by free-choice profiling: judges evaluated and freely described the characteristics of each wine using a free vocabulary of sensory descriptors, by creating their own list of attributes with no need to explain the significance of the words, neither to limit the number of words. They were only asked to avoid hedonic attributes (Barbe et al., 2021; Souza Gonzaga et al., 2021). The textual data were preprocessed according to Symoneaux et al. (2012): removal of mistakes, elimination of connectors and auxiliary terms, lemmatization, regrouping synonyms, management of ambiguous words (polysemy and homographs). The frequency of occurrence of sensory descriptors was acquired and submitted to statistical analysis.

2.5. Statistical analysis

Two-way Analysis of Variance (ANOVA) with interactions, one-way ANOVA, Tukey's post hoc test, heatmap with cluster analysis and principal components analysis (PCA) of volatile compounds were carried out with Origin Pro 2022 (OriginLab, Northampton, MA, USA). Correspondence analysis (minimum term frequency = 3) and Co-occurrence network (minimum term frequency = 3, filter edges = Jaccard, top 40 edges) were out on the results of sensory analysis using the KH coder software (http://khcoder.net/en/) (Paradiso et al., 2022).

3. Results and discussion

3.1. Chemical analysis

The enological parameters of wines are reported in the Supplementary Table S1. All wines showed complete fermentation of sugars. In spite of the small size of maceration vessels, volatile acidity, though increasing as maceration time increased, remained below the limit of 1.08 g acetic acid L^{-1}, with the exception of one of the M-6 replicates, that reached 1.3 g acetic acid L^{-1}, leading to a mean value of 1.01 g acetic acid L^{-1}. Malolactic fermentation occurred during the second month of maceration, based on the levels of malic and lactic acids.

Fig. 1 reports the results of the analysis of phenolic compounds on Minutolo and Verdeca wines macerated for 1, 2, 4 and 6 months, as well as of the control wines produced by traditional white winemaking.

Fig. 1A shows TP and TF behavior. A marked increase in TP during maceration occurred: Verdeca and Minutolo control wines presented concentrations of 569 and 226 mg L^{-1} respectively, while a maximum of 6670 and 5682 mg L^{-1} was reached in M-4 and V-4 respectively, similarly to those typically found in red wines (Salemnia et al., 2019). These results agree with many studies in literature, such as the one conducted by Bene and Kállay (2019), in which an increase in total polyphenols was reported in wines obtained with 5 weeks maceration, reaching a concentration of 1632 mg L^{-1}. Also Bestulić et al. (2022) and Korenika et al. (2014) reported higher concentrations, in almost all categories of phenolics evaluated in macerated wines compared with traditional ones. This trend is probably the result of increased extraction from the skins that occurred during maceration, assisted by the presence of ethanol produced during fermentation (Lukić et al., 2015). The slight decrease in phenols observed in 6-months macerated samples suggests that, after four months of maceration, phenol extraction was complete. Moreover, this slight decrease, may indicate precipitation or reabsorption reactions of phenols on the skins, which occur at very long maceration times (6 months), as previously described by Baiano et al. (2014) and Recamales et al. (2006)).

Fig. 1B shows the concentration trends of VRF, PA, and the ratio VRF/PA. VRF is an index giving information related to the size of condensed tannins and astringency (Ossola et al., 2017): it consists of flavan monomers and low molecular weight oligomers and is negatively related to oxidative polymerization. These compounds are mainly located in seeds and defined as aggressive tannins as they impart bitter characteristic to wines (Noble, 1994; Picariello et al., 2018). In the

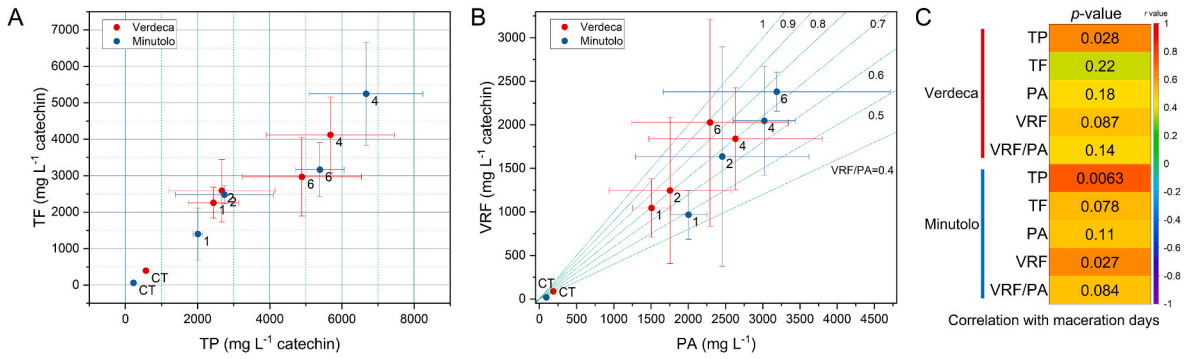

Fig. 1. Indices of phenolic compounds in control and macerated white wines. A: Contents of total phenols (TP) and total flavonoids (TF); B: Proanthocyanidins (PA), Vanillin reactive flavans (VRF) and the ratio VRF/PA; C: correlation between phenolic compounds parameters and maceration time.

current study data show increasing concentrations of VRF starting from value of 19 and 87 mg L^{-1} respectively in Minutolo and Verdeca control wines, to 2026 and 2380 mg L^{-1} in 6-months macerated Minutolo and Verdeca samples. Also, the VRF/PA increased as maceration advanced, starting from values of 0.4 in controls (V-CT, M-CT), up to values of 0.9 in V-6 and 0.7–0.8 in M-6. The simultaneous increase of these two parameters may indicate the progressive extraction of seed phenolics that is promoted by ethanol production (Canals et al., 2005), during prolonged maceration.

