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Aging of Red Wine (*cv*. Negroamaro) in Mediterranean Areas: Impact of Different Barrels and Apulian Traditional Amphorae on Phenolic Indices, Volatile Composition and Sensory Analysis

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Abstract: This study investigated on the impact of different aging containers on the volatile composition and quality of Negroamaro wine, a key variety from Apulia, Italy. Seven vessel types were evaluated: traditional Apulian amphorae (*ozza*), five types of oak barrels (American oak, French oak, European oak, a French + European oak, and a multi-wood mix), galls bottles as control. The impact of the vessels was evaluated after 6 months aging through characterization of phenolic, volatile and sensory profiles. Amphorae allowed a specific evolution of the wine's primary aromas, including as fruity and floral notes, while enhancing volatile compounds like furaneol, which contributes to caramel and red fruit nuances and also 3-methyl-2,4-nonanedione, a key compound related to anise, plum and premature aging, depending on its concetration. This container also demonstrated effectiveness in stabilizing anthocyanin-tannin complexes, supporting color stabilization. Oak barrels allowed to obtain different outcomes in terms of color stabilization, volatile profile, aroma and astringency. French oak exhibited the highest phenolic and tannin levels, enhancing anthocyanin stabilization and color intensity. European oak followed closely, while American oak excelled in color stabilization with tannins less reactive to polymers. Mixed wood barrels showed lower phenolic extraction and the best astringency evolution.

Keywords: Ozza; 3-methyl-2,4-nonanedione; β-damascenone; furaneol; free-choice profiling; terracotta; earthenware

1. Introduction

Aging is a winemaking step that strongly influences wine evolution, involving a series of chemical and physical reactions, that significantly modify wine composition and affect its stability [1–3]. It is possible to choose among several aging materials that have a different impact both on chemical and sensory wines characteristics [3,4]. The choice depends mostly on costs, varietal characteristics and oenological goals [3,5].

Stainless steel is an inert material that release negligible amounts of substances in wine. It is diffused and appreciated, especially on large scale production, as it can process high volumes and ensure high levels of sanitization, pressure resistance, temperature control, easy cleaning and maintenance and the resulting product can be therefore standardized [6,7].

Wood is widely diffused in winemaking as consumers appreciate spicy, toasted and smoked notes that it confers to the wine [1]. Its interaction with wine is related to porosity degree and oxygen transmission rate (OTR) of the barrel staves, that allow micro-oxygenation: introducing controlled amounts of oxygen into the wine, wood promotes color stabilization and improves aroma and texture [8]. It allows an extensive exchange of substances, hydrolysable tannins and volatile compounds

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[9,10], in different amounts based on species, geographical origin, toasting level, age of the barrel, time of contact, etc. [3]. However, barrels have some drawbacks such as a short shelf life, high time and cost of production, difficult sanitization [1].

Finding new aging materials is important to preserve wine quality, differentiate production and give winemaking a higher sustainability. Therefore, increasing attention is being paid to earthenware. It is a material with varying degrees of porosity, mainly based on the raw materials and production technique, that, with a suitable coating, provide several advantages: it enables temperature control and can impart micro-oxygenation benefits, without conferring exogenous volatile and phenolic compounds that may cover wine varietal characteristics [11–13].

The employment of earthenware to produce aging containers such as amphorae or Georgian *qvevri* has also an historical relevance as it was the first material used to produce wine containers [14].Therefore, its revival is nowadays increasingly attracting the wine world [14]. Also in Apulia, a southern Italian region, earthenware vessels were traditionally produced and called ʹʹcapasoniʹʹ or ʹʹozzeʹʹ and they are still used in some area as to produce, stock and transport wine and other foods [11,15–18].

Negroamaro is a variety of *Vitis vinifera* cultivated in *Salento* (southern area of Apulia), whose wines have become important for the Italian wine market [19,20]. Negroamaro is a non-aromatic variety, therefore, it is important to preserve its nature, enhance the stability of its aroma and color as well as improve its sensorial complexity. In particular, the preservation of primary volatile compounds can be a relevant goal as they are at risk of disappearing in grapes and consequently in wines due to global warming [21]. Also tuning the sensory impact of wine tannins has become a primary goal in a climate change scenario, since phenolic maturity of grapes, and particularly of grape seeds, is decoupling with technological maturity and the resulting wines can be characterized by undesirable astringency subqualities and/or bitterness [22,23]. Finally, colour appears as another critical issue for red wines produced in warm climate, due to reduced anthocyanin accumulation, extraction and stability [24].

In this view, the effect of five different types of barrels and a traditional Apulian amphora (called *ozza*) was evaluated on a Negroamaro wine. Colour and phenolic indices, volatile profile and sensory characteristics were analysed and compared after 6 months of aging.

2. Materials and Methods

2.1. Aging Experiment

The Negroamaro wine used for the trial (vintage 2021) was provided by the winery Vecchia Torre (Leverano, Italy). The wine had 14.1% ethanol, pH 3.6, total acidity 6.7 gL^{-1} tartaric acid, total SO_2 126 mg L^{-1}. Wine was distributed into five types of 225 L barrels (American oak, French oak, European oak, Mix, French+European oak) as described in Table 1, provided by Toneleria Nacional Italy s.r.l. (Prato, Italy) and traditional Apulian amphorae (ozze) having a capacity of 150-200 L (Figure S1). Amphorae were internally glazed according to the traditional manufacturing process, closed with ceramic dishes and sealed with lime [17]. Three barrels per each type and two amphorae were used for the experiment. Dark glass 0.75 L bottles were used as control. *Aging* was carried out for six months.

Table 1. Characteristics of different types of barrels used for the aging (TN Coopers, 2021-2022).

Name of the barrel	Characteristics	
Mix	Blend of French oak (*Q. petraea* (Matt.) Liebl.), American oak (*Q. alba* L.), European oak (*Q. petraea* (Matt.) Liebl.), acacia (*Robinia pseudoacacia* L.),	Dried up to 48 months. Fine/extra fine grane

	lenga (*Nothofagus pumilio* (Poepp. & Endl.) Krasser)	
America oak	*Q. alba* L.	Air dried up to 48 months; fine grane; mature woods over 90 years old
French oak	*Q. petraea* (Matt.) Liebl.	PEFC certification; dried up to 36 months; fine grane; mature woods over 180 years old, cultivated with the Haute Futaie technique (tall trunk)
European oak	*Q. petraea* (Matt.) Liebl.	PEFC certification; fine grane; Air dried up to 48 months
French-European oak	Blend of French oak (*Q. petraea* (Matt.) Liebl.), European oak (*Q. petraea Q. petraea* (Matt.) Liebl.).	Dried up to 48 months; fine/extra fine grane.

