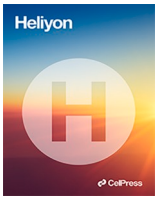
Heliyon 10 (2024) e34310

Contents lists available at ScienceDirect

Heliyon

journal homepage: www.cell.com/heliyon



Research article

Use of vine-shoots stilbene extract to the reduction of SO_2 in red and rosé Italian wine: Effect on phenolic, volatile, and sensory profiles

Mirella Noviello [a], Claudia Antonino [a], Giuseppe Gambacorta [a], Vito Michele Paradiso [b,**], Francesco Caponio [a,*]

[a] Department of Soil, Plant and Food Sciences, University of Bari Aldo Moro, Via Amendola, 165/A, 70126, Bari, Italy
[b] Department of Biological and Environmental Sciences and Technologies, University of Salento, Via Provinciale Monteroni, 73100, Lecce, Italy

ARTICLE INFO

Keywords:
Sulfur dioxide
Stilbenes
Vine-shoots
Rosé wine
Red wine

ABSTRACT

Sulfur dioxide (SO_2) is one of the most used additives in wine industry for its antioxidant and antimicrobial activity. However, due to health concerns, consumers' demand of wines with either reduced or totally replaced SO_2 has increased. This study aimed to assess the effect of partial and total replacement of SO_2 with a vine-shoots extract rich in stilbenes in rosé (cv. Sangiovese) and red (cv. Negramaro) wines respectively. Color as well as phenolic, volatile, and sensory profiles of wines were evaluated at bottling and during storage.

The results showed that the vine-shoots extract increased the levels of *trans*-resveratrol, catechin, and gallic acid in wines. Moreover, the positive correlation of procyanidin dimers in red wine suggested an increase of the polymerization reactions. The amount of added extract probably provided lower antimicrobial protection compared to SO_2, as indicated by the higher levels of ethyl phenol. The decrease of individual anthocyanins and oxidation aldehydes observed in wines with SO_2 replacement and the higher levels of caftaric acid in the rosé wine with the extract suggested a shift of the oxidative protection, with a lower protection towards anthocyanin degradation and higher protection towards carbonyl formation and oxidation of readily oxidizable phenolic acids.

1. Introduction

Sulfur dioxide (SO_2) is the wine industry's most widely used antioxidant and antimicrobial-acting preservative [1]. SO_2 exhibits enforcement action against both enzymatic and chemical oxidations [2]. The main anti-oxidative activity of sulfur dioxide in wine is due to the reduction of dissolved oxygen with a slow process which protect wines from chemical oxidation of some polyphenols and odorous substances [1–3].

Moreover, SO_2 limits acetic and lactic acid bacteria and other undesirable microorganisms growth, favoring the activity of *Saccharomyces cerevisiae* [4]. This additive is added in grape or must (before the start of alcoholic fermentation) and in wine (during filtration, decanting, and aging or before bottling). Insufficient amounts could cause oxidation or microbial spoilage, compromising

* Corresponding author.
** Corresponding author.
E-mail addresses: vito.paradiso@unisalento.it (V.M. Paradiso), francesco.caponio@uniba.it (F. Caponio).

https://doi.org/10.1016/j.heliyon.2024.e34310
Received 10 May 2024; Received in revised form 3 July 2024; Accepted 8 July 2024
Available online 9 July 2024

wine quality, while excessive amounts entail certain drawbacks in wine and in health of sensitive consumers [5]. According to the legislation of European Union (Reg. (EC) No 606/2009, Annex I B) "the total sulfur dioxide content of wines, other than sparkling wines and liqueur wines, on their release to the market for direct human consumption, may not exceed 150 mg per litre for red wines and 200 mg per litre for white and rosé wines". The World Health Organization defined an acceptable daily intake of 0.7 mg SO_2 per kg body weight.

In the recent years, the consumers' demand for wines without additives or with low additive content is increased [6]. For these reasons, wine industry is intent to develop new practices for SO_2 replacement or reduction, such as: chemical methods (i.e., dimethyl dicarbonate, lysozyme, sorbic acid, chitosan or bacteriocins and antibiotics) [2]; antioxidant activity substitutes (i.e., glutathione or oenological tannins) [7]; the emerging non-thermal technologies, such as pulsed electric fields, high pressure processing, power ultrasound or ultraviolet irradiation [8]; bioprotection, which consists in using *non-Saccharomyces* strains to prevent microbial deviation [9]. Moreover, a wide range of phenolic substances, such as stilbenes, have been studied in attempts to replace the antioxidant activity of SO_2 [10]. Stilbenes are a small yet important class of non-flavonoid polyphenols, with a chemical structure both in monomeric and oligomeric states and constituted by a diphenylethylene group oriented in *cis* or *trans* [11]. They are naturally synthesized by many families of plants, including *Vitaceae*, as phytoalexins in response to biotic and abiotic stress [11]. In particular, wastes and by-products from the wine supply chain, such as pruning residues and organic and inorganic winemaking residues (grape pomace, grape seeds, grape stalks, and wine lees, as well as wastewater), that correspond to approximately 30 % (w/w) of the starting grapes [12], are very rich in phenolic compounds [13]. Among these, vine-shoots are a promising source of antioxidant and antimicrobial compounds, especially stilbenes, among which *trans*-resveratrol (Rsv) and *ε*-viniferin (Vf) are the most abundant [14,15].

Several studies assessed stilbene extracts from vine-shoots as a preservative in wine to reduce the use of SO_2 in winemaking. Raposo et al. [16] and Cruz et al. [17] used a commercial extract obtained from vine-shoots (named Vineatrol®) in red and white winemaking processes. The results showed good storage ability and an improvement in color and sensory features. Similarly, Gutiérrez-Escobar et al. [18], adding pure stilbene extract in Syrah wine, improved the preservation of rosé wine quality and limited changes in wine parameters. However, a critical issue of the use of pure/enriched stilbene extracts regards the yields and purification cost from raw extracts. To fill this gap, in our work a hydroalcoholic raw extract from vine-shoots were obtained without further costs of purification processes, with a view to sustainability and circular economy. In this context, the aims of this study were to evaluate the preservative action of a raw vine-shoot extract as partial or total replacement of SO_2 in winemaking and the effects on the chromatic characteristics and on the phenolic, volatile, and sensory profile of the experimental wines analyzed at bottling and after 3, 6, and 12 months, compared with control wines containing sulfur dioxide.

2. Materials and methods

2.1. Vine-shoot extraction

Vine-shoots of five varieties of *Vitis vinifera* L. (Italia, Montepulciano, Negroamaro, Nero di Troia, Paglieri), selected according to the results obtained in a previous study in terms of stilbenes concentration [15], were considered for this study. All vine-shoots were sampled during winter (February 2021) from a varietal collection located in Locorotondo (Puglia, Italy; coordinates: longitude 17°13′3.741″ E, latitude 40°45′42.763″ N), grown under the same conditions. Then, about 10 kg of vine-shoots were stored intact under controlled conditions (darkness, at 15 ± 3 °C) for 6 weeks [19]. The extraction of stilbenes from a mix of vine-shoots was carried out as reported in a previous study [15].

2.2. Vine-shoot extract characterization

Glucose and fructose were determined using an HPLC 1260 Infinity Series chromatograph (Agilent Technologies, Santa Clara, CA, USA) equipped with a refractive index detector (RID) and a cationic exchange column 300 × 7.8 mm (Rezex™ RCM-Monosaccharide Ca^{2+}, 8 μm, Phenomenex, Torrance, CA, USA). The analysis was performed in isocratic conditions using Milli-Q water as a mobile phase with a flow of 0.6 mL min^{-1}, column temperature of 80 °C, and RID of 35 °C. Standard solutions of glucose and fructose were filtered through a 0.45 μm nylon filter and injected at different concentration to obtain calibration curves [20]. Ash contents were determined according to the AOAC method 923.03. An UHPLC Ultimate 3000RS Dionex interfaced by H-ESI II probe with a LTQ Velos pro linear ion trap mass spectrometer (Thermo Fisher Scientific, Waltham, MA, USA) was used for the qualitative analysis of phenolic compounds of the extract as reported by Pasqualone et al. [21] with some modification. Specifically, a Hypersil Q C18 (Thermo Fisher Scientific, Waltham, MA, USA) column 100 mm of length, 2.1 mm internal diameter and 1.9 μm of particle size held at 30 °C and at a constant flow of 0.55 mL min^{-1} was used and a binary gradient constituted by 0.1 % of formic acid in water (solvent A) and acetonitrile (solvent B), from 6 % to 70 % of B linear gradient in 33 min. The samples were filtered using syringe filters (LLG Labware, Meckenheim, Germany) in RC by 0.22 μm before injection into the equipment. The injection volume was 5 μL. All data were acquired and processed using Xcalibur v.2 (Thermo Fisher Scientific, Waltham, MA, USA). The mass spectrometer parameters were taken by Makhlouf et al. [22]. Tentative identification of compounds utilized mass spectra (MS^2) and λ_{max} accordingly to Goufo et al. [23], and Supplementary Table S1 reports the identified phenolic profile of vine-shoot extract. The quantification of *trans*-resveratrol (Rsv) and ε-viniferin (Vf) was carried as reported in Noviello et al. [15] using an UltiMate 3000 HPLC (Thermo Fisher Scientific, Waltham, MA, USA), with a Acclaim™ 120C18 columns (120 Å 3 × 150 mm, 3 μm) maintained at 25 °C using a mobile phase consisting of 1 % aqueous acetic acid (v/v) (solvent A) and methanol (solvent B). The separation was conducted with a flow rate of 0.6 mL min^{-1} as reported below: 0 min (20 % B), 10 min (20 % B) 6.5 min (37 % B), 12.6 min (50 % B), and 21.0 min (100 % B). The lyophilized vine-shoot extract (EX)

contained: 27.47 mg g^{-1} of stilbenes (23.2 mg g^{-1} of Rsv and 4.27 mg g^{-1} of Vf), 16.8 g 100 g^{-1} of glucose, 22.9 g 100 g^{-1} of fructose, and 1.1 g 100 g^{-1} of ashes.