Fig. 1.C emphasizes the correlation between total phenols, flavonoids, proanthocyanidins, vanillin-responsive flavans, VRF/PA and maceration time. In general, data of Minutolo wines were more correlated to maceration time compared to Verdeca ones, probably due to a better phenol extractability. As expected, a positive correlation between maceration and TP was observed for both varieties. Furthermore, flavonoids were the most abundant class of phenolic compounds and were closely correlated to TP content ($p < 0.01$, data not shown), which indicates the strong impact of the duration of maceration on the phenolic class of flavonoids, in contrast to traditional white wine where hydroxycinnamic acids are the most representative class of phenols (Reynolds, 2010).

3.2. Volatile composition

Table 1 shows the concentrations of volatiles (acetate esters, branched ethyl esters, straight chain ethyl esters, other esters, C-6 compounds, aldehydes, ethers and acetals, ketones, acids, alcohols, sulfur compounds, terpenes and norisoprenoids) detected in control and macerated wine samples. Influence of cultivar and time variables on the composition of the volatile profile of wines was evaluated by two-way ANOVA. Maceration time had a strong influence on all classes of volatile compounds, while the cultivar variable influenced only some classes of compounds (acetate esters, other esters, C-6 compounds, aldehydes, ketones, acids, terpenes, acetals/ethers and norisporenoids). Interaction between maceration time and cultivar affected all classes of volatiles, indicating different behaviors of cultivars towards maceration.

In the present study, different categories of esters exhibited distinct trends with respect to both maceration time and cultivar variables.

Acetate esters were generally present in higher concentrations in Minutolo wines: their concentration triplicated after 1–2 months of maceration, reaching 1934.9 μg L^{-1}, as respect to control (557 μg L^{-1}) and then decreased in longer macerated samples. Verdeca initially had higher concentration in acetate ester than Minutolo, but this remained constant or at least decreased throughout maceration. Acetate esters, characterized by fruity notes, are fermentative products. A reduction during maceration can be explained by their hydrolysis at the acid pH of wine (Marais, 1978). The main acetate ester in both cultivars was ethyl acetate, related to volatile acidity but associated with pineapple, fruity, solvent and balsamic notes when present at lower levels (Buican et al., 2023). The level of ethyl acetate reached a maximum after 2 months of maceration in Minutolo wine (1706.64 μg L^{-1}) and after 1 month of maceration for Verdeca (1000.79 μg L^{-1}). Also (Lukić et al., 2015) found increased concentrations of ethyl acetate in white wines macerated during and after fermentation.

Ethyl esters, instead, showed the lowest concentration in M-2 and V-2 respectively of 656.4 μg L^{-1} and 619.2 μg L^{-1}. However, after 4 and 6 months of macerations, their concentration increased, reaching the highest value in M-6 and V-6 of 2991.4 μg L^{-1} and 2447.2 μg L^{-1}, respectively. The initial reduction may be due to chemical hydrolysis determined by the acidic pH of the wine, but also adsorption on lees (Lukić et al., 2015). The following increment can be mainly attributed to newly-formed ethyl esters, above all succinate that strongly increased, reaching a maximum after 6 months of maceration, 2539.8 μg L^{-1} and 2086.91 μg L^{-1} respectively in Minutolo and Verdeca.

Branched-chain ethyl esters concentrations did not differ from the control during the early stages of maceration, while they increased in samples of both varieties macerated at 4 and 6 months maybe due to the esterification between ethanol and acids deriving from higher alcohols oxidation (Díaz-Maroto et al., 2005). The maximum concentration was found for ethyl isopentyl succinate, without differences between cultivars. Regarding other esters, they were present in higher concentrations after maceration, expecially, in Verdeca samples, monoethyl succinate that reached a maximum of 122.36 in V-6 μg L^{-1}. Only isoamyl octanoate was present in both control and macerated samples. In Verdeca samples it was at the highest concentration in the control, and then decreased after maceration, while, in Minutolo wines, it reached a maximum after 2 months of maceration. As regards esters detected in macerated white wines, literature reports conflicting results. In the study conducted by Wang et al. (2016), where maceration was applied during white winemaking, throughout the duration of fermentation (approximately 3 weeks), a general increase in esters concentration was observed. On the contrary Lukić et al. (2015) reported a marked decrease in esters, especially straight chain ethyl esters.

Concerning C-6 compounds, Verdeca samples had higher values compared to Minutolo. In both varieties concentrations increased up to 2 months of maceration and then decreased markedly. They are pre-fermentative aromas which derive from membrane lipids via the lipoxygenase pathway. Their presence in wines is not always desired, however, if present in small concentration, they may contribute positively to white wine aroma, conferring pleasant herbaceous notes (Zalacain et al., 2007).

Aldehydes concentration was higher in Minutolo samples and, in both varieties, was variable depending on maceration time. Aldehydes (mostly represented by nonanal) reached a maximum in control samples and then, as maceration progressed, it decreased probably depending on oxidation into the corresponding acids (Waterhouse et al., 2016), except for M-2. Acetaldehyde was present at the highest concentrations after one and two months of maceration, in both varieties, as a results of

oxidation of ethanol, probably occurring in higher extent in the early stages of maceration, with higher availability of dissolved oxygen.