2.2. Chemical Analysis

Chemical analyses of samples were conducted using FTIR Winescan FT 120 (Foss, Hillerød, Danmark) analyzer. Color parameters were determined with the modified Somers method [25]. Total phenols were analysed by Folin-Ciocâlteu assay [26], free and total anthocyanins using Di Stefano and Cravero method [27], total tannins with Ribereau-Gayon and Stonestreet method [28], tannins-anthocyanins complex with Glories method [29]. Astringency was evaluated with methylcellulose precipitation assay (MCPT) [25].

2.3. Analysis of Volatile Compounds

Volatile compounds were analysed and quantified by solid phase microextraction-gas chromatography/mass spectrometry (SPME-GC/MS) according to Lukić and Horvat (2017). Odour activity values (OAV) were determined for volatile compounds as the ratios between the measured concentration and the odour threshold reported in literature [20,31–57] (Table S1).

2.4. Sensory Analysis

Free choice profiling was carried out to characterize the wines [26]: judges freely assessed and described the characteristics of each wine using a free vocabulary of sensory descriptors, with the only request being to avoid the use of hedonic descriptors [58,59]. A panel composed of 8 judges (5 males, 3 females; age 23-48), winemakers and professionals with experience in wine tasting and knowledge of Apulian cultivars, participated at wine evaluation sessions. One training session was carried out to familiarize with the sensory methodology and further training was not required as the method was based on free description [26,59,60]. The experimental replicates were independently analysed. Samples were coded and presented in random order in glasses complying with the requirements of the ISO 3591 (2) standard [61], at serving temperature (17 ± 2 °C). Panellists individually evaluated each wine in an open-plan sensory facility with a forced 1-min break between each wine, with water and plain crackers available to cleans the palate. Two sensorial sessions were carried out. In each session two batches of 4 or 5 samples were analysed, with an interval between batches for rinsing and de-fatiguing mouth [62].

The textual data were pre-processed according to [26], removing mistakes, eliminating connectors and auxiliary terms, lemmatizing, 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

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One way and Two-way Analysis of Variance (ANOVA), Tukey's post hoc test, heatmap with cluster analysis, Fisher's LSD test and Principal Components Analysis (PCA) 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/) [63].

Principal components analysis (PCA), partial least squares discriminant analysis (PLS-DA, with 5-fold cross-validation) and heatmap clustering with Euclidean distance (Ward method), were carried out using the MetaboAnalyst6.0 platform (www.metaboanalyst.ca).

3. Results

3.1. Phenols

Table 2 shows the phenolic indices, MCPT assay and color parameters of samples at initial time (T-0) and after 6 months of *aging* in different vessels. The phenolic and color indices evidenced different interactions between the *aging* materials and the wine. As expected, the contact with wood led to a release of phenolic compounds, mostly ellagitannins [64], as shown by the higher levels of total phenols and total tannins compared to the others. Among woods, French oak held the highest values of total phenols (86.30 a.u.), followed by European oak, French+European oak and finally American oak. A greater concentration of total phenols in French oak barrels, compared to American oak ones, was also reported by [65,66]. As a consequence, an increase in tannin value occurred during *aging*, reaching a maximum in American and French oak. The Mix barrels determined lower total phenols concentration compared to other woods, maybe due to a lower release of ellagitannins, also confirmed by a low total tannin value (3.27 gL^{-1}), that could also explain the low astringent perception reported during sensorial analysis for this sample [67].

Table 2. Phenolic composition and color indices of the wine at time 0 (T-0) and of the wines obtained using different aging materials. Results of multiple comparisons among aged wines after one-way ANOVA are reported*.

	T-0	*p*	Glass	Amphora	Mix	Fre-Eur oak	American oak	European oak	French oak
TP (U.A.)	79.77	0.00	79.25±0.21^{e}	81.3±0.28^{cd}	80.83±0.60^{d}	84.3±0.46^{b}	82.13±0.06^{c}	84.7±0.20^{b}	86.3±0.00^{a}
TA (U.A.)	20.1	0.00	16.3±0.00^{d}	18.35±0.07^{b}	17.53±0.06^{c}	18.4±0.17^{b}	16.03±0.15^{d}	18.9±0.10^{a}	17.67±0.15^{c}
TT ($g*L^{-1}$)	2.98	0.00	3.05±0.07^{c}	3.15±0.07^{c}	3.27±0.06^{bc}	3.43±0.06^{ab}	3.63±0.12^{a}	3.47±0.06^{ab}	3.57±0.06^{a}
FA (U)	13.5	0.00	9.2±0.00^{ab}	9.65±1.20^{ab}	9.1±0.1^{b}	10.13±0.06^{a}	5.43±0.06^{c}	10.23±0.12^{a}	8.67±0.06^{b}
A-T (U)	3.8	0.03	4.1±0.00^{c}	5.1±0.71^{b}	4.87±0.06^{b}	4.87±0.06^{b}	6.33±0.06^{a}	4.93±0.06^{b}	5.33±0.06^{b}
free/cond A	3.6	0.00	2.24±0.00^{a}	1.93±0.50^{ab}	1.87±0.04^{ab}	2.08±0.04^{a}	0.86±0.00^{c}	2.07±0.02^{a}	1.63±0.02^{b}
MCPT (mg L^{-1})	236	0.04	234.91±15.54^{a}	238.60±9.81^{a}	240.14±22.18^{a}	256.59±^{a}	250.11±11.46^{a}	257.37±19.14^{a}	273.24±5.28^{a}
CD	11.12	0.01	10.91±0.19^{b}	13.00±1.62^{a}	12.91±0.22^{a}	12.86±0.31^{ab}	13.82±0.42^{a}	12.67±0.37^{a}	12.89±0.41^{a}

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Hue	0.84	0.00	0.86±0.01[a]	0.80±0.04[b]	0.80±0.00[b]	0.79±0.01[b]	0.76±0.03[b]	0.80±0.00[b]	0.79±0.01[b]

*, Tukey's HSD test for multiple comparisons. Different letters indicate significantly different means ($p < 0.05$).