2.3. Wine sampling

Negroamaro red wine and Sangiovese rosé wine of the vintage 2021 were sampled from the wineries Azienda Agricola Conti Zecca (Leverano, Puglia, Italy) and Divella s.r.l. (Santeramo in Colle, Puglia, Italy), respectively. The experimental plan was summarized in Fig. 1 and detailed below. In red and rosé control wines 9 g 100 L^{-1} and 21 g 100 L^{-1} of $K_2S_2O_5$ were used, respectively. Moreover, in the case of rosé wine a sample with a lower concentration dose of $K_2S_2O_5$ (14 g 100 L^{-1}) was produced to obtain the wine with partial replacement of SO_2 with EX. In fact, the collected red and rosé wines were divided each in two aliquots. In particular: i) Negroamaro red wine with 50 mg L^{-1} of total SO_2 (red control wine, R–C); ii) Negroamaro red wine with 50 mg L^{-1} of vine-shoot stilbenes (red wine with extract, R-EX); iii) Sangiovese rosé wine with 120 mg L^{-1} of total SO_2 (rosé control wine, r-C); iv) Sangiovese rosé wine with 80 mg L^{-1} of total SO_2 plus 40 mg L^{-1} of vine-shoot stilbenes (rosé wine with extract, r-EX). Three different batches for each experimental wine were considered. All samples (R–C, R-EX, r-C, r-EX) were stored at room temperature (20 ± 5 °C) in dark condition. After each considered storage time (at bottling and after 3, 6, and 12 months) all wines were analyzed in triplicate. Negroamaro red wine: alcoholic degree 12.8 % (v/v), total acidity (g L^{-1} tartaric acid) 5.00 g L^{-1}, pH 3.64, volatile acidity (g L^{-1} acetic acid) 0.50 g L^{-1}, lactic acid 0.76 g L^{-1}. Rosé wine: alcoholic degree 10.5 % (v/v), total acidity (g L^{-1} tartaric acid) 5.00 g L^{-1}, pH 3.68, volatile acidity (g L^{-1} of acetic acid) 0.54 g L^{-1}, lactic acid 1.67 g L^{-1}. The chemical parameters of Negroamaro red wine and rosé wine have remained unchanged over experimental time as shown in Supplementary Table S2.

2.4. Wines characterization

2.4.1. Phenolic compounds, flavonoids, anthocyanins, antioxidant activity, color indices, and sulfur dioxide determinations

The Total Phenol Content (TPC) was determined according to the Folin-Ciocalteu method as described in Difonzo et al. [24]. More in detail, to 980 μL of H_2O Milli-Q, 20 μL of appropriately diluted extract and 100 μL of Folin–Ciocalteu reagent were added. After 3 min, 800 μL of 7.5 % Na_2CO_3 were added and then the sample was stored in the dark for 60 min. The absorbance was read at 720 nm (Cary60 UV–Vis, Agilent Technologies, Mulgrave, Australia). The results were expressed as mg of gallic acid equivalents (GAE) per mL of wine (mg GAE L^{-1}). Flavonoids (F, as mg L^{-1} of (+)-catechin) and anthocyanins (A, as mg L^{-1} of malvidin-3-glucoside) were determined according to Gambacorta et al. [25] using an Cary60 UV–Vis spectrophotometer (Agilent Technologies, Mulgrave, Australia).

The DPPH and ABTS-TEAC assays were performed according to the procedure of Tarantino et al. [26]. Color indices, that were color intensity (CI: $Abs_{420\ nm}$+ $Abs_{520\ nm}$+ $Abs_{620\ nm}$) and hue (H: $Abs_{420\ nm}/Abs_{520\ nm}$) and the total SO_2 (mg L^{-1}) was assessed according to the Glories procedure [27], using cuvettes of 1 mm for red and of 10 mm for rosé wine, and Ripper-Schmitt official methods, respectively.

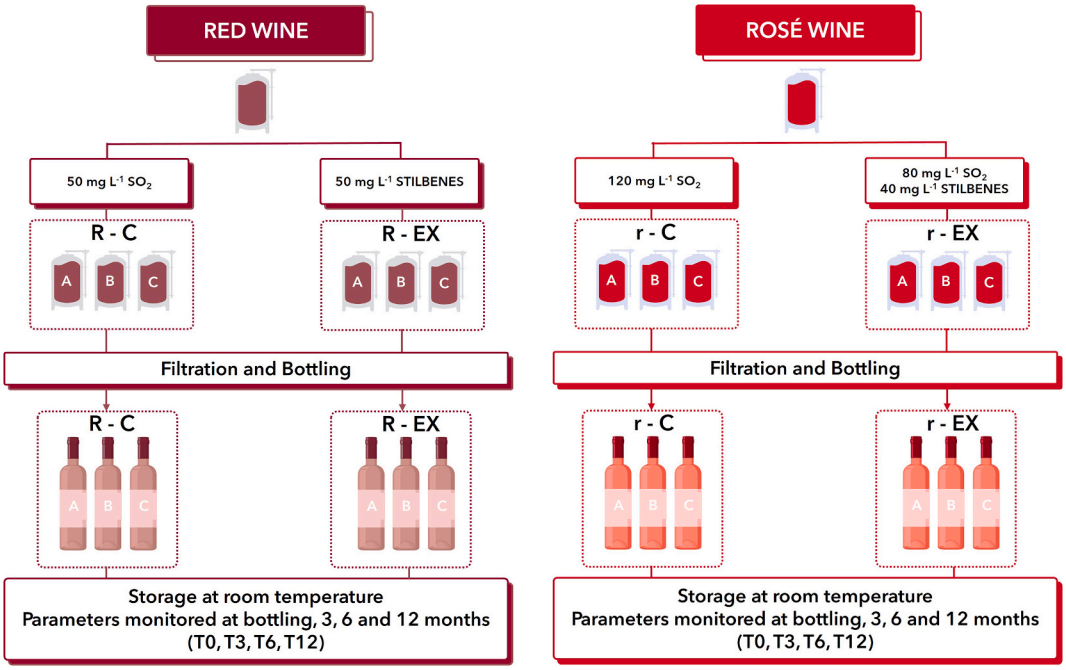


Fig. 1. The experimental plan.

2.4.2. Phenolic profile

The identification and quantification of phenolic compounds was performed as reported in Noviello et al. [28], using an UHPLC Ultimate 3000RS Dionex composed by quaternary pump, autosampler, column compartment, and detector and interfaced by H-ESI II probe with a LTQ Velospro linear ion trap mass spectrometer (Thermo Fisher Scientific, Waltham, MA, USA). Specifically, a Hypersil GOLD aQ C18 column was used (100 mm of length, 2.1 mm internal diameter and 1.9 μm of particle size). The column was held at 30 °C and at a constant flow of 0.3 mL min^{-1}. The solvent A was composed of water and formic acid (90:10 v/v), while the solvent B of acetonitrile and formic acid (99.9:0.1 v/v). The gradient program of solvent A was as follows: 0–20 min from 98 % to 30 %; 20–24 min isocratic at 30 % with the equilibration at the initial conditions for 9 min. The PDA detector was set to scan from 220 to 600 nm of wavelength managed by a 3D field. The MS parameter conditions were as follows: capillary temperature 320 °C; source heater temperature 280 °C; nebulizer gas N_2; sheath gas flow 33 psi; auxiliary gas flow 5 arbitrary units; S-Lens RF Level 60 %. The samples were analyzed with two methods: a full scan method from 100 to 1000 *m/z* and a data-dependent experiment to collect MS^2 data. In this case the data-dependent settings were full scan from 140 to 800 *m/z* for negative ionization mode and from 200 to 1000 for positive ionization, in both cases with activation level 500 counts, isolation width 2 Da, default charge state 2, and CID energy 35. Retention time, mass spectra (MS^2) and λ_{max}, were used for tentative identification of compounds, as reported to literature [29–35]. Quantitative analysis was performed according to the external standard method based on calibration curves obtained by injecting different concentrations of standard solutions (R^2= 0.9960–0.9996). Specifically, the standard used were: (+)-catechin, (−)-epicatechin, malvidin-3-*O*-glucoside, quercetin were phyproof® reference substances (PhytoLab, Dutendorfer, Germany); gallic acid, caftaric acid were purchased from Sigma-Aldrich (St. Louis, MO, USA); *trans*-resveratrol was purchased from United States Pharmacopeia (USP, Maryland, United States). Results were expressed in mg L^{-1}.