Acids concentration was significantly higher in control samples, compared to macerated ones: V-CT had the highest concentration of 7377.3 μg L^{-1}, followed by M-CT with a concentration of 1552.4 μg L^{-1}. Acids in wines are usually associated with unpleasant odors when present at high levels (Bakker & Clarke, 2011). From the first month of maceration, a marked decrease was observed: concentrations in samples M-1 and V-1 were, respectively, 285.1 μg L^{-1} and 193.6 μg L^{-1}, and remained stable throughout maceration. This decrease could be attributed to reabsorption phenomena on skins and above all lees (Alexandre et al., 1997). The high concentration of acids in control samples was mostly due to octanoic acid in both varieties, and decanoic acid in Verdeca. Acetic acid and nonanoic acid concentrations, instead, increased with maceration. In particular, acetic acid reached 257,96 μg L^{-1} in 6 months macerated Minutolo wines and 181,96 μg L^{-1} in Verdeca one. In fact, volatile acidity resulted higher in macerated samples but still below legal limits (Zoecklein et al., 1990).

Acetals and ethers were detected in macerated samples. Acetaldehyde ethyl amyl acetal probably resulted from mild oxidation processes. Lukić et al. (2015) report an increase of acetals in long macerated white wines.

Alcohols concentration increased in both varieties as maceration progressed, up to two months, and then decreased, remaining the same or above control concentration. Alcohols present in higher concentration both in Minutolo and Verdeca wines were phenylethyl alcohol, isoamyl acohol and isobutanol. The increase observed in the early stages of maceration is in agreement with what was found by Wang et al. (2016) in 3 weeks macerated wines. The following decrease may depend on their adsorption on macromolecules (Aith Barbará et al., 2020) or oxidation into aldehydes and finally acids.

Sulfur compounds concentration showed an increase for both cultivars and, for Minutolo, more than doubled, following maceration, in accordance with the study conducted by Fedrizzi et al. (2012). This increase may be due to both turbidity of the must and increased extraction of amino acid precursors of these molecules, released from the skins during maceration (Karagiannis & Lanaridis, 1999). Their presence is indicative of reductive processes, which, along with the oxidized products found in the same samples, makes it necessary to monitor oxygen supply during maceration and redox equilibria along the profile of the maceration vessel. Methionol was the most representative and was present in both control and macerated samples. The others arose along maceration.

In Minutolo wines, terpenes reached the highest concentration (6189.7 μg L^{-1}) after one month of maceration. The presence of terpenes in Minutolo was expected since it is known to be a semi-aromatic grape variety (Baiano et al., 2015; Paradiso et al., 2022). The main terpenes were linalool and terpineol. The increase in terpenes following maceration was expected as they are located in the skins, along with their precursors, and were extracted into the must during maceration (Wang et al., 2016), especially in the monoterpene glycoside form that is than hydrolyzed into volatile during fermentation (Buican et al., 2023). This behavior agrees with other research (Lukić et al., 2015; Radeka et al., 2008; Wang et al., 2016). According to a classification of wines based on terpenes concentration and proposed by Mateo and Jiménez (2000), this wine can be classified as intensely flavored muscat. While remaining above that of M-CT, as maceration progresses, terpenes concentration decreased, showing in M-6 a 48% reduction compared to M-3. Nevertheless, an increment of the complexity in terpenes composition was also registered in longer macerated samples: in addition to terpenes mentioned above, in M-4 myrcene, hotrienol, a-citronellol and *cis*-geraniol were also found, while in M-6 the presence of two oxidized terpenes (linalool and nerol oxide) was also reported. V-CT had a low terpenes concentration of 29.8 μg L^{-1}, which remained almost unchanged throughout maceration. In 6-months macerated Verdeca wines, beside linalool and terpineol, also *p*-menthan-1-ol was found.

The highest concentration of norisoprenoid compounds occurred in control samples and was attributed to β-damascenone that significantly decreased with maceration. In macerated samples, amounts were lower (and related to the non-cyclic volatile norisprenoid 6-methyl-5-hepten-2-one) or even zero. This result is in agreement with the results obtained by Lukić et al. (2015), who found a reduction in the concentration of norisoprenoids, in particular β-damascenone, in macerated samples.

In the heatmap of Fig. 2, 81 volatiles detected in samples by gas chromatography are reported, used to discriminate wines with different characteristics and grouped those with a similar volatile profile. On the vertical axis, samples are divided into 4 different clusters, while on the horizontal axis 6 main clusters of volatiles were identified. Control wines (V-CT and M-CT) formed a separate cluster as they were markedly different from all macerated samples. They were both characterized by a cluster consisting in aldehydes (octanal and nonanal); several ethyl esters related to fruity character; Phenylethyl acetate that impart flower notes; medium chain fatty acids; two alcohols and a norisoprenoid compound (β-damascenone), a varietal aroma deriving from carotenoids, that is known, although present in small concentrations, to contribute significantly to the aroma definition of wines because of its low perception threshold (Gómez-Míguez et al., 2007), enhancing floral and fruity aromas.

After one and two months of maceration, time variable had a greater influence than cultivar in discriminating samples as it is possible to identify two clusters, the first comprising samples macerated for one month (M-1 and V-1), and the second represented by samples macerated for two months (M-2 and V-2). M-1 and V-1 shared a group of volatiles mainly constituted of fermentation esters. In this cluster there was also an acetal, acetaldehyde ethyl amyl acetal, a marker of oxidation and evolution in wines. It may derive from the reaction between ethanol, pentanol and acetaldehyde, that take place along with a mild oxidation (Lukić et al., 2015). There are also some alcohols and a sulfur compound, methionol, a potent aroma that may be negatively related to pleasant descriptors in wines (Fang & Qian, 2005). M-1 was characterized by two terpenes: linalool and terpineol, both related to floral aromas.

Samples macerated for two months, M-2 and V-2 were different from previous ones being characterized by a cluster of volatiles comprising various alcohols; acetaldehyde; acetate esters; a branched ethyl ester (ethyl isovalerate); hexanoic acid, 2-methyl and 3-hexen-1-ol. Ethyl esters of branched fatty acids characterized all macerated samples as expected, since they are indicative of the evolution of wine aroma (Díaz-Maroto et al., 2005).