As expected, wine in bottles showed the lowest level of total phenols and total tannins as in the starting wine. On the other hand, amphora had a concentration in total phenols significantly higher compared to glass bottles. This could imply a good protection against degradation of anthocyanins, as shown below. The MCPT assay is related to condensed tannins and the tannin-polymer interaction [25]. Its values were not affected by different *aging* materials according to the Tukey's HSD test, due to a certain variability of data among replicates. However, the Fisher's LSD test indicated that the MCPT assay values of French oak were higher than those found in bottles, amphora and Mix barrels (data not shown). MCPT assay and total tannins showed a linear relation (adjusted $R^2 = 0.578$, p-value = 0.029, see supplementary materials). The linearity consistently increased excluding the data from American oak (adjusted $R^2 = 0.887$, p-value = 0.003, see Figure 1), presenting unexpectedly lower MCPT assay value, compared to the observed total tannins content. Therefore, American oak could have released tannins with lower reactivity towards polymers.

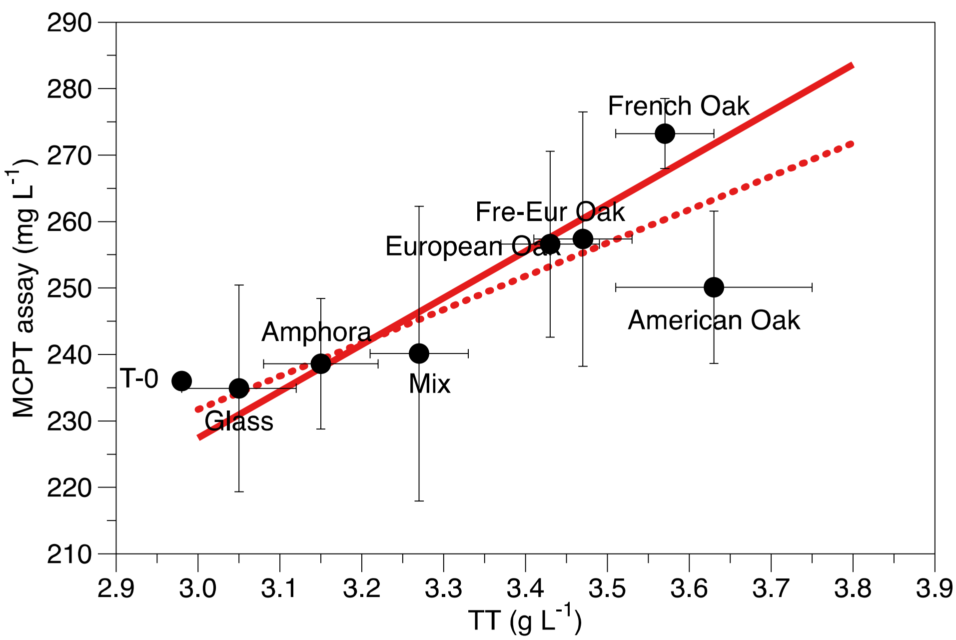

Figure 1. Linear regressions of TT (Total Tannins) concentration (g L^{-1}) and MCPT assay's results (mg L^{-1}).

Free anthocyanins diminished after *aging* in all theses, as a result of condensation as well as degradation phenomena [68,69]. The decrease was less intense in European oak, French and European oak and secondly in amphorae. Also [70] found a higher concentration of anthocyanins in wine aged in unglazed pottery vessels compared to wines aged in other vessels, emphasizing a good behavior of amphorae in protecting anthocyanins. Concurrently with the decrease in anthocyanins, an increase of anthocyanins-tannins complexes was observed in all theses. French oak and American oak showed the highest condensation activity. Consequently, the ratio between free and complexed anthocyanins diminished, with American oak holding the lowest value. Therefore, American oak appeared to release tannins with a marked attitude towards color stabilization rather than interaction with macromolecules. These results, together with high value of color density and low hue, confirmed a good attitude of American oak in stabilizing color as demonstrated by Hernández et al. (2007). Interestingly, anthocyanin-tannins complexes concentration increased also in amphora, still emphasizing a good capacity of earthenware vessels to stabilize color [72]. This attitude was also

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confirmed by the color indices [25]; the high value of color density and the low value of hue, indicated an improvement of chromatic characteristics of wine [73]. This behaviour is possibly due to the micro-oxidation phenomena that occurs throughout earthenware vessel walls, even in glazed terracotta, that promote the formation of anthocyanins-tannins complexes [70,74–76].

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3.2. Volatile Compounds

The evolution of the volatile pattern after aging has been analysed by the PCA reported in Figure 2. PC1 explained 37.3% of the variability between samples. Along this dimension, differences were mainly due to the aging time, as T-0 was clearly separated from the other samples. As expected, T-0 was characterized by primary aromas, es. several terpenes (geraniol, nerol, linalool, cis-nerolidol), together with fermentation products, such as ethyl esters and acetate esters. At time 0 wine was also characterized by higher alcohols and lactones, such as δ-undecalactone, γ-octalactone and γ-decalactone that may confer coconut, fruity-sweet aroma [77–79]. Acids, that generally represent 10% of Negroamaro volatile fraction (Capone et al., 2013; Tufariello et al., 2012), were also abundant. Among acids, most representative were short chain fatty acid such as propionic and isobutyric acids, followed by medium chain fatty acids (hexanoic acid and octanoic acids). After aging, wines volatile profile significantly changed, as a result both of wine oxidative evolution (e.g., acetaldehyde, benzaldehyde) [80,81] and esterification phenomena (e.g., ethyl lactate), together with the emergence of woody aromas released from the barrels staves, such as vanillin, eugenol, isoeugenol, furanic compounds etc. [82,83]. Along PC2 (15,6% of variability), samples are distinguished according to the aging vessels. Interestingly, wines aged in Mix barrels were grouped together with wines in glass bottles, while the other were grouped together, even though they spread along the axis. Amphorae, which behaved differently along this principal component, were grouped each to a different group. This highlights the expected heterogeneity of handmade amphorae [74]. The PC2, therefore, highlighted a slight shift (15,6% of data variability) in the volatile pattern towards the lower-left quadrant, with decreasing impact of wood and oxidation volatiles and a relative increase of the role of esters, alcohols, volatile phenols. This trend characterized glass and one of the amphorae, as expected, and wines aged in French oak barrels, that seemed to provide better modulation of oxidative evolution of the volatile pattern.