2.4.3. Volatile organic compounds (VOCs) determination

VOCs were determined by Solid Phase Micro-Extraction (SPME) coupled by Gas Chromatography-Mass Spectroscopy (GC-MS), as reported by Prezioso et al. [36]. Into 20 mL vials, 1 ± 0.05 mL of the samples, 0.2 g mL^{-1} of NaCl (to increase the ionic strength) were weighed, the vials were closed with a silicone/PTFE septum and an aluminum cap. A mother solution of 2-octanol (Sigma Aldrich, Milan, Italy) used as an internal standard, 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. In fact, a semi-quantitation of the compounds was done, and the amounts were expressed as μg of 2-octanol equivalents $Litre^{-1}$. Samples were loaded into an autosampler Triplus RSH (ThermoFisher Scientific, Rodano, Italy). The stabilization of the headspace in the vial was obtained by equilibration for 10 min at 50 °C. A divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) 50/30 mm SPME fiber assembly (Supelco, Bellefonte, PA, USA) at 50 °C for 30 min was used for the extraction process. The fiber was desorbed at 200 °C for 2 min in the injection port of the gas chromatograph, operating in split-less mode. Trace1300 gas chromatograph equipped with a mass spectrometer ISQ Series 3.2 SP1 were used to the GC-MS analyses. The compounds were separated in a Thermo capillary column VF-WAX MS (60 m, 0.25 mm, 0.25 μm) with an injection port temperature of 200 °C, an oven temperatures of 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 as described below: detector voltage, 1700 V; source temperature, 250 °C; ionization energy, 70 eV; scan range, 33–150 amu. Tentative identification of the peaks was realized using Xcalibur V2.0 Qual Browse software (Thermo Fisher Scientific, Waltham, MA, USA) by matching with the reference mass spectra of the NIST library.

2.4.4. Sensory evaluation

Eight expert tasters (4 women and 4 men) performed sensory analysis. The tasters expressed written consent according to the ethical guidelines of the Food Science and Technology Laboratory of the Department of Soil, Plant and Food Science, University of Bari (Italy). The judges were winemakers and professionals, participated at many wine evaluation sessions and experienced in wine tasting.

In each session, wine samples were presented in a completely randomized order to each taster. The judges were not given any information about the origin of the samples. Then, each wine was evaluated by the judges using an evaluation form that included descriptors as described in Trani et al. [37] with some modification. Specifically, the descriptors were grouped by visual phase (clarity, color intensity and viscosity), olfactory phase (intensity, persistence, balance), and taste phase (flavor intensity, persistence, balance, tannic, sweetness, acidity, and body) and overall judgment. The tasters rated each attribute on a scale from 0 (absence) to 10 (highest perception). The average attribute scores were submitted to quantitative descriptive analysis (QDA) to generate the sensory profile of the wines. The olfactory profile of the wines was also evaluated using the check-all-that-apply (CATA) approach [38]. Judges were asked to report the perception of odor attributes (such as fruity, floral, chocolate, toasted, vanilla, oak) in the sample.

2.5. Statistical analysis

Two-way analysis of variance (two-way ANOVA), followed by Tukey's test ($p \leq 0.05$), was applied to determine significant differences between the samples using OriginPro software. A clustering analysis with the construction of a polar heatmap was used to evaluate the VOCs of wines. Partial correlation analysis (metadata: treatment; covariate: time; correlation measure: point biserial correlation) were performed using Metaboanalyst 6.0. Correspondence analysis of the highest frequency descriptors was carried out on the results of CATA sensory analysis using the KH coder software (https://khcoder.net/en/).

3. Results and discussion

3.1. SO_2 content, total polyphenols content, antioxidant activity and color indices

Table 1 reports the SO_2 content, total phenolic content (TPC), total anthocyanins (A), total flavonoids (F), antioxidant activity (AA) and color indices value of red wine (cv. Negroamaro) and rosé wine (cv. Sangiovese) at different times of storage and the results of the two-way ANOVA applied to the data obtained, respectively.

Regarding the influence of the considered variables, the treatment variable (*Tr*) showed that the addition of vine-shoots extract (EX) had a significant impact on all tested parameters, with the exception for the color intensity (CI) of red wine, while the storage time variable (*T*) significantly influenced all the parameters. Moreover, the first order interaction (*Tr***T*) was significant for all the parameters, with the exception of flavonoids and hue (H), this latter only for red wine.

Considering the single obtained values, total SO_2 significantly decreased during storage time, as expected, in all the wines, with a reduction of about 30 % for rosé wines and about 50 % for red wine. The values obtained for TPC, A, and F for control red and rosé

Table 1
SO_2 content, total phenolic content, antioxidant activity, color indices, of red and rosé wines.

Parameters	Samples	Storage time (T, months)				*Tr*	*T*	*Tr***T*
		Bottling (T0)	T3	T6	T12			
Total SO_2 (mg L^{-1})	R–C	*50 ± 0.1a	26 ± 1c	38 ± 2b	22 ± 1d	***	***	***
	R-EX	–	–	–	–			
	r-C	120 ± 0.1a	93 ± 2c	98 ± 1b	81 ± 2e	***	***	***
	r-EX	80 ± 0.1d	70 ± 1g	80 ± 2f	57 ± 1h			
TPC (mg L^{-1} of gallic acid equivalents)	R–C	1691 ± 48c	1650 ± 18c	1957 ± 19b	1433 ± 10d	***	***	***
	R-EX	2297 ± 139a	1896 ± 26b	2151 ± 46a	1687 ± 78c			
	r-C	680 ± 1d	508 ± 10e	937 ± 37b	377 ± 5f	***	***	***
	r-EX	870 ± 51bc	822 ± 33c	1523 ± 15a	650 ± 3d			
A (mg L^{-1} of malvidin-3-glucoside)	R–C	429 ± 6ab	297 ± 52cd	489 ± 9a	158 ± 2e	***	***	*
	R-EX	383 ± 13b	250 ± 36d	357 ± 42bc	110±6e			
	r-C	20 ± 2a	18 ± 2 ab	18 ± 1ab	8 ± 1d	***	***	*
	r-EX	17 ± 1 ab	14 ± 2bc	12 ± 2c	7 ± 1d			
F (mg L^{-1} of (+)-catechin)	R–C	1599 ± 31d	1456 ± 116d	2542 ± 195b	1089 ± 117e	***	***	ns
	R-EX	2427 ± 71bc	2135 ± 123c	3380 ± 192a	1606 ± 23d			
	r-C	250 ± 3cd	247 ± 11cd	354 ± 42c	154 ± 6d	***	***	ns
	r-EX	813 ± 9a	809 ± 14a	849 ± 103a	647 ± 39b			
ABTS (μmol Trolox Equivalent L^{-1})	R–C	6221 ± 44e	7067 ± 171d	8924 ± 60b	2842 ± 65g	***	***	*
	R-EX	6721 ± 173d	8080 ± 209c	9678 ± 123a	3487 ± 94f			
	r-C	2640 ± 55c	2022 ± 44e	2244 ± 38d	841 ± 34g	***	***	***
	r-EX	2493 ± 123c	2864 ± 31b	3185 ± 17a	1178 ± 20f			
DPPH (μmol Trolox Equivalent L^{-1})	R–C	4637 ± 89d	4531 ± 327d	6889 ± 211c	8231 ± 28b	***	***	***
	R-EX	6291 ± 182c	4739 ± 399d	7643 ± 135b	9084 ± 12a			
	r-C	1030 ± 88e	1114 ± 89e	1343 ± 45d	1863 ± 60b	***	***	***
	r-EX	2499 ± 159a	1613 ± 11c	2081 ± 21b	2662 ± 57a			
CI	R–C	0.94 ± 0.01bc	0.95 ± 0.04bc	0.98 ± 0.01 ab	0.98 ± 0.06ab	ns	**	***
	R-EX	1.05 ± 0.02a	0.92 ± 0.03bc	0.94 ± 0.03bc	0.86 ± 0.03c			
	r-C	0.86 ± 0.01de	0.75 ± 0.04f	0.82 ± 0.02ef	0.93 ± 0.06d	***	***	***
	r-EX	1.43 ± 0.01a	1.06 ± 0.02c	0.93 ± 0.01d	1.19 ± 0.02b			
H	R–C	0.72 ± 0.01de	0.70 ± 0.01e	0.73 ± 0.01cde	0.78 ± 0.03ab	***	***	ns
	R-EX	0.77 ± 0.01bc	0.75 ± 0.01bcd	0.76 ± 0.01bcd	0.81 ± 0.01a			
	r-C	1.68 ± 0.01d	1.71 ± 0.09cd	1.60 ± 0.01e	1.76 ± 0.05bc	***	***	*
	r-EX	1.60 ± 0.01e	1.84 ± 0.15 ab	1.89 ± 0.01a	1.85 ± 0.02a			
A_{420nm}	R–C	0.35 ± 0.01bc	0.34 ± 0.01bc	0.36 ± 0.01bc	0.37 ± 0.02bc	ns	**	***
	R-EX	0.40 ± 0.01a	0.34 ± 0.01bc	0.35 ± 0.01bc	0.33 ± 0.01c			
	r-C	0.50 ± 0.01d	0.44 ± 0.02f	0.47 ± 0.01e	0.55 ± 0.02d	***	***	***
	r-EX	0.78 ± 0.01a	0.63 ± 0.01c	0.58 ± 0.01d	0.69 ± 0.01b			
A_{520nm}	R–C	0.48 ± 0.01bc	0.49 ± 0.02ab	0.49 ± 0.01ab	0.47 ± 0.01bc	***	***	***
	R-EX	0.52 ± 0.01a	0.46 ± 0.01c	0.47 ± 0.02bc	0.41 ± 0.01d			
	r-C	0.29 ± 0.01d	0.26 ± 0.02e	0.30 ± 0.01d	0.31 ± 0.02cd	***	***	***
	r-EX	0.49 ± 0.01a	0.34 ± 0.01c	0.31 ± 0.01cd	0.38 ± 0.01b			
A_{620nm}	R–C	0.11 ± 0.01a	0.12 ± 0.01a	0.13 ± 0.01a	0.14 ± 0.04a	ns	ns	ns
	R-EX	0.13 ± 0.01a	0.12 ± 0.01a	0.13 ± 0.01a	0.12 ± 0.01a			
	r-C	0.07 ± 0.01ab	0.06 ± 0.01ab	0.05 ± 0.01b	0.07 ± 0.02ab	**	***	ns
	r-EX	0.16 ± 0.01ab	0.10 ± 0.01ab	0.05 ± 0.01a	0.12 ± 0.01ab			