Samples macerated for 4 and 6 months were substantially different from both controls and 1–2 months macerated samples, which probably implies that the influence of maceration in discriminating wines becomes more pronounced when maceration times are extended. Furthermore, as maceration progresses, differences between cultivars became more marked, as one cluster consists of Verdeca samples macerated for 4 and 6 months, while Minutolo samples macerated for 4 and 6 months were not clustered together, indicating further evolution of Minutolo wines as maceration progressed. Long macerated wines shared a cluster of volatiles including several esters; thiophene 3,4-dimethyl; benzyl alcohol and hotrienol. Among esters there are some markers of malolactic fermentation, such as ethyl lactate and ethyl 2-hydroxy-isovalerate, as showed also in Table 1. Also isoamyl lactate characterizing V-4 and acetoin characterizing V-6, are indicators of this process. A correlation between white wines maceration and the beginning of malolactic fermentation was also found by Sancho-Galán et al. (2021). Longer macerated Minutolo differed from Verdeca one for two different volatile clusters: the first (in M-6) mainly constituted by ethyl esters, a norisoprenoid compound (the non-cyclic volatile norisoprenoid 6-methyl-5-hepten-2-one) (Meng et al., 2019), an aldehyde (3-furaldehyde) and two oxidized terpenes (linalool and terpineol oxides), which found a correlation between long maceration time and oxidation, also confirmed by Aleixandre-Tudo et al. (2015); the latter (most

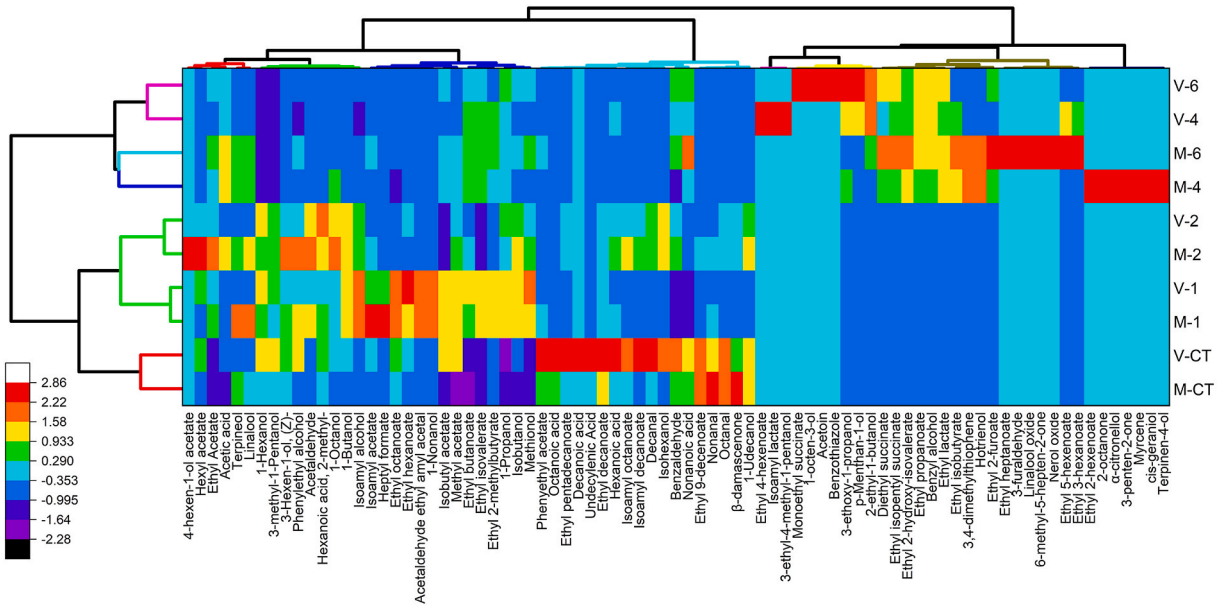

Fig. 2. Volatiles heatmap with hierarchical clustering of control and macerated Minutolo (M) and Verdeca (V) white wines. CT, control wines; 1, 1-month maceration; 2, 2-months maceration; 4, 4-months maceration; 6, 6-months maceration.

representative of M-4) comprising several terpenes (α-citronellol, *cis*--geraniol, myrcene, terpinene-4-ol), two ketones (3-penten-2-one and 2-octanone). A cluster of volatiles including a terpene (*p*-menthan-4-ol) is also observed in the Verdeca samples macerated for 4 and 6 months. Also Wang et al. (2016) found a positive correlation between maceration time and terpenes in wines.