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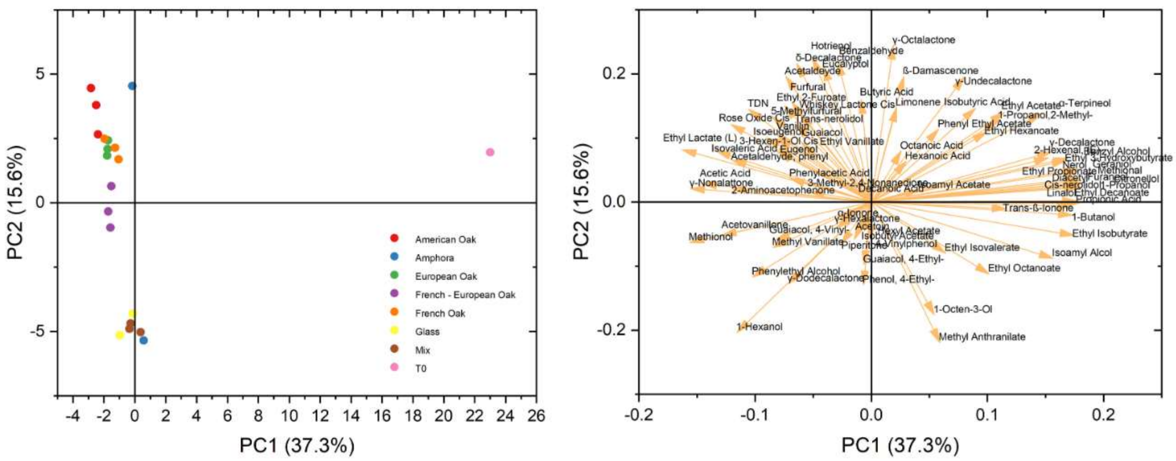

Figure 2. Principal component analysis based on OAV of volatile compounds find in Negroamaro wines, before aging (T-0) and after aging in different materials (Glass, Amphora, Mix, French, European oak, American oak, European oak, French oak).

3.2.1. Discrimination Among Woods

After six months of aging, differences among the volatile aroma profiles of samples aged in different barrels emerged, according to [84] that demonstrated during the first months of aging, extraction of aromas from wood to wine reach a maximum. In order to underline these differences, a further elaboration of volatile data found in wood aged Negroamaro was carried out. Figure 3 reports the heatmap with clusterization of wines and volatiles. The heatmap that was based on 80 volatile compounds found throughout gas-chromatography, clusterized samples into 5 different groups corresponding to the different types of barrels. This confirms the consistency and repeatability of the impact of the type of barrel on the volatile profile of the Negroamaro wine [1,84,85].

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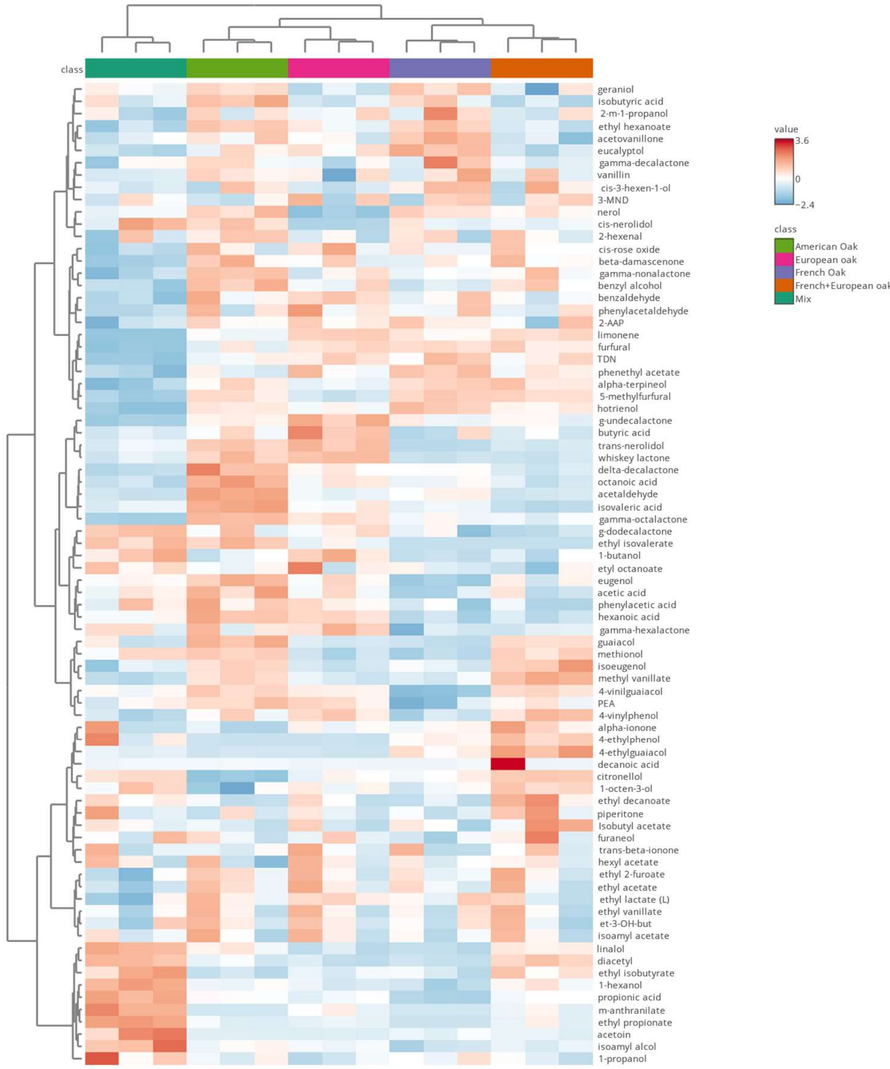

Figure 3. Heatmap with clusterization of the volatile compounds in Negroamaro wine aged in different wooden barrels. (B) Selected volatile compounds with key markers. (C) Partial Least Squares Discriminant Analysis (PLS-DA) highlighting the differences among samples.Abbreviations for volatile names: 2-m-1-propanol (2-methyl-1-propanol); PEA (phenylethyl alcohol); 3-MND (3-methyl-2,4-nonanedione); 2-AAP (2-aminoacetophenone); ethyl 3-hydroxybutyrate (et-3-OH-but); m-anthranilate (methyl anthranilate); TDN (1,1,5-trimethyl-1,2-dihydronaphthalene).