*Data followed by different letters indicate statistically significant differences at $p \leq 0.05$ according to two-way ANOVA with interaction followed by Tukey's test. Average value ± standard deviation (n = 3). Abbreviations: R–C, red control wine; R-EX, red wine with extract; r-C, rosé control wine; r-EX, rosé wine with extract; TPC, Total phenolic content; A, anthocyanins; F, flavonoids; ABTS, 2,20-azinobis-(3-ethylbenzothiazoline-6-sulfonic acid); DPPH, 2,2-diphenyl-1-picrylhydrazyl; CI, color intensity; H, hue; Tr, treatment variable; T, storage time variable; ns, not significant; *, **, ***, significant at $p \leq 0.05$, $p \leq 0.01$, $p \leq 0.001$, respectively.

Table 2
Phenolic compounds (mg L^{-1}) of red wine.

Compounds				Storage time (T, months)										
				Bottling (T0)		T3		T6		T12				
				R–C	R-EX	R–C	R-EX	R–C	R-EX	R–C	R-EX	*Tr*	*T*	*Tr*T*
Anthocyanins	**RT**	**MH^+(*m/z*)**	**Fragments (*m/z*)**											
Delphinidin 3-glucoside[b]	6.00	465	303	*41.1 ± 3.7a	33.6 ± 1.3b	21.0 ± 3.8c	13.3 ± 0.9d	13.9 ± 2.3d	5.8 ± 0.5e	3.8 ± 0.2e	1.9 ± 0.2e	***	***	ns
Cyanidin 3-glucoside[b]	6.47	449	287	10.8 ± 0.9a	9.7 ± 0.9a	5.8 ± 0.7b	3.5 ± 0.9cd	4.2 ± 0.5bc	2.3 ± 0.3de	0.9 ± 0.03e	0.7 ± 0.2e	***	***	*
Petunidin 3-glucoside[b]	6.87	479	317	82.7 ± 7.4a	69.4 ± 1.6b	35.6 ± 3.4c	21.5 ± 1.3de	28.6 ± 3.7cd	13.8 ± 2.1ef	5.0 ± 0.2fg	3.1 ± 0.3g	***	***	*
Malvidin 3,5-glucoside[b]	7.23	655	331	9.6 ± 1.0 ab	11.0 ± 1.1a	7.5 ± 1.1bc	5.9 ± 1.1cd	6.0 ± 0.9cd	4.6 ± 0.5de	1.7 ± 0.3f	2.2 ± 0.5ef	ns	***	*
Peonidin 3-glucoside[b]	7.35	463	301	85.1 ± 5.7a	72.2 ± 1.0b	40.3 ± 7.3c	29.0 ± 1.4d	29.0 ± 3.8d	14.1 ± 1.5e	6.1 ± 0.2e	4.5 ± 1.0e	***	***	*
Malvidin 3-glucoside[a]	7.64	493	331	722.9 ± 40.8a	623.9 ± 5.7b	345.5 ± 60.9c	230.7 ± 13.8d	232.4 ± 30.8d	131.7 ± 11.9e	44.0 ± 0.8f	31.7 ± 4.3f	***	***	*
Delphinidin 3-(6″-acetyl) glucoside[b]	7.91	507	303	12.4 ± 0.5a	11.2 ± 2.0a	6.2 ± 1.1b	5.4 ± 0.7bc	4.1 ± 0.5bc	2.9 ± 0.5cd	1.5 ± 0.01d	1.2 ± 0.1d	*	***	ns
Vitisin A[b]	8.1	561	399	9.7 ± 1.5a	9.5 ± 1.3a	9.2 ± 0.9 ab	8.1 ± 1.6 ab	7.1 ± 0.9abc	6.3 ± 0.8bc	4.2 ± 0.2c	4.5 ± 0.4c	ns	***	ns
Vitisin B[b]	8.3	517	355	4.4 ± 0.01b	3.3 ± 0.2c	4.5 ± 0.2b	4.5 ± 0.3b	4.7 ± 0.3a	5.1 ± 0.2 ab	1.2 ± 0.1e	2.4 ± 0.3d	ns	***	***
Petunidin-3-(6″-acetyl)-glucoside[b]	8.71	521	317	19.0 ± 1.1a	16.5 ± 2.8a	11.1 ± 0.5b	6.5 ± 0.5c	8.0 ± 0.5bc	5.5 ± 0.8c	1.5 ± 0.1d	1.8 ± 0.3d	***	***	*
Peonidin-3-(6″-acetyl)-glucoside[b]	9.27	505	301	21.1 ± 1.2a	16.4 ± 1.8b	9.2 ± 1.7c	7.4 ± 1.3c	6.3 ± 0.8c	3.1 ± 0.2d	1.1 ± 0.2d	0.7 ± 0.2d	***	***	*
Malvidin 3-(6″-acetyl)-glucoside[b]	9.49	535	331	193.2 ± 10.7a	161.7 ± 0.7b	89.6 ± 15.1c	61.1 ± 3.2d	59.6 ± 8.5d	32.8 ± 3.0e	9.9 ± 0.2f	7.3 ± 1.0f	***	***	*
Petunidin 3-(6″-*t*-coumaroyl)-glucoside[b]	10.13	625	317	7.2 ± 0.6a	4.6 ± 0.3b	4.2 ± 0.01bc	4.4 ± 0.4b	3.4 ± 0.2c	2.2 ± 0.3d	1.0 ± 0.1e	1.3 ± 0.2e	***	***	***
Malvidin 3-(6″-*c*-coumaroyl)-glucoside[b]	10.27	639	331	5.9 ± 0.3a	4.9 ± 0.5a	2.7 ± 0.4b	2.1 ± 0.7bc	2.1 ± 0.4bc	2.8 ± 0.4b	0.9 ± 0.4d	1.1 ± 0.1cd	ns	***	**
Peonidin 3-(6″-*t*-coumaroyl)-glucoside[b]	10.67	609	301	22.2 ± 1.0a	18.9 ± 2.1b	9.4 ± 1.2c	5.2 ± 0.7d	5.1 ± 1.0d	2.5 ± 0.6de	0.8 ± 0.01e	0.6 ± 0.1e	***	***	*
Malvidin 3-(6″-*t*-coumaroyl)-glucoside[b]	10.83	639	331	85.1 ± 4.1a	71.3 ± 2.5b	35.5 ± 5.5c	16.2 ± 6.0de	20.6 ± 3.1d	10.6 ± 1.0ef	3.6 ± 0.1f	3.0 ± 0.5f	***	***	**
Σ Anthocyanins				*1332.3 ± 73.0*a	*1138.2 ± 7.9*b	*637.3 ± 96.7*c	*427.6 ± 29.9*d	*435.0 ± 55.9*d	*246.2 ± 23.9*e	*87.1 ± 1.6*f	*67.8 ± 9.3*f	***	***	*
Phenolic acids	**RT**	**MH^-(*m/z*)**	**Fragments (*m/z*)**											
Gallic acid[a]	1.13	169	125	102.5 ± 5.5cd	114.1 ± 4.4abc	94.6 ± 7.3cd	139.8 ± 0.8a	105.7 ± 11.6bc	116.0 ± 11.3abc	77.8 ± 1.5d	129.6 ± 17.7ab	***	ns	*
Caftaric acid[a]	2.23	311	179,149,135	46.7 ± 2.6a	44.1 ± 0.9ab	39.9 ± 4.2abc	37.9 ± 6.5abc	37.7 ± 4.7abc	35.4 ± 2.6bcd	26.8 ± 0.3cd	31.8 ± 3.6d	ns	***	ns
Coutaric acid[c]	3.49	295	163,149	20.8 ± 0.8a	19.3 ± 0.4ab	17.8 ± 1.6abc	16.8 ± 2.2abc	17.1 ± 1.8abc	15.3 ± 1.0bc	9.8 ± 3.1d	13.3 ± 1.3cd	ns	***	ns

(*continued on next page*)

Table 2 (*continued*)