3.3. Sensory analysis

In order to assess the sensory evolution due to prolonged maceration of Minutolo and Verdeca wines, 6 panelists tasted and evaluated control, 4 and 6 months macerated wine samples. Fig. 3 shows the sensory space of samples according to the results of correspondence analysis carried out on the frequency of occurrence of free-choice descriptors (Barbe et al., 2021). Moving along the first dimension (49.35 % of variability), the descriptors are distributed according to cultivars; instead, moving along the second dimension (24.43 % of variability), there are attributes that emerge with maceration. It can be observed that the separation between macerated and unmacerated samples is very clear, and even in terms of cultivar, samples are distributed differently. M-CT was described as terpenic, coherently with the terpene contents reported in the volatile composition, slightly bitter, with hints of varnish and acetone. Verdeca control, on the other hand, turned out to be characterized by yellow flowers and spicy notes. As a result of maceration, wines made from both cultivars were richer in flavor descriptors and more complex. Minutolo wines acquired floral hints and citrus notes that may be attributed to increased concentrations of linalool and geraniol (Buican et al., 2023) found in these wines. The development of bitterness, reported by panelists, can be attributed to the increased concentration in VRF detected in longer macerated samples during phenolic analysis and it has been also confirmed by various authors (Aleixandre-Tudo et al., 2015; Jagatić Korenika et al., 2018; Sancho-Galán et al., 2021; Soares et al., 2020). Verdeca macerated wines gave a good response to prolonged maceration as V-4 presented flowery and fruity notes and apricot and citrus flavor, while V-6 enhanced the apricot notes typical of macerated white wines (Palomo et al., 2007). These fruity notes found in these wines could be related to higher concentrations of newly formed ethyl esters (Buican et al., 2023). In-mouth velvety sensations characterized both V-4 and V-6 wines. On the other hand, M-6 wine was characterized by a tannic character and peach flavor. In both cv fruity notes were observed in macerated samples, in contrast with Sancho-Galán et al. (2021) who observed a decrease in fruitiness character as the percentage of skins used during maceration increased. Overall, the panel judged 4 months macerated samples (M-4 and V-4) as balanced and pleasant, with interesting citrus and floral hints, that were replaced by the stone fruit notes of six-month macerated wines. Lastly, concerning color, Minutolo wines changed from pale yellow in the control, to orangey after maceration, in agreement with Sokolowsky et al. (2015) e da Bestulić et al. (2022). In contrast, Verdeca wines remained clear, straw yellow, even after prolonged maceration.

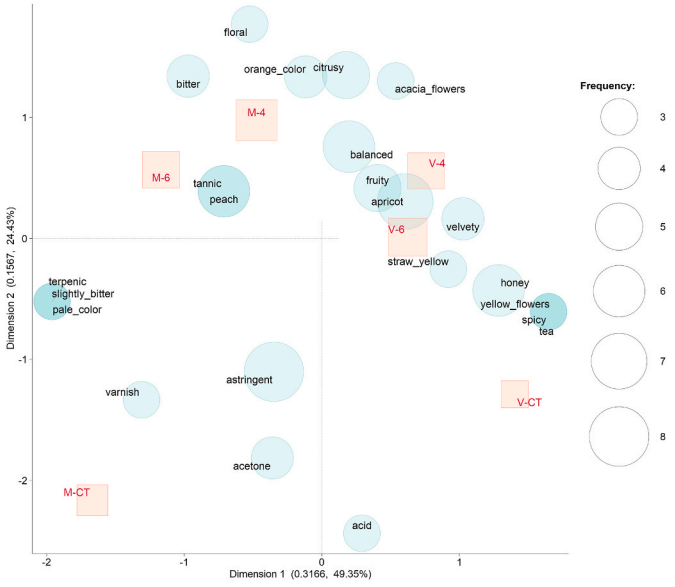

Fig. 3. Correspondence analysis based on the frequency of attribution of aromatic descriptors to Minutolo (M) and Verdeca (V) control samples (V-CT, M-CT), 4-months macerated samples (V-4, M-4), six months macerated samples (V-6, M-6).

3.4. Practical implications, strengths, and weaknesses of this research

The results obtained describe the behavior of two white grape varieties from Apulia (Italy), Verdeca and Minutolo, when used to produce macerated white wines. The different response of these two varieties to varying duration of maceration highlights their potential to provide wines with unique characteristics. This could be a tool for producers aiming either at product diversification (Carbone, 2021), or at linking wine production to territory (Festa et al., 2020). Moreover, this winemaking strategy could be adopted to meet the demand of wines with novel sensory profiles, especially from highly involved consumers (Oyinseye et al., 2022). Also, advantages in terms of exploitation of biodiversity and fight towards genetic erosion could be reached (Miazzi et al., 2020). The evaluation of two local, scarcely known, cultivars, with different technological attitudes, submitted to the same maceration process can be considered a point of strength of this research, as well as the adoption of a suitable approach to explore a novel sensory space. On the other hand, information about the sensory profile of wines with shorter maceration times is still lacking and requires further investigation. Moreover, the present research was carried out on a very small scale and the results should be confirmed on a larger scale of processing. Therefore, further research should be carried out on larger scale productions, to optimize winemaking parameters (e.g. solid/liquid ratio, duration of maceration) as well as to adapt vineyard management to these specific oenological objectives.

4. Conclusions

In this study, maceration applied to white grapes varieties (Minutolo and Verdeca) permitted to obtain wines with unique chemical and sensory profiles compared to white wines obtained with traditional winemaking. In particular, maceration had a significant impact on phenols composition and concentration, that reached a maximum after 4 months of contact with skin and seeds. Furthermore, volatile profile was modified: in macerated samples oxidation and aging compounds appeared that are unusual for traditional white wines. Furthermore, maceration successfully enhanced varietal identity of Minutolo, improving terpenes composition. According to sensory analysis samples presented clearly distinct profiles according to both cultivar and maceration time. Interesting results emerged and highlighted firstly a good attitude of both varieties to undergo maceration and secondly a good response of Verdeca to longer maceration times. However, further studies should be carried out in a larger scale to optimize the duration of maceration and other parameters, such as solid/liquid ratio, in order to exploit their specific sensory properties and avoid possible excess of astringence. It would be also usefule to applicate and evaluate this technique to other local varieties to evaluate their response.

Ethical statement

No human ethics committee or formal documentation process is available. Participants to sensory analysis gave informed consent via the statement "I am aware that my responses are confidential, and I agree to participate in this survey" where an affirmative reply was required to enter the survey. They were able to withdraw from the survey at any time without giving a reason. The products tested were safe for consumption.