Wine aged in Mix barrels resulted with a volatile profile quite different respect to the othe types of barrel. Mix was characterized from a cluster of volatiles that, in addition to fermentation products (alcohols, acids and some esters), includes higher levels of methyl-anthranilate, typical of foxy character, linalool, with flowery notes and previously reported in Negroamaro wines [20] as well as acetoin and diacetyl, maybe resulting from lactic acid bacteria activities [86]. Also γ-dodecalactone, a lactone typically found in grapes and generally associated to pleasant odours [78] and esters such as ethyl isovalerate and ethyl octanoate resulted as characteristic compounds for wine aged in Mix barrels. American oak was characterized by cluster of grapes lactones including γ-octalactone, δ-decalactone and whiskey lactone which derives from wood and is involved in modifying the perception of fruity aromas, with sweet, peach and coconut notes [42], decreasing red fruits notes and increasing blackberry fruits and spicy scents [77]. In literature, a higher concentration of oak

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lactones in American oak, compared to European oak, was reported [84]. Several volatile phenols were associated to American oak. They may be the result of lignin degradation [87]. Between them, isoeugenol and guaiacol may be related to a positive influence on aroma profile (spicy, vanilla, woody notes), while, 4-vinylguaiacol, if present in high concentration, may confers unpleasant notes [88] although, in this sample, it was present with very low OAV (0.00128) (data shown in supplementary table). Figure 3 highlighted more intense oxidation processes occurring with American oak, as it was characterized by acetaldehyde, the major oxidation by-product of the Fenton oxidation [89], phenyl-acetaldehyde whose correlation with aged and oxidated wines was confirmed by several studies [90–92] and lastly acetic acid. Samples aged in European oak were clusterized as the most similar to American oak, though with less intense signals for lactone and wood-derived compounds compared to American oak. Nevertheless, in the heatmap also whisky-lactone was attributed to this sample, underlining the impact of the oak, with a maximum OAV (7.76) in European oak similar to American oak. European oak was marked by compounds such as limonene, *trans*-nerolidol are highly characterizing, together with eucalyptol. The Negroamaro wine aged in French oak barrels showed, instead, low levels of volatile phenols and lactones compared to the other types of barrels. French oak profile in the heatmap includes two main cluster. The first one comprises two terpenes (α-terpineol and hotrienol), TDN, phenyl ethyl acetate, furfural, 5-methylfurfural. Furfuryl aldehydes are important compounds in oak-aged wines [93]; they impart almonds and toasted almond notes and their presence are found in higher concentration in French oak compared to American oak [94]. The other cluster includes, beside fermentation products, also terpenes (geraniol, eucalyptol), a ketone (acetovanillone), an aldehyde (vanillin) and a lactone (γ-decalactone). Vanillin and its derivatives are wood compounds that confer notes of vanilla, typically reported into wood aged wines aged [94], also confirmed in the present study by sensorial analysis. Finally, French+European oak increased the relative levels of volatile phenols and guaiacols. French+European oak was characterized from a group of aromas comprising piperitone, furaneol, citronellol, β-ionone, α-ionone, 4-ethyl phenol and 4-ethyl guaiacol. Some of them are primary aromas associated to positive notes such as piperitone, a terpene reminiscent of mint [50] that may explain the balsamic notes found in this sample during sensory analysis. Furaneol and β-ionone, both associated to red fruits aromas [32,95]. 4-ethyl phenol and 4-ethyl guaiacol can have a negative influence on wine aroma and may imply contamination by *Brettanomyces/Dekkera* yeasts [87]. Lastly, 1-octen 3-ol resulted as a distinctive compound in French-European oak, that may derive from vine or grape metabolism and associated to mushroom off-flavour in wine [96]. In spite of this, mushroom defect was not reported in this sample during sensory analysis.

Figure 4 reports the results of PLS-DA analysis, including the loading plot of the first two components of the model (Figure 4A), the scores plot of the first two principal components with the 95 % confidence ellipses (Figure 4B) and the plot of the most important variables in the model according to the weighted sum of absolute regression coefficients (Figure 4C). Samples were clearly discriminated on the plane of the first two components (48 % of cumulative variability, Figure 4B), with no overlap of the confidence ellipses.

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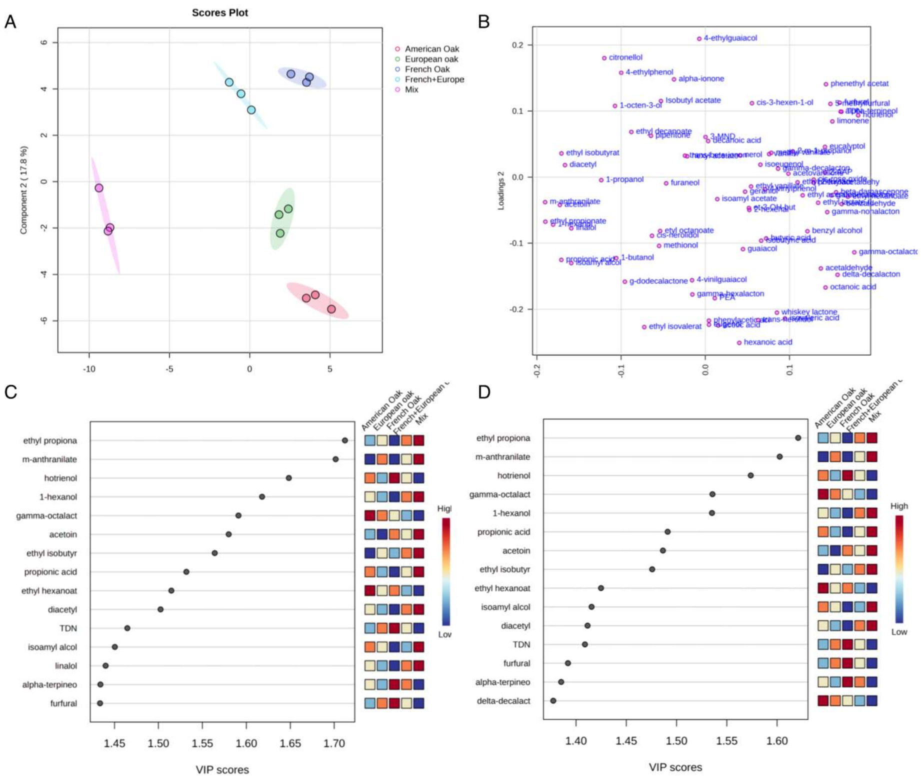

Figure 4. Partial Least Squares Discriminant Analysis (PLS-DA) of the volatile compounds in Negroamaro wine aged in different wooden barrels. (A), Scores plot with 95 % confidence regions; (B), Loading plot of the variables; (C), Most important variables in the model according to VIP score for component 1; (D), Most important variables in the model according to VIP score for component 2 (the colored boxes on the right indicate the relative concentrations of the corresponding metabolite in each group under study). Abbreviations for volatile names: 2-m-1-propanol (2-methyl-1-propanol); PEA (phenylethyl alcohol); 3-MND (3-methyl-2,4-nonanedione); 2-AAP (2-aminoacetophenone); ethyl 3-hydroxybutyrate (et-3-OH-but); m-anthranilate (methyl anthranilate); TDN (1,1,5-trimethyl-1,2-dihydronaphthalene).