Compounds				Storage time (T, months)										
				Bottling (T0)		T3		T6		T12				
				R–C	R-EX	R–C	R-EX	R–C	R-EX	R–C	R-EX	*Tr*	*T*	*Tr*T*
Fertaric acid[c]	4.71	325	193,149	5.9 ± 0.1a	5.6 ± 0.2 ab	5.2 ± 0.5 ab	5.2 ± 0.6 ab	5.3 ± 0.8 ab	4.6 ± 0.5bc	3.0 ± 0.3cd	3.6 ± 0.4d	ns	***	ns
Σ Phenolic acid				*175.9 ± 8.8 ab*	*183.0 ± 5.8ab*	*157.5 ± 13.6b*	*199.6 ± 9.3a*	*165.8 ± 16.8 ab*	*171.3 ± 15.3 ab*	*117.4 ± 1.9c*	*178.3 ± 23.0 ab*	***	**	**
Flavonols	**RT**	**MH⁻(*m/z*)**	**Fragments (*m/z*)**											
Procyanidin dimers[d]	2.58	577	451,425,407,289	51.2 ± 3.6ab	52.6 ± 1.2a	43.7 ± 4.7b	34.8 ± 1.1c	29.7 ± 3.2cd	25.6 ± 2.7d	17.5 ± 0.4e	22.3 ± 2.9de	ns	***	**
(+)-Catechin[a]	3.23	289	245,179,205	38.8 ± 2.2bc	51.0 ± 1.8a	26.6 ± 3.8d	41.3 ± 2.7b	26.2 ± 3.9d	35.1 ± 3.4bcd	13.6 ± 0.4e	30.3 ± 6.2cd	***	***	ns
Procyanidin dimers[d]	4.45	577	451,425,407,289	59.7 ± 4.5a	62.7 ± 2.1a	50.9 ± 4.9ab	44.5 ± 8.0bc	55.1 ± 8.1 ab	35.6 ± 3.6c	42.0 ± 1.8bc	50.6 ± 1.2 ab	ns	***	***
Procyanidin dimers[d]	4.54	577	451,425,407,289	55.1 ± 1.8a	55.8 ± 1.6a	35.8 ± 1.0bc	43.0 ± 6.0b	36.5 ± 4.9bc	38.5 ± 3.5bc	22.8 ± 0.6d	32.9 ± 1.5c	**	***	ns
(−)-Epicatechin[a]	5.44	289	245,205	23.0 ± 2.5bc	29.2 ± 0.9ab	29.7 ± 2.9ab	31.8 ± 3.3a	29.5 ± 3.4 ab	33.1 ± 2.3a	20.1 ± 1.1c	24.8 ± 1.1bc	***	***	ns
Σ Flavanols				*194.8 ± 38.7*a	*216.7 ± 34.7*a	*158.1 ± 33.7*ab	*172.6 ± 4.0*a	*157.7 ± 28.9*ab	*149.9 ± 3.6*ab	*104.2 ± 8.0*c	*145.0 ± 6.4*bc	ns	***	ns
Flavonols	**RT**	**MH⁻(*m/z*)**	**Fragments (*m/z*)**											
Quercetin[a]	10.44	301	273,257,179,151	3.3 ± 0.2a	3.4 ± 0.1a	0.9 ± 0.2cd	1.5 ± 0.5bc	1.2 ± 0.2bc	1.2 ± 0.7bc	1.4 ± 0.1bc	2.1 ± 0.5b	***	***	***
Stilbenes	**RT**	**MH⁺(*m/z*)**	**Fragments (*m/z*)**											
trans-Resveratrol[a]	9.08	229	135,211,119,107, 183,193	0.6 ± 0.01e	59.9 ± 1.2a	0.4 ± 0.1e	34.9 ± 0.5b	0.4 ± 0.1e	16.0 ± 0.7d	0.3 ± 0.1e	38.6 ± 1.7e	***	***	***
Total				*1707.0 ± 111.6a*	*1601.2 ± 36.5a*	*954.2 ± 52.7b*	*840.1 ± 35.9bc*	*760.1 ± 95.4c*	*584.6 ± 37.9d*	*309.0 ± 7.2e*	*431.7.9 ± 26.7de*	*	***	**

*Data followed by different letters in the same line indicate statistically significant differences at $p \leq 0.05$ according to two-way ANOVA with interaction followed by Tukey's test. Average value ± standard deviation (n = 3). Abbreviations: R–C, red control wine; R-EX, red wine with extract; Tr, treatment variable; T, storage time variable; ns, not significant; *, **, ***, significant at $p \leq 0.05$, $p \leq 0.01$, $p \leq 0.001$, respectively.

[a] Quantified using corresponding standards.

[b] expressed as Malvidin 3-glucoside equivalents.

[c] expressed as caftaric acid equivalents.

[d] expressed as (+)-Catechin equivalents.

wines were in agreement with those observed by other authors [39]. The contribution of the extract to the phenolic content of wines remained throughout the experiment, as indicated by the significant increase of TPC and F in each time of sampling, while a concomitant lower amount of A, significant only after 6 months, was observed, in agreement with previous studies [16,40]. The trend observed for anthocyanins during storage was similar to data reported in similar studies [18,40] due to their implication in different reactions of degradation or transformation [41,42]. Also the AA, evaluated by ABTS and DPPH assays, generally significantly increased in wines treated with EX. The storage time determined a significant decrease of TPC, F, A, and ABTS after 12 months of storage due to the oxidation, polymerization and complexation reactions which occur during storage [43]. The DPPH assay showed an opposite trend during storage compared to ABTS, with a significant increase. In fact, although the methods are very similar, each phenolic wine compound have a different response to each specific radical used in the assay [44]. The ABTS method is reported in literature as better reflecting the oxygen radical absorbance capacity in food matrices, and particularly highly pigmented hydrophilic antioxidants [45].

As regards the color parameters, CI was significantly higher in rosé wine added with EX for all time considered, probably due to the decoloring effect of sulfur dioxide which reduced the absorbance at 520 nm (red pigments) in the control wine. In a similar way, Gutiérrez-Escobar et al. [18] showed that the SO_2-added rosé wines (cv. Syrah) had a lower CI than wines treated with a stilbene vine-shoots extract with a purity of 99 %. Moreover, in all time considered the addition of EX increased the value of absorbance at 420 nm (yellow pigments). In a similar manner but in a white wine (Sauvignon blanc), Cruz and colleagues [17] reported an increase of absorbance at 420 nm (brown color) when Vineatrol® was used as an alternative to SO_2. Also in our case the brown color of the EX added could have resulted in more intense yellow tone of wine.

The trend observed for red wines was in accordance with Raposo et al. [16]. In particular, CI at bottling was significant higher in R-EX, whereas after 12 months of storage was significantly higher in R–C, in lines with the value of absorbance at 520 nm. This could be attributed to a short-term co-pigmentation of stilbenes with anthocyanins with hyperchromic effects, as suggested previously [46]. Finally, as regards H, the value slight increased during storage and with the addition of EX. For red wine, the increase of H value generally indicates the transition to typical brick-red color of aged wines, due to the reduction of anthocyanins (responsible for the red color of wine) and the formation of more stable polymeric complexes that contribute to color stabilization [47]. For rosé wine, the H trend suggests a more rapid evolution of color from red to yellow/orange [48].

3.2. Phenolic profile

The phenolic profiles of red and rosé wines and the results of two-way ANOVA analysis were reported in Tables 2 and 3, respectively. A total of 27 and 25 phenolic compounds were identified and quantified in red and rosé wines, respectively, including anthocyanins, phenolic acid, flavanols, and stilbenes. As shown, *T* was the variable that more significantly influenced the results while *Tr* variable influenced significantly above all anthocyanins and, obviously, stilbenes in both wines, and phenolic acids only in the rosé wine.

The total polyphenol compounds significantly decreased during storage and particularly for red wine where a decrease up to 80 % was observed (due to the dominant contribution of free anthocyanins), while for rosé wine the relative decrease was clearly lower. The decrease observed for red wine was attributed to anthocyanins amount, the most numerous and abundant class among phenolic compounds, due to the degradation or polymerization reactions which occur during wine storage [41,42]. For rosé wine, phenolic acid and flavanols were the most abundant phenolic compound classes and no significant variation were observed during storage. Among the single phenolic compounds, the most representative were: malvidin 3-glucoside (about il 50 % of total anthocyanins in red wine and up to 60 % in rosé wine) followed by malvidin 3-(6″-acetyl)-glucoside, petunidin 3-glucoside, peonidin 3-glucoside, and malvidin 3-(6″-*t*-coumaroyl)-glucoside for anthocyanins; gallic and caftaric acids and procyanidins for phenolic acid and flavanol class, respectively. The data were in accordance with findings reported in literature [41,49].

As regards the treatment, compared to the control wine, the addition of extract in red wines reduced the total anthocyanins concentration at bottling (−14.6 %) and after 3 (−33.3 %) and 6 months (−43.4 %) of storage. In a similar manner, r-EX wines had a lower concentration of total anthocyanins than r-C at bottling (−18.6 %) and after 3 months of storage (−24.8 %). However, no significant difference was found between the samples after 6 and 12 months of storage. These results were in agreement with Raposo et al. (2018) in which the sulfited control wines (cv. Syrah) showed the higher total concentration of anthocyanins, compared to wines treated with Vineatrol® at two different doses of stilbenes (50 and 100 mg L^{-1}). This can be due to the capacity of SO_2 to reduce the polymerization of anthocyanins by binding them [50]. Moreover, the adsorption of anthocyanins by the raw stilbenic extract or their reaction with other extract compounds (such as phenolic compounds, see Table S1) could explain this decrease. In fact, as reported before, anthocyanins are involved in a series of reactions, such as degradation and oxidation, aggregation and precipitation with other macromolecular compounds or formation of more stable pigments [51–53].