CRediT authorship contribution statement

Ilaria Prezioso: Data curation, Formal analysis, Visualization, Writing – original draft. **Gabriele Fioschi:** Data curation, Validation, Writing – review & editing. **Laura Rustioni:** Conceptualization, Writing – review & editing. **Marco Mascellani:** Conceptualization, Investigation, Methodology, Resources, Writing – review & editing. **Giuseppe Natrella:** Investigation, Writing – review & editing. **Pasquale Venerito:** Investigation, Writing – review & editing. **Giuseppe Gambacorta:** Resources, Writing – review & editing. **Vito Michele Paradiso:** Conceptualization, Methodology, Supervision, Visualization, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Acknowledgement

This project received no specific funding. The present study was designed as a part of Ilaria Prezioso's PhD thesis, funded under the PNRR Doctoral Programme, Mission 4, component 1 Ministerial Decree no. 351/2022. Prof. Vito Michele Paradiso is the supervisor of the thesis.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2023.115698.

References

Aith Barbará, J., Primieri Nicolli, K., Souza-Silva, É. A., Camarão Telles Biasoto, A., Welke, J. E., & Alcaraz Zini, C. (2020). Volatile profile and aroma potential of tropical Syrah wines elaborated in different maturation and maceration times using comprehensive two-dimensional gas chromatography and olfactometry. *Food Chemistry, 308*, Article 125552. https://doi.org/10.1016/j.foodchem.2019.125552

Aleixandre-Tudo, J. L., Weightman, C., Panzeri, V., Nieuwoudt, H. H., & du Toit, W. J. (2015). Effect of skin contact before and during alcoholic fermentation on the chemical and sensory profile of South African Chenin blanc white wines. *South African Journal for Enology & Viticulture, 36*, 366–377.

Alexandre, H., Lubbers, S., & Charpentier, C. (1997). Research report: Interactions between toxic fatty acids for yeasts and colloids, cellulose and yeast ghost using the equilibrium dialysis method in a modelwine system. *Food Biotechnology, 11*, 89–99. https://doi.org/10.1080/08905439709549924

Baiano, A., Mentana, A., Quinto, M., Centonze, D., Longobardi, F., Ventrella, A., Agostiano, A., Varva, G., De Gianni, A., Terracone, C., & Del Nobile, M. A. (2015). The effect of in-amphorae aging on oenological parameters, phenolic profile and volatile composition of Minutolo white wine. *Food Research International, 74*, 294–305. https://doi.org/10.1016/j.foodres.2015.04.036

Baiano, A., Varva, G., De Gianni, A., Viggiani, I., Terracone, C., & Del Nobile, M. A. (2014). Influence of type of amphora on physico-chemical properties and antioxidant capacity of 'Falanghina' white wines. *Food Chemistry, 146*, 226–233. https://doi.org/10.1016/j.foodchem.2013.09.069

Bakker, J., & Clarke, R. J. (2011). *Wine: Flavour chemistry*. John Wiley & Sons.

Barbe, J.-C., Garbay, J., & Tempère, S. (2021). The sensory space of wines: From concept to evaluation and description. *A Review. Foods, 10*, 1424. https://doi.org/10.3390/foods10061424

Bene, Z., & Kállay, M. (2019). Polyphenol contents of skin-contact fermented white wines. *Acta Alimentaria, 48*, 515–524. https://doi.org/10.1556/066.2019.48.4.13

Bestulić, E., Rossi, S., Plavša, T., Horvat, I., Lukić, I., Bubola, M., Ilak Peršurić, A. S., Jeromel, A., & Radeka, S. (2022). Comparison of different maceration and non-maceration treatments for enhancement of phenolic composition, colour intensity, and taste attributes of Malvazija istarska (*Vitis vinifera* L.) white wines. *Journal of Food Composition and Analysis, 109*, Article 104472. https://doi.org/10.1016/j.jfca.2022.104472

Buican, B.-C., Colibaba, L. C., Luchian, C. E., Kallithraka, S., & Cotea, V. V. (2023). "Orange" wine—the resurgence of an ancient winemaking technique: A review. *Agriculture, 13*, 1750. https://doi.org/10.3390/agriculture13091750

Canals, R., Llaudy, M. C., Valls, J., Canals, J. M., & Zamora, F. (2005). Influence of ethanol concentration on the extraction of color and phenolic compounds from the skin and seeds of tempranillo grapes at different stages of ripening. *Journal of Agricultural and Food Chemistry, 53*, 4019–4025. https://doi.org/10.1021/jf047872v

Carbone, A. (2021). From flasks to fine glasses: Recent trends in wine economics. *Ital Econ J, 7*, 187–198. https://doi.org/10.1007/s40797-021-00151-6

Darias-Martín, J. J., Rodríguez, O., Díaz, E., & Lamuela-Raventós, R. M. (2000). Effect of skin contact on the antioxidant phenolics in white wine. *Food Chemistry, 71*, 483–487. https://doi.org/10.1016/S0308-8146(00)00177-1

Díaz-Maroto, M. C., Schneider, R., & Baumes, R. (2005). Formation pathways of ethyl esters of branched short-chain fatty acids during wine aging. *Journal of Agricultural and Food Chemistry, 53*, 3503–3509. https://doi.org/10.1021/jf048157o

Fang, Y., & Qian, M. (2005). Aroma compounds in Oregon Pinot Noir wine determined by aroma extract dilution analysis (AEDA). *Flavour and Fragrance Journal, 20*, 22–29. https://doi.org/10.1002/ffj.1551

Fedrizzi, B., Versini, G., Finato, F., Casarotti, E. M., Nicolis, E., & Ferrarini, R. (2012). Evaluation of the impact of an archaic protocol on white wine free aroma compounds. In M. C. Qian, & T. H. Shellhammer (Eds.), *ACS symposium series* (pp. 117–131). Washington, DC: American Chemical Society. https://doi.org/10.1021/bk-2012-1104.ch008.