In PLS-DA French oak and French+European oak were close but still separated. Similarities are probably due to the presence, in both samples, of French wood. Wine aged in Mix barrels resulted as the most clearly discriminated. Interestingly, the most important variables involved in the discrimination of wines (Figure 4 C and D) are only in minor extent related to wood-derived compounds. The majority of variables with the highest VIP score, in fact, are esters and variateal aromas, including terpenes (linalool, hotrienol, α-terpineol), norisoprenoids (TDN) and varietal esters (methyl-anthranilate). Therefore, the most relevant difference among the types of barrels on Negroamaro wine aging was observed in the modulation of varietal and fermentative volatiles, more than on the release of wood-derived volatiles.

3.2.2. Focus on Some Grape-Derived Compounds

The present section focuses on the fate of some relevant grape-derived compounds after aging of the Negroamaro wine in vessels of different materials.

3.2.2.1. 3-methyl-2,4-nonanedione

In Negroamaro wines tested in this study 3-methyl-2,4-nonanedione was present in concentration higher than its odour threshold (0.016 μg L^{-1}). Pons et al. (2013) found that its aromatic characteristics depends on its concentrations; a range of 0,09-0,17 μg L^{-1} is related to hints of mint and

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anise, in the range of 0,17-0,25 μg L^{-1} plum, 0,25 μg L^{-1} determine fig nuances, finally, over 0,33 μg L^{-1} determine rancid odor. At time 0, 3-methyl-2,4-Nonanedione concentration in Negroamaro wine was 0.016 μg L^{-1} while, after six months increased in each sample (Figure 5). This behaviour was expected since 3-methyl-2,4-nonanedione is a β-diketone originated from ketols oxidation during aging, implying the early aging, with the loss of fresh, fruity and varietal character and the development of dried fruit flavours and cooked fruit nuances [98,99].

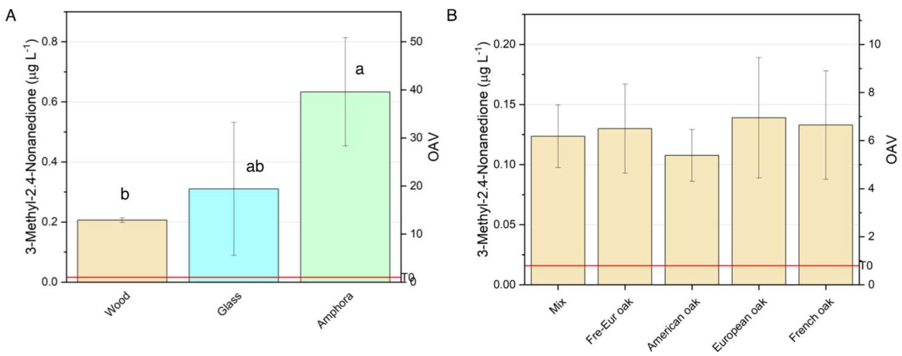

Figure 5. 3-methyl-nonanedione concentration in samples aged in wood, glass and amphora (A); 3-methyl-nonanedione concentration in samples aged in different types of barrels (B). Different letters mean significant differences at $p < 0.05$. The red line indicates the concentration of the compound before aging (T0).

Figure 5 discriminates aged samples in different materials, in terms of 3-methyl-2,4-nonanedione. Panel A reports the mean values found in wines aged in different materials, and the highest average concentration was observed in aged wines in amphora. High values reported in amphora samples were probably due to the oxidative character explained by one of the two amphorae used, which presented a different coating that might induce an higher permeability to gases [74]. Also in glass samples concentrations were higher than those found in barrels as the oxygen in the gaseous headspace of the bottle, could have been rapidly consumed by oxygen reactive species [100]. Although higher than those of T0, 3-methyl-2,4-nonanedione concentrations in all aged wine samples were in the range related to plum notes and not to unpleasant odours, as exception of the amphora with a more oxidative character whose concentration (0,467 μg L^{-1}, data shown in supplementary table) can related to oxidative odor, according to Peterson et al., 2020. No significant difference was found comparing the types of barrels (Figure 5 B), even though quite higher levels were found in all cases compared to T-0 wine. This suggests that formation of 3-MND from precursors could occur during aging [98], and that wood could have led to further reaction or adsorption of this compound.

However, the levels of 3-MND found in the Negroamaro wine evaluated in this study were quite high compared to the range reported in literature [97]. This finding suggests to carry out further research on the occurrence and fate of this compound in Negroamaro wines, due to its role in premature aging of red wines [97].

3.2.2.2. Furaneol

In this study, significant concentrations of furaneol, all widely above its perception threshold (5 μg L^{-1}), were found in all samples. Higher concentrations are reached in amphora and glass, amounting to 110.5 and 111.5 respectively, while barrels accounted similar values ranging from 24.90 to 41.07 μg L^{-1} (Figure 6). Furaneol is the major furan found in strawberry, generated from sugar degradation with a temperature-dependent process [88]. In some cases, the origin of this molecule in wine has been attributed to wood [101], anyway, in this case, due to the presence of high concentration even in absence of wood aging (glass and amphorae), the first hypothesis can be discarded. This compound has therefore a varietal origin and is found in several Italian red grape

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varieties; It can enhance fruity notes of some wines, giving aromas that vary from red fruits (e.g.,: strawberry) to cooked fruit and intense caramel, depending on its concentration [95,102]. Aging in amphora could, therefore, considered as a tool to preserve such varietal compounds in aged wine.

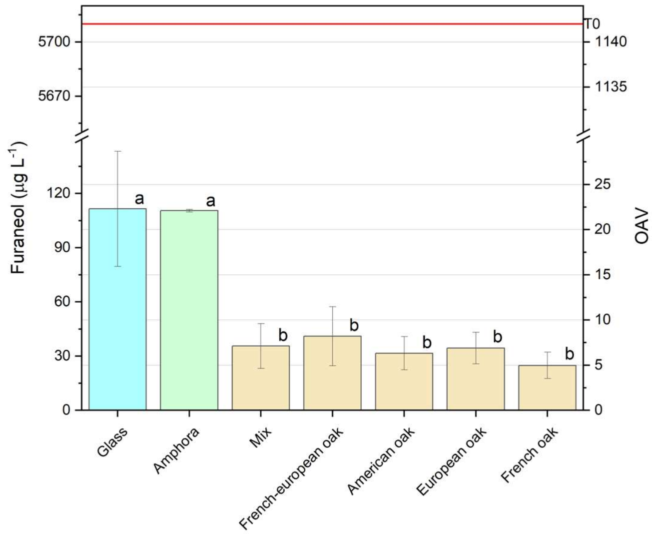

Figure 6. Furaneol concentrations (mean ± S.D.) in Negroamaro wine aged in different containers. Different letters mean significant differences at $p < 0.05$. The red line indicates the concentration of the compound before aging (T0).