As regards individual pigments, the addition of the extract determined a significant decrease of delphinidin 3-glucoside, petunidin 3-glucoside, peonidin 3-glucoside, and malvidin 3-glucoside in both wines and malvidin 3-(6″-acetyl)-glucoside, malvidin 3-(6″-*t*-coumaroyl)-glucoside only in red wine. No statistically significant differences were found after 12 months of storage, with the exception of vitisin B, that was in slightly higher amount in the red wine with extract, and cyanidin-3-glucoside that was slightly more abundant in the control rosé wine. Moreover, the addition of the extract determined generally higher amount of phenolic acid and flavanols, even if only in few cases with statistical differences, and significantly higher amount of stilbenes. In particular, gallic acid, (+)-catechin, and *trans*-resveratrol significantly increased when vine-shoot extract was added, which amount significantly decreased during storage.

Table 3
Phenolic compounds (mg L^{-1}) of rosé wine.

Compounds				Storage time (T, months)								Tr	T	Tr*T
				Bottling (T0)		T3		T6		T12				
				r-C	r-EX	r-C	r-EX	r-C	r-EX	r-C	r-EX			
Anthocyanins	**RT**	**MH^+(m/z)**	**Fragments (m/z)**											
Delphinidin 3-glucoside[b]	6.00	465	303	4.2 ± 0.2a	3.4 ± 0.01b	3.0 ± 0.2b	2.1 ± 0.3c	1.8 ± 0.1cd	1.4 ± 0.1d	0.9 ± 0.2e	0.5 ± 0.1e	***	***	*
Cyanidin 3-glucoside[b]	6.47	449	287	1.6 ± 0.1a	1.3 ± 0.1b	1.2 ± 0.1b	0.8 ± 0.1c	0.8 ± 0.1c	0.7 ± 0.1cd	0.6 ± 0.1d	0.3 ± 0.1e	***	***	ns
Petunidin 3-glucoside[b]	6.87	479	317	7.3 ± 0.3a	6.0 ± 0.1b	5.7 ± 0.2b	4.0 ± 0.6c	3.5 ± 0.1cd	3.0 ± 0.1d	1.4 ± 0.4e	1.2 ± 0.2e	***	***	**
Malvidin 3,5-glucoside[b]	7.23	655	331	0.6 ± 0.1a	0.6 ± 0.1 ab	0.5 ± 0.1abc	0.5 ± 0.1abc	0.4 ± 0.1bc	0.4 ± 0.1c	0.4 ± 0.1c	0.3 ± 0.1c	ns	***	ns
Peonidin 3-glucoside[b]	7.35	463	301	5.4 ± 0.3a	4.4 ± 0.1b	4.2 ± 0.2b	3.1 ± 0.4c	2.9 ± 0.1c	2.3 ± 0.1c	1.4 ± 0.5d	1.3 ± 0.2d	***	***	*
Malvidin 3-glucoside[a]	7.64	493	331	39.0 ± 1.5a	32.6 ± 0.5b	30.0 ± 1.4b	22.6 ± 3.3c	19.4 ± 0.2cd	16.0 ± 0.4d	10.7 ± 0.3e	6.9 ± 0.5e	***	***	ns
Delphinidin 3-(6″-acetyl) glucoside[b]	7.91	507	303	0.6 ± 0.02a	0.6 ± 0.02a	0.3 ± 0.03b	0.2 ± 0.03c	0.2 ± 0.03c	0.2 ± 0.02c	–	–	*	***	**
Vitisin A2	8.1	561	399	0.6 ± 0.01a	0.6 ± 0.04a	0.5 ± 0.01ab	0.5 ± 0.01abc	0.5 ± 0.01abc	0.4 ± 0.03bc	0.4 ± 0.01abc	0.4 ± 0.02c	**	***	ns
Petunidin-3-(6″-acetyl)-glucoside[b]	8.71	521	317	0.6 ± 0.04a	0.5 ± 0.03b	0.4 ± 0.02bc	0.4 ± 0.01c	0.3 ± 0.05d	0.3 ± 0.04d	–	–	**	***	*
Peonidin-3-(6″-acetyl)- glucoside[b]	9.27	505	301	0.4 ± 0.04a	0.3 ± 0.03bc	0.3 ± 0.02b	0.3 ± 0.01bc	0.2 ± 0.03c	0.2 ± 0.03c	–	–	**	***	**
Malvidin 3-(6″-acetyl)-glucoside[b]	9.49	535	331	3.0 ± 0.2a	2.5 ± 0.1 ab	2.3 ± 0.1bc	1.8 ± 0.2cd	1.6 ± 0.03d	1.1 ± 0.3e	0.7 ± 0.1e	0.7 ± 0.1e	***	***	ns
Petunidin 3-(6″-*t*-coumaroyl)-glucoside[b]	10.13	625	317	0.3 ± 0.05a	0.3 ± 0.03 ab	0.2 ± 0.03abc	0.3 ± 0.01a	0.2 ± 0.01bcd	0.1 ± 0.07e	0.2 ± 0.01cd	0.2 ± 0.04de	*	***	**
Peonidin 3-(6″-*t*-coumaroyl)-glucoside[b]	10.27	639	331	0.3 ± 0.01a	0.3 ± 0.03a	0.3 ± 0.03a	0.3 ± 0.03 a	0.2 ± 0.01a	0.2 ± 0.01a	0.2 ± 0.03a	0.2 ± 0.01a	ns	ns	ns
Malvidin 3-(6″-*t*-coumaroyl)-glucoside[b]	10.67	609	301	1.2 ± 0.1a	1.1 ± 0.1a	1.1 ± 0.05a	0.7 ± 0.1b	0.6 ± 0.02bc	0.7 ± 0.2bc	0.6 ± 0.1bc	0.4 ± 0.1c	**	***	*
Σ Anthocyanins	10.83	639	331	*64.9 ± 2.4a*	*54.4 ± 0.8b*	*50.1 ± 2.4b*	*37.6 ± 5.1c*	*32.7 ± 0.4cd*	*27.0 ± 0.2d*	*17.6 ± 1.7e*	*12.4 ± 0.8e*	***	***	*
Phenolic acids	**RT**	**MH^-(m/z)**	**Fragments (m/z)**											
Gallic acid[a]	1.13	169	125	35.5 ± 1.8c	43.8 ± 2.8b	44.8 ± 2.5b	49.1 ± 0.9b	43.5 ± 1.9b	48.6 ± 3.3b	43.5 ± 2.8b	56.8 ± 3.8a	***	***	ns
Caftaric acid[a]	2.23	311	179,149,135	27.2 ± 07 ab	29.3 ± 0.4a	23.5 ± 0.1bc	26.6 ± 1.5 ab	23.3 ± 0.9bc	23.9 ± 0.5bc	21.8 ± 3.2c	24.4 ± 1.5bc	**	***	ns
Coutaric acid[c]	3.49	295	163,149	10.0 ± 0.7a	8.1 ± 0.2b	8.3 ± 0.1b	8.8 ± 1.4ab	8.2 ± 0.2b	8.1 ± 0.1b	8.0 ± 0.4b	8.6 ± 0.5ab	ns	ns	**
Fertaric acid[c]	4.71	325	193,149	4.4 ± 0.1a	4.2 ± 0.02 ab	4.5 ± 0.1a	3.6 ± 0.5b	3.9 ± 0.1ab	3.7 ± 0.2b	4.1 ± 0.1ab	3.9 ± 0.3ab	***	*	ns
Σ Phenolic acid				*76.9 ± 3.0b*	*85.4 ± 3.3ab*	*81.1 ± 2.5b*	*88.2 ± 4.3 ab*	*78.9 ± 2.9b*	*84.4 ± 3.9ab*	*77.4 ± 6.3b*	*93.6 ± 6.0a*	***	ns	ns

(*continued on next page*)

Table 3 (*continued*)

Compounds				Storage time (T, months)								Tr	T	Tr*T
				Bottling (T0)		T3		T6		T12				
				r-C	r-EX	r-C	r-EX	r-C	r-EX	r-C	r-EX			
Flavonols	**RT**	**MH⁻(*m/z*)**	**Fragments (*m/z*)**											
Procyanidin dimers[d]	2.58	577	451,425,407,289	35.0 ± 1.2a	35.0 ± 0.8a	31.7 ± 0.2 ab	30.1 ± 3.7b	24.1 ± 0.8c	21.1 ± 1.0c	21.4 ± 1.3c	23.6 ± 1.1c	ns	***	ns
(+)-Catechin[a]	3.23	289	245,179,205	9.1 ± 0.2c	22.8 ± 0.6a	8.4 ± 0.5c	15.5 ± 2.5b	7.4 ± 2.1c	13.4 ± 1.2b	7.0 ± 0.7c	14.1 ± 1.2b	***	***	***
Procyanidin dimers[d]	4.45	577	451,425,407,289	14.1 ± 0.7a	13.0 ± 0.7a	17.4 ± 0.8a	15.4 ± 1.8a	18.7 ± 2.3a	20.7 ± 0.1a	13.4 ± 1.2a	19.3 ± 2.5a	ns	**	ns
Procyanidin dimers4	4.54	577	451,425,407,289	20.1 ± 1.3 ab	20.8 ± 0.2a	15.9 ± 0.3c	17.4 ± 0.6bc	17.2 ± 0.6c	16.5 ± 0.5c	15.0 ± 0.7c	17.9 ± 2.3c	*	***	ns
(−)-Epicatechin[a]	5.44	289	245,205	3.6 ± 0.3abc	3.4 ± 0.7bc	3.5 ± 0.1abc	3.5 ± 0.8abc	4.7 ± 0.4 ab	4.0 ± 0.6 ab	2.5 ± 0.3c	5.0 ± 0.8a	ns	*	***
Σ Flavanols				*81.8 ± 2.3ab*	*95.1 ± 2.0a*	*76.9 ± 0.9b*	*82.0 ± 9.2ab*	*72.1 ± 6.1bc*	*75.8 ± 3.2b*	*59.0 ± 3.9c*	*79.9 ± 6.8b*	***	***	*
Stilbenes	**RT**	**MH⁺(*m/z*)**	**Fragments (*m/z*)**											
trans-Resveratrol[a]	9.08	229	135,211,119,107,											
183,193	0.2 ± 0.01e	40.3 ± 0.8a	0.1 ± 0.01e	13.7 ± 0.3c	0.1 ± 0.01e	16.9 ± 2.3b	0.3 ± 0.01e	6.1 ± 0.5d	***	***	***			
Total				*223.9 ± 6.6b*	*275.2 ± 6.7a*	*208.2 ± 5.0bcd*	*221.5 ± 18.4bc*	*183.8 ± 9.4de*	*204.2 ± 8.1bcd*	*154.5 ± 11.6e*	*192.1 ± 13.7e*	***	***	*