Festa, G., Riad Shams, S. M., Metallo, G., & Cuomo, M. T. (2020). Opportunities and challenges in the contribution of wine routes to wine tourism in Italy – a stakeholders' perspective of development. *Tourism Management Perspectives, 33*, Article 100585.

Gambacorta, G., Antonacci, D., M L. Faccia, M., Gatta, B., S, P., Coletta, A., & E, L. (2011). Phenolic composition of Aglianico and Nero di Troia grapes and wines as affected by cover cropping and irrigation. *Italian Journal of Food Science, 4*, 381–394.

Giriboni, P. P., Xavier, A., Roque, V., Vargas, G., Souza, F., & Costa, V. B. (2016). Physical and chemical analyzis of Chardonnay wine with different periods of skin contact. *BIO Web Conf, 7*, 1–2. https://doi.org/10.1051/bioconf/20160702021

Gómez-Míguez, M. J., Cacho, J. F., Ferreira, V., Vicario, I. M., & Heredia, F. J. (2007). Volatile components of Zalema white wines. *Food Chemistry, 100*, 1464–1473. https://doi.org/10.1016/j.foodchem.2005.11.045

ISO. (1977). *Sensory analysis - apparatus - wine-tasting glass*.

Jackson, R. S. (2017). *Wine tasting: A professional handbook* (3rd ed.). Elsevier/Academic Press.

Jagatić Korenika, A.-M., Maslov, L., Jakobović, S., Palčić, I., & Jeromel, A. (2018). Comparative study of aromatic and polyphenolic profiles of Croatian white wines produced by cold maceration. *Czech Journal of Food Sciences, 36*, 459–469. https://doi.org/10.17221/448/2017-CJFS

Karagiannis, S., & Lanaridis, P. (1999). The effect of various vinification parameters on the development of several volatile sulfur compounds in Greek white wines of the cultivars batiki and muscat of hamburg. *American Journal of Enology and Viticulture, 50*, 334–342. https://doi.org/10.5344/ajev.1999.50.3.334

Korenika, A.-M. J., Žulj, M. M., Puhelek, I., Plavša, T., & Jeromel, A. (2014). Study of phenolic composition and antioxidant capacity of Croatian macerated white wines. *Mitteilungen Klosterneuburg, 64*, 171–180.

Lukić, I., Jedrejčić, N., Ganić, K. K., Staver, M., & Peršurić, Đ. (2015). Phenolic and aroma composition of white wines produced by prolonged maceration and maturation in wooden barrels. *Food Technology and Biotechnology, 53*, 407–418. https://doi.org/10.17113/ftb.53.04.15.4144

Marais, J. (1978). The efiect of pH on esters and quality of Colombar wine during maturation. *VITIS - Journal of Grapevine Research, 17*. https://doi.org/10.5073/vitis.1978.17.396-403, 396–396.

Mateo, J. J., & Jiménez, M. (2000). Monoterpenes in grape juice and wines. *Journal of Chromatography A, 881*, 557–567. https://doi.org/10.1016/S0021-9673(99)01342-4

Meng, N., Yan, G.-L., Zhang, D., Li, X.-Y., Duan, C.-Q., & Pan, Q.-H. (2019). Characterization of two *Vitis vinifera* carotenoid cleavage dioxygenases by heterologous expression in *Saccharomyces cerevisiae*. *Molecular Biology Reports, 46*, 6311–6323. https://doi.org/10.1007/s11033-019-05072-3

Miazzi, M. M., D'Agostino, N., Di Rienzo, V., Venerito, P., Savino, V. N., Fucilli, V., Ruffa, P., Roseti, V., Pirolo, C., La Notte, P., Montemurro, C., & Taranto, F. (2020). Marginal grapevine germplasm from Apulia (southern Italy) represents an unexplored source of genetic diversity. *Agronomy, 10*, 563. https://doi.org/10.3390/agronomy10040563

Milat, A. M., Boban, M., Teissedre, P.-L., Šešelja-Perišin, A., Jurić, D., Skroza, D., Generalić-Mekinić, I., Ljubenkov, I., Volarević, J., Rasines-Perea, Z., Jourdes, M., & Mudnić, I. (2019). Effects of oxidation and browning of macerated white wine on its antioxidant and direct vasodilatory activity. *Journal of Functional Foods, 59*, 138–147. https://doi.org/10.1016/j.jff.2019.05.025

Noble, A. C. (1994). Bitterness in wine. *Physiology & Behavior, 56*, 1251–1255. https://doi.org/10.1016/0031-9384(94)90373-5

OIV - International Organisation of Vine and Wine. (2020). *Resolution OIV-ECO 647-2020. White wine with maceration*. URL https://www.oiv.int/standards/white-wine-with-maceration.

Ossola, C., Giacosa, S., Torchio, F., Río Segade, S., Caudana, A., Cagnasso, E., Gerbi, V., & Rolle, L. (2017). Comparison of fortified, sfursat, and passito wines produced from fresh and dehydrated grapes of aromatic black cv. Moscato nero (*Vitis vinifera* L.). *Food Research International, MACROWINE 2016 Conference – Macromolecules and Secondary Metabolites of Grapevine and Wine, 98*, 59–67. https://doi.org/10.1016/j.foodres.2016.11.012.