3.2.2.3. β-Damascenone

β-damascenone is a C-13 norisoprenoid, carotenoid-derived aroma compound. This ketone can be associated to honey-like, flowery aromas and is considered to enhance fruity aromas [103]. Its presence is due to degradation of neoxanthine carotenoid compound or to glycosylated precursors by acid-catalyzed conversion of megastigma-6,7-diene-3,5,9-triol and megastigma-5-ene-7-yne-3,9-diol, derived-compound of lutein [104,105]. Concentrations of β-damascenone in red wines range between 0.5 – 4 μg L^{-1} with a threshold of 0.05 μg L^{-1} in model wines [106,107]. Different studies showed some characteristics of β-damascenone and observed in red wines an enhancing of some fruity aromas, hiding herbaceous aroma of isobutyl methoxypyrazine, which could suggested an indirect sensorial impact rather than a direct effect on red wines sensorial bouquet [40]; in young white wines it showed scarce effect on aroma [105]. Figure 7 reports β-damascenone concentrations in the Negroamaro wine before and after aging in different types of vessels. In all cases, 6-months aging reduced its concentrations in wines. This trend is confirmed in literature [106,108], where a decrease was observed at the end of the aging (both models and real wines). This could be due to the diverse precursors of grape varieties and to the acid-catalyzed reaction degrading the C13-norisoprenoid itself. Specifically, the amount of β-damascenone is decreased with the greatest reduction by 44% in Mix wood barrel samples, while, in opposite, American oak sample was pretty similar to control, with a reduction of 3%. Glass and Amphora showed similar amount, with a 21% and 16% of decrease respectively. One way-ANOVA showed significant differences between American oak and Mix wood barrel samples ($p < 0.05$).

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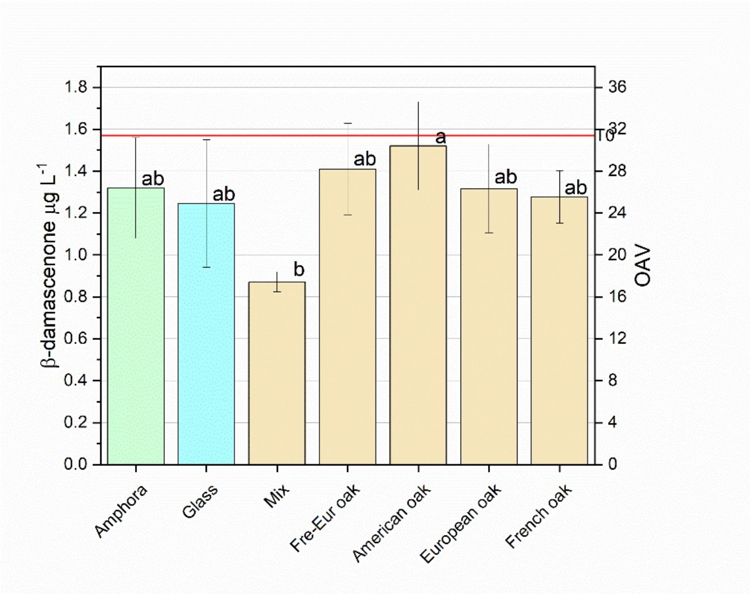

Figure 7. β-damascenone concentrations (mean ± S.D.) in Negroamaro wine aged in different containers. Different letters mean significant differences at $p < 0.05$. The red line indicates the concentration of the compound before aging (T0).

3.2.2.4. TDN

TDN (1,1,5-trimethyl-1,2-dihydronaphthalene) is a C13-Norisopenoid compound which typically characterize aged wines (particularly aged Riesling wines) [53,109]. If present in high amount, it typically confers kerosene or petrol-like aromas, masking other sensorial compounds, which it not suits for consumers [110,111]. The most common concentration in European wines ranges between 5 and 50 µg L^{-1} but it's not unusual to find 250 µg L^{-1} in Australian wines [109]. TDN is found at low levels in grapes and young wines, while its levels increase during wine aging, as the result of the progressive hydrolysis of glycosidic odorless precursors [110]. However increasing levels have been reported in the last decades and have been related to climate change [110]. Its content in grapes is generally low, although, different viticulture techniques, such as defoliation (direct effect) or water irrigation (undirect effect), can affect the formation of TDN in wines [112,113]. The perception threshold is constantly under studies due to differences in sensory impact; it is currently reported at 2 µg L^{-1} [53,114]. Figure 8 reports the amount of TDN after aging in different vessels, compared with the level found before aging. In all cases TDN increased in all theses with the high peak in French oak, increasing by 274%, and low peak in Mix, increasing by 77%. All other theses are similar, with an increase concentration in the range of 206 - 261%. This trend confirms previous mentioned works where wine aging affects increasing of concentration of TDN. The comparison between 6-months aged wines showed no differences among French oak, European oak, Fre-European wood barrel samples and Amphora sample, while Mix sample showed significant differences as compared to all other theses. Others sample, such as Glass and American oak wood barrel showed central values.

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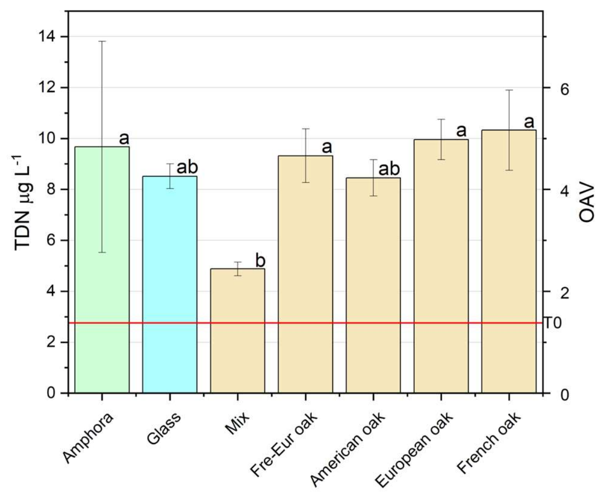

Figure 8. TDN (1,1,5-trimethyl-1,2-dihydronaphthalene) concentrations (mean ± S.D.) in Negroamaro wine aged in different containers. Different letters mean significant differences at $p < 0.05$. The red line indicates the concentration of the compound before aging (T0).