*Data followed by different letters indicate statistically significant differences at $p \leq 0.05$ according to two-way ANOVA with interaction followed by Tukey's test. Average value ± standard deviation (n = 3). Abbreviations: r-C, rosé control wine; r-EX, rosé wine with extract. Tr, treatment variable; T, storage time variable; ns, not significant; *, **, ***, significant at $p \leq 0.05$, $p \leq 0.01$, $p \leq 0.001$, respectively.

[a] Quantified using corresponding standards.

[b] expressed as Malvidin 3-glucoside equivalents.

[c] expressed as Caftaric acid equivalents.

[d] expressed as (+)-Catechin equivalents.

Table 4
Volatile compounds (VoCs, μg L^{-1}) in Negramaro red wines.

Compounds			Storage time (T, months)								*Tr*	*T*	*Tr*T*
			Bottling (T0)		T3		T6		T12				
			R–C	R-EX	R–C	R-EX	R–C	R-EX	R–C	R-EX			
Carboxylic acids	**RT (min)**	**Most abundant ions (m/z)**											
Acetic acid	28.06	43,45,60	*211 ± 22bc	180 ± 24bc	148 ± 14c	149 ± 2c	194 ± 15bc	242 ± 35b	330 ± 51a	169 ± 3c	**	***	***
Hexanoic acid	43.23	60,73,87	69 ± 3bc	58 ± 4cd	57 ± 2cd	73 ± 1bc	96 ± 12a	84 ± 16 ab	38 ± 5de	33 ± 3e	ns	***	*
Octanoic acid	50.47	60,73,101	283 ± 78a	272 ± 30a	301 ± 19a	297 ± 37a	355 ± 49a	274 ± 49a	119±4b	102 ± 21b	ns	***	ns
Nonanoic acid	53.77	60,73,57,115,129	8 ± 2c	13 ± 1c	12 ± 1c	12 ± 1c	63 ± 9a	43 ± 4b	9 ± 1c	5 ± 1c	**	***	***
Decanoic acid	57.03	60,73,129	22 ± 2c	16 ± 1cd	48 ± 3a	41± 5ab	39 ± 3b	22 ± 3c	11 ± 2d	9 ± 0.5d	***	***	**
Total			*593 ± 100ab*	*538 ± 59b*	*567 ± 13ab*	*571 ± 37ab*	*747 ± 81a*	*666 ± 105 ab*	*506 ± 52b*	*317 ± 18c*	**	***	ns
Alcohols													
1-Propanol	10.56	31,29,27	116 ± 7b	131 ± 4a	59 ± 2de	48±3e	77±6c	54 ± 9de	131 ± 3a	68 ± 1cd	***	***	***
Isobutanol	12.41	43,41,42	677 ± 77a	735 ± 42a	515 ± 36b	487 ± 17b	664 ± 13a	460 ± 76b	528 ± 36b	534 ± 15b	*	***	***
1-Butanol	14.43	56,31,41	13±1ab	13±1ab	11 ± 1bc	9 ± 1c	13 ± 1a	9 ± 2c	11 ± 1abc	11 ± 1abc	**	***	**
Isoamyl alcohol	17.1	55,42,70	18325 ± 975a	19886 ± 576a	11017 ± 710b	11142 ± 26b	12349 ± 1647b	10349 ± 1742b	12258 ± 604b	12245 ± 305b	ns	***	*
4-Methyl-1-pentanol	21.71	56,41,43,69	6±1bc	7±1abc	6 ± 1c	7 ± 1abc	8 ± 1a	7 ± 1abc	7 ± 1abc	8 ± 1ab	ns	*	*
3-Methyl-1-pentanol	22.28	56,55,69	19 ± 3a	18 ± 3a	15 ± 0.5a	15 ± 0.2a	17 ± 1a	16 ± 3a	16 ± 1a	18 ± 1a	ns	ns	ns
1-Hexanol	23.44	56,43,41,55,69	261 ± 9ab	267 ± 1a	214 ± 15bc	224±6abc	238 ± 13abc	200 ± 41c	231 ± 11abc	224 ± 4abc	ns	***	**
3-Hexen-1-ol	24.86	67,41,39,55,82	7 ± 1a	7 ± 1 ab	5 ±1 ab	5 ± 0.1b	5 ±1 ab	5 ± 1b	7 ± 1a	6 ±1 ab	**	***	*
Methionol	38.58	106,61,58,57,31	8 ± 1b	12 ± 1a	12 ± 0a	10 ± 0 ab	12 ± 2a	11 ± 2ab	12 ± 1a	13 ± 1a	ns	*	**
Benzyl alcohol	44.46	79,108,107,77	17 ± 2bc	16 ± 1bc	12 ± 0c	15 ± 1bc	19 ± 2ab	17 ± 5bc	25 ± 2a	19 ± 1b	ns	***	*
Phenylethyl alcohol	45.67	91,92,65,122	4990 ± 546 ab	5413 ± 759a	3283 ± 129d	3906 ± 80bcd	3622 ± 440cd	3056 ± 513d	4570 ± 400abc	4255 ± 235abcd	ns	***	ns
Total			*24438 ± 1494a*	*26503 ± 1322a*	*15153 ± 745b*	*15868 ± 92b*	*17615 ± 2118b*	*14184 ± 2372b*	*17797 ± 1029b*	*17400 ± 517b*	ns	***	*
Esters													
Methyl acetate	5.91	43,74	5±1ab	6 ± 1a	5 ± 1ab	6 ± 1a	4 ± 1b	4 ± 1ab	6 ± 1a	5 ± 1ab	ns	**	ns
Ethyl acetate	6.82	43,61,45,29,70	600 ± 55bc	664 ± 45b	535 ± 40bc	509 ± 17c	655 ± 38bc	584 ± 111bc	998 ± 46a	949 ± 19a	ns	***	ns
Isobutyl acetate	9.85	43,56,73	4 ± 1c	5 ± 1abc	4 ± 1bc	4 ± 1c	6 ± 1ab	4 ± 1c	6 ± 1a	4 ± 1c	***	***	**
Ethyl butyrate	10.59	71,43,29,88,27	–	–	37 ± 1ab	34 ± 1ab	40 ± 3a	33 ± 7 ab	31 ± 2b	35 ± 3ab	ns	***	*
Ethyl isovalerate	11.66	88,57,29,85,60	–	–	3 ± 0.2bc	2 ± 0.1c	4 ± 0.2b	3 ± 1b	6 ± 1a	4 ± 0.2b	***	***	***
Isoamyl acetate	13.74	43,70,55,41,61	162 ± 14b	117 ± 45bc	249 ± 13a	229 ± 6a	273 ± 17a	167 ± 23b	162 ± 8b	102 ±8c	***	***	*
Ethyl hexanoate	18.38	88,29,43,27,99	45 ± 3d	39 ± 8d	130 ± 5c	116 ± 11c	283 ± 24a	102 ± 17c	117 ± 9c	179 ± 3b	***	***	***
Hexyl acetate	20.05	43,56,55,61,42	7 ± 1a	7 ± 0.1a	7 ± 0.1a	7 ± 1a	8 ± 1a	7 ± 1a	4 ± 1b	5 ± 1b	ns	***	*
Ethyl lactate	23.15	45,29	216 ± 1c	272 ± 19bc	227 ± 10c	216 ± 17c	301 ± 23bc	259 ± 53c	468 ± 70a	356 ± 17b	ns	***	**
Ethyl octanoate	27.10	88,101,57,127,60	191 ± 8c	143 ± 18c	641 ± 14a	412 ± 17b	771 ± 115a	580 ± 117 ab	420 ± 100b	720 ± 34a	ns	***	***
Isoamyl lactate	32.70	45,43,70,55,71	21 ± 3d	25 ± 3cd	25 ± 1cd	24 ± 2cd	40 ± 2b	32 ± 5bc	51 ± 5a	27 ± 2cd	***	***	***
Diethyl succinate	36.92	101,29,129,27,28	450 ± 28d	520 ± 105cd	530 ± 41cd	534 ± 19cd	746 ± 92c	562 ± 87cd	1848 ± 112a	1527 ± 114b	**	***	**
Butyl ethyl succinate	40.1	101,129,29	5 ± 1d	7 ± 0.2c	8 ± 1bc	10 ± 1a	9 ± 0.2b	8 ± 0.2bc	6 ±1c	4 ± 1d	*	***	***
Phenethyl acetate	42.29	104,43,91	309 ± 22a	281 ± 31a	224 ± 12b	209 ± 8b	212 ± 19b	159 ± 18c	137 ± 5c	151 ± 7c	*	***	*
Ethyl isopentyl succinate	45.14	101,129,71	42 ± 1c	44 ± 3c	47 ± 2bc	46 ± 1bc	76 ± 5a	75 ± 5a	77 ± 7a	56 ± 4b	**	***	***