Oyinseye, P., Suárez, A., Saldaña, E., Fernández-Zurbano, P., Valentin, D., & Sáenz-Navajas, M.-P. (2022). Multidimensional representation of wine drinking experience: Effects of the level of consumers' expertise and involvement. *Food Quality and Preference, 98*, Article 104536. https://doi.org/10.1016/j.foodqual.2022.104536

Palomo, E. S., González-Viñas, M. A., Díaz-Maroto, M. C., Soriano-Pérez, A., & Pérez-Coello, M. S. (2007). Aroma potential of Albillo wines and effect of skin-contact treatment. *Food Chemistry, 103*, 631–640. https://doi.org/10.1016/j.foodchem.2006.08.033

Paradiso, V. M., Sanarica, L., Zara, I., Pisarra, C., Gambacorta, G., Natrella, G., & Cardinale, M. (2022). Cultivar-dependent effects of non-*Saccharomyces* yeast starter on the oenological properties of wines produced from two autochthonous grape cultivars in southern Italy. *Foods, 11*, 3373. https://doi.org/10.3390/foods11213373

Picariello, L., Gambuti, A., Petracca, F., Rinaldi, A., & Moio, L. (2018). Enological tannins affect acetaldehyde evolution, colour stability and tannin reactivity during forced oxidation of red wine. *International Journal of Food Science and Technology, 53*, 228–236. https://doi.org/10.1111/ijfs.13577

Radeka, S., Herjavec, S., Peršurić, Đ., Lukić, I., & Sladonja, B. (2008). Effect of different maceration treatments on free and bound varietal aroma compounds in wine of *Vitis vinifera* L. Cv. Malvazija istarska bijela. *Food Technology and Biotechnology, 46*, 86–92.

Radeka, S., Rossi, S., Bestulić, E., Budić-Leto, I., Kovačević Ganić, K., Horvat, I., Lukić, I., Orbanić, F., Zaninović Jurjević, T., & Dvornik, Š. (2022). Bioactive compounds and antioxidant activity of red and white wines produced from autochthonous Croatian varieties: Effect of moderate consumption on human health. *Foods, 11*, 1804. https://doi.org/10.3390/foods11121804

Recamales, Á. F., Sayago, A., González-Miret, M. L., & Hernanz, D. (2006). The effect of time and storage conditions on the phenolic composition and colour of white wine. *Food Research International, 39*, 220–229. https://doi.org/10.1016/j.foodres.2005.07.009

Reynolds, A. (2010). *Managing wine quality: Viticulture and wine quality*. Elsevier.

Rustioni, L., Altomare, A., Shanshiashvili, G., Greco, F., Buccolieri, R., Blanco, I., Cola, G., & Fracassetti, D. (2023). Microclimate of grape bunch and sunburn of white grape berries: Effect on wine quality. *Foods, 12*, 621. https://doi.org/10.3390/foods12030621

Salemnia, S., Garcia-Torres, R., Herman, D., & Fajardo-Lira, C. (2019). Red, white, and orange? A new look into an old wine (P20-007-19). *Current Developments in Nutrition, Nutrition 2019 Abstracts, 3*. https://doi.org/10.1093/cdn/nzz040.P20-007-19. nzz040.P20-007-19.

Sancho-Galán, P., Amores-Arrocha, A., Jiménez-Cantizano, A., & Palacios, V. (2021). Influence of the presence of grape skins during white wine alcoholic fermentation. *Agronomy, 11*, 452. https://doi.org/10.3390/agronomy11030452

Soares, S., Brandão, E., Guerreiro, C., Soares, S., Mateus, N., & de Freitas, V. (2020). Tannins in food: Insights into the molecular perception of astringency and bitter taste. *Molecules, 25*, 2590. https://doi.org/10.3390/molecules25112590

Sokolowsky, M., Rosenberger, A., & Fischer, U. (2015). Sensory impact of skin contact on white wines characterized by descriptive analysis, time–intensity analysis and temporal dominance of sensations analysis. *Food Quality and Preference, 39*, 285–297. https://doi.org/10.1016/j.foodqual.2014.07.002

Souza Gonzaga, L., Capone, D. L., Bastian, S. E. P., Danner, L., & Jeffery, D. W. (2019). Using content analysis to characterise the sensory typicity and quality judgements of Australian cabernet sauvignon wines. *Foods, 8*, 691. https://doi.org/10.3390/foods8120691

Souza Gonzaga, L., Capone, D. L., Bastian, S. E. P., & Jeffery, D. W. (2021). Defining wine typicity: Sensory characterisation and consumer perspectives. *Australian Journal of Grape and Wine Research, 27*, 246–256. https://doi.org/10.1111/ajgw.12474

Symoneaux, R., Galmarini, M. V., & Mehinagic, E. (2012). Comment analysis of consumer's likes and dislikes as an alternative tool to preference mapping. A case study on apples. *Food Quality and Preference, 24*, 59–66. https://doi.org/10.1016/j.foodqual.2011.08.013

Townshend, E. (2018). *Doctoral dissertation: Investigating the phenolic composition of orange wines: Studying the effects of oxidation and skin contact on white wine tannin*. Lincoln University.

Wang, J., Huo, S., Zhang, Y., Liu, Y., & Fan, W. (2016). Impact of various maceration techniques on the phenolic and volatile composition of Chenin Blanc wines. *International Journal of Food Science and Technology, 51*, 2360–2366. https://doi.org/10.1111/ijfs.13215

Waterhouse, A., Sacks, G., & Jeffery, D. (2016). *Understanding wine chemistry*. John Wiley & Sons, Inc. https://doi.org/10.1002/9781118730720

Zalacain, A., Marín, J., Alonso, G. L., & Salinas, M. R. (2007). Analysis of wine primary aroma compounds by stir bar sorptive extraction. *Talanta, 71*, 1610–1615. https://doi.org/10.1016/j.talanta.2006.07.051

Zoecklein, B. W., Fugelsang, K. C., Gump, B. H., & Nury, F. S. (1990). Volatile acidity. In B. W. Zoecklein, K. C. Fugelsang, B. H. Gump, & F. S. Nury (Eds.), *Production wine analysis* (pp. 98–113). Springer. https://doi.org/10.1007/978-1-4615-8146-8_5.