3.3. Sensory Analysis

Figure 9 reports the sensory space of samples deriving from the results of the correspondence analysis carried out on the frequency of occurrence of free-choice olfactory descriptors of wines at time 0 and after aging in different containers. Samples were grouped considering the frequency of attribution of aroma descriptors to the samples. The panel on the right side highlights the differences among aged wines by applying the function of magnification of the area near origin included in the analysis software. A clear distinction emerged among samples: time 0 was different from all aged wines, with notes of mediterranean herbs, oregano and thyme that are typically reported in Negroamaro wines [115,116], as well as some floral (rose) fruity notes (cherry, blueberry, cherry jam). After six months of aging, wines aroma was enriched and samples were distinguished according to the container used with wood origin determining a differentiation as highlighted in Figure 9B. In particular, aging in amphora imparted distinct olfactive characters, with a slight evolution of the fruity and floral notes, associated to raspberry and violet, and descriptors including caramel and clove. Hence, amphorae determined a varietal aroma evolution without cover it with woody notes, in agreement with previous reports [3].This profile was quite different from that deriving from bottle aging, characterized by increased tobacco and chocolate notes. A slight differentiation in olfactory profiles could be observed among different woods. Barrels containing European and American oak imparted notes ranging from balsamic (together with mint, eucalyptus, rosemary), coconut, plum. This coulkd be probably related to higher levels of lactones [42]. On the other hand, French oak and Mix barrels kept fruity notes (as black fruits and blackcurrant) and imparted spicy characteristics.

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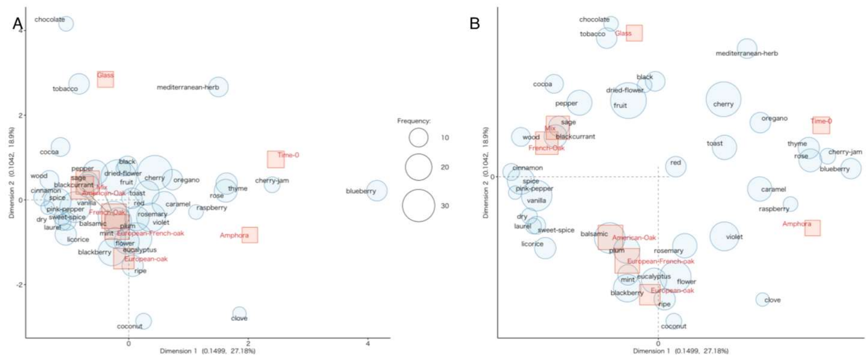

Figure 9. Correspondence analysis of the aroma attributes of the Negroamaro wine before aging (T-0) and after aging in different materials (Glass, Amphora, Mix, French, European oak, American oak, European oak, French oak). A, original scale map; B, map with the enlarge the area near origin function (3x). Red squares correspond to wines, blue bubbles correspond to taste attributes. Size of bubbles represents term frequency.

Figure 10 reports the sensory space of samples deriving from the results of the correspondence analysis carried out on the frequency of occurrence of free-choice taste descriptors of wines at time 0 and after aging in different containers. The map clearly shows the effect of aging on the Negroamaro astringency. At time 0, the wine was described as unripe, dry and rough, that is related to negative hedonic properties [117], while six-months aging determined an evolution mouthfeel as wine acquired a more complex profile, though panelists still found some aggressive traits in the wine astringency after six-months aging. Only wine aged in the Mix barrels clearly showed an interesting evolution of the astringency, with velvety, mouthcoat and evolved taste. This kind of barrel provided, therefore, the best performances in a six-month aging period on Negroamaro wine.

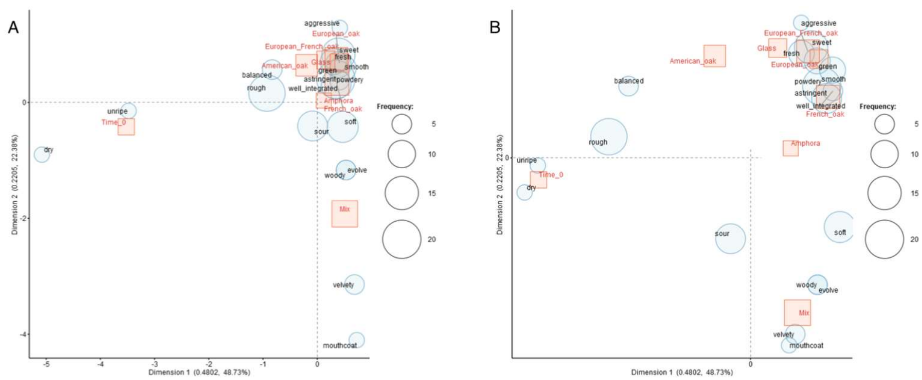

Figure 10. Correspondence analysis of the taste attributes of the Negroamaro wine before aging (T-0) and after aging in different materials (Glass, Amphora, Mix, French, European oak, American oak, European oak, French oak). A, original scale map; B, map with the enlarge the area near origin function (3 x). Red squares correspond to wines, blue bubbles correspond to odor attributes. Size of bubbles represents term frequency.

4. Conclusions

This study pointed out several differences determined by different aging materials on Negroamaro wine, obtaining wines with unique chemical and sensory characteristics. Wood barrels, particularly French and American oak, significantly increased total phenolic compounds and stabilized anthocyanin-tannin complexes, enhancing color intensity and complexity. Amphorae effectively preserved phenolic content and color stability. A clear differentiation could be observed on the evolution of the volatile profile, with regard to volatiles released as well as to the pattern of varietal aromas. As a consequence, the sensory profile of the Negroamaro wine could be led to

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different outcomes. Amphorae kept primary fruity and floral aromas with a specific aging evolution. Wooden barrels contributed distinct aromatic profiles based on wood type. The findings reveal significant differences in the influence of aging containers and highlight the promising potential of traditional Apulian amphorae as a sustainable and versatile aging container in winemaking. Future research should focus on optimizing amphora size and coatings to ensure consistency and explore their long-term impact on wine quality. The revival of these traditional vessels could contribute to sustainable winemaking practices and the valorization of regional heritage, offering a unique identity to wines from Apulia.

Supplementary Materials: The following supporting information can be downloaded at the website of this paper posted on Preprints.org.

Author Contributions: Conceptualization, V.M.P.; methodology, V.M.P. and I.P.; validation, V.M.P. and I.P.; formal analysis, I.P. and G.F.; investigation, I.P., G.F., G.C. and C.D.; resources, V.M.P.; data curation, I.P., G.C. and C.D.; writing—original draft preparation, I.P. and V.M.P.; writing—review and editing, G.F., G.C. and C.D.; visualization, I.P. and V.M.P.; supervision, V.M.P.; project administration, V.M.P.; funding acquisition, V.M.P. All authors have read and agreed to the published version of the manuscript.

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