(*continued on next page*)

Table 4 (*continued*)

Compounds			Storage time (T, months)								*Tr*	*T*	*Tr*T*
			Bottling (T0)		T3		T6		T12				
			R–C	R-EX	R–C	R-EX	R–C	R-EX	R–C	R-EX			
Total			*2056 ± 102d*	*2128 ± 143d*	*2673 ± 72c*	*2358* ±16cd	*3426 ± 100b*	*2580* ±421cd	*4339 ± 225a*	*4122 ± 131a*	**	**	**
Aldehydes													
Acetaldehyde	4.79	29,44,43,15,42	6 ± 1d	5 ± 0.4d	12 ± 2bc	8 ± 1cd	26 ± 4a	4 ± 1d	15 ± 2b	12 ± 1bc	***	***	***
Octanal	20.85	43,44,41,56,84	5 ± 1bc	5 ± 0.5bc	5 ± 0.3bc	4 ± 0.1c	7 ± 3ab	9 ± 1a	–	4 ± 1c	*	***	***
Nonanal	25.41	57,41,43,56,44,55	26 ± 1c	13 ± 2d	23 ± 2c	15 ± 1d	35 ± 2b	72 ± 4a	6 ± 1e	24 ± 2c	***	***	***
Benzaldehyde	31.10	77,106,105,51,50	12 ± 2cd	12 ± 1cd	40 ± 4a	11 ± 2cd	30 ± 4b	11 ± 2cd	14 ± 1c	8 ± 1d	***	***	***
Total			*49 ± 3c*	*35 ± 3d*	*79 ± 7b*	*38* ± 2cd	*98 ± 6a*	*96 ± 4a*	*36 ± 3d*	*48 ± 2c*	***	***	***
Terpenes													
Linalool	31.66	71,93,55,43,41,80	33 ± 2 ab	34 ± 2 ab	25 ± 1b	27 ± 2b	38 ± 4a	32 ± 6 ab	38 ± 5a	29 ± 1ab	ns	**	*
α-Terpineol	37.71	59,93,121,136,67	9 ± 1cd	10 ± 1c	7 ± 1cd	8 ± 1cd	10 ± 1c	7 ± 1cd	22 ± 1a	15 ± 2b	***	***	***
Total			*43* ± 3bcd	*44* ± 3bcd	*33 ± 1d*	*35* ± 3cd	*47 ± 4b*	*39* ± 7bcd	*60 ± 5a*	*44 ± 1bc*	***	***	***
Norisoprenoids													
Damascenone	42.42	69,121,41	13 ± 2b	16 ± 1a	11 ± 1c	9 ±1d	9 ± 1d	9 ± 1d	9 ± 1d	8 ± 1d	ns	***	***
Other compounds													
Acetoin	20.77	45,43	5 ± 1b	8 ± 1a	8 ± 3a	8 ± 1a	9 ± 1a	8 ± 1a	6 ± 1b	5 ± 1b	ns	*	*
4-Ethylphenol	54.29	107,122,77	–	102 ± 2b	–	243 ± 19a	–	266 ± 26a	–	9 ± 1c	***	***	***
Total			5 ± 1c	110 ± 2b	8 ± 0.3c	250 ± 19a	9 ± 1c	274 ± 25a	6 ± 0.4c	14 ± 2d	***	***	***
Total VOCs			*27197 ± 1660ab*	*29374 ± 1441a*	*18519* ±828cd	*19130* ±49cd	*21952* ±2289cd	*17847 ± 2928d*	*22752 ± 1304bc*	*21954* ±596cd	ns	***	*

*In row, data followed by different letters indicate statistically significant differences at $p \leq 0.05$ according to two-way ANOVA with interaction followed by Tukey's test. Average value ± standard deviation (n = 3). R–C, red control wine; R-EX, red wine with extract; *Tr*, treatment variable; *T*, storage time variable; ns, not significant; *, **, ***, significant at $p \leq 0.05$, $p \leq 0.01$, $p \leq 0.001$, respectively.

Table 5
Volatile compounds (μg L^{-1}) in Sangiovese rosé wines.

Compounds			Storage time (T, months)								*Tr*	*T*	*Tr*T*
			Bottling (T0)		T3		T6		T12				
			r-C	r-EX	r-C	r-EX	r-C	r-EX	r-C	r-EX			
Carboxylic acids	**RT (min)**	**Most abundant ions (m/z)**											
Acetic acid	28.06	43,45,60	124 ± 5cd	181 ± 13ab	115 ± 6d	153 ± 19bc	125 ± 9cd	121 ± 6cd	201 ± 25a	202 ± 12a	***	***	**
Hexanoic acid	43.23	60,73,87	44 ± 4c	46 ± 10c	70 ± 8b	88 ± 13a	68± 5b	69 ± 4b	50 ± 7c	46 ± 3c	*	***	**
Octanoic acid	50.47	60,73,101	261 ± 8bcd	341 ± 13 ab	357 ± 44 ab	437 ± 84a	313 ± 23b	297 ± 26bc	208 ± 15cd	181 ± 15d	ns	***	*
Nonanoic acid	53.77	60,73,57,115,129	8 ± 1b	13 ± 1b	13 ± 1b	16 ± 2b	41 ± 8a	48 ± 4a	10 ± 2b	10 ± 1b	*	*	ns
Total			*437 ± 15b*	*581 ± 10ab*	*555 ± 49ab*	*694 ± 116a*	*546 ± 43b*	*536 ± 27b*	*469 ± 46b*	*440 ± 11b*	*	***	*
Alcohols													
1-Propanol	10.56	31,29,27	37±1b	43±5a	17 ± 1b	24 ± 4a	15 ± 1b	14 ± 1b	25 ± 2a	23 ± 1a	*	***	**
Isobutanol	12.41	43,41,42	328 ± 12abc	380 ± 31a	274 ± 27cd	343 ± 49abc	225 ± 15d	223 ± 16d	356 ± 19 ab	303 ± 25bc	ns	***	***
1-Butanol	14.43	56,31,41	11±0.2ab	11±0.1ab	9 ± 1bc	13 ± 2a	8 ± 1cd	7 ± 0.3d	12 ± 0.3ab	9 ± 1bc	ns	***	***
Isoamyl alcohol	17.1	55,42,70	11537 ± 383b	12597 ± 481a	7694 ± 748de	9343 ± 1204cd	6264 ± 542ef	5875±244f	10279 ± 577bc	8834 ± 393cd	ns	***	**
4-Metyl-1-pentanol	21.71	56,41,43,69	8 ± 1a	7 ± 1 ab	5 ± 0.4bc	7 ± 1ab	5 ± 1c	4 ± 0.6c	7 ± 0.1ab	6 ± 0.1bc	ns	***	**
3-Metyl-1-pentanol	22.28	56,55,69	13± 0.5ab	12 ± 0.5b	12 ± 1b	15 ± 2a	8 ± 1c	8 ± 0.2c	13 ± 1 ab	12 ± 0.3b	ns	***	**
1-Hexanol	23.44	56,43,41,55,69	344 ± 11ab	381 ± 13a	297 ± 25b	365 ± 36a	232 ± 17c	220 ± 15c	359 ± 25a	325 ± 19ab	ns	***	**
3-Hexen-1-ol	24.86	67,41,39,55,82	9 ± 1ab	9 ±1ab	8 ± 1b	11 ± 2a	7 ± 1b	7 ± 0.6b	10 ± 0.4ab	9 ± 0.1ab	ns	**	ns
Methionol	38.58	106,61,58,57,31	15 ±1b	20 ± 0.2a	7 ± 1c	9 ± 2c	13 ± 2b	13 ± 2b	21 ± 1a	20 ± 1a	**	***	*
Benzyl alcohol	44.46	79,108,107,77	48 ± 1c	75 ± 5b	49 ± 4c	71 ± 11b	44 ± 4c	46 ± 2c	98 ± 6a	79 ± 2b	**	***	***
Phenylethyl Alcohol	45.67	91,92,65,122	5327 ± 162ab	5956 ± 806ab	3938 ± 323cd	5087 ± 680bc	2879 ± 232d	2880 ± 208d	6425 ± 161a	5430 ± 234ab	ns	***	**
Total			*17657 ± 560 ab*	*19470 ± 1247a*	*12310 ±1122cd*	*15287 ± 1990b*	*9698 ± 810de*	*9297 ± 414e*	*17602 ± 656 ab*	*15051 ± 211bc*	*	**	**
Esters													
Ethyl acetate	6.82	43,61,45,29,70	627 ± 29cd	798 ± 94ab	543 ± 21de	473 ± 58ef	445 ± 39ef	352 ± 14f	920 ± 29a	697 ± 40bc	*	***	***
Ethyl butyrate	10.59	71,43,29,88,27	18 ± 1abc	20 ± 2a	20 ± 2ab	18 ± 5abc	15 ± 1bc	11 ± 1c	19 ± 1ab	15 ± 1bc	ns	***	*
Isoamyl acetate	13.74	43,70,55,41,61	42 ± 1c	45 ± 6c	44 ± 6a	55 ± 11c	42 ± 3c	43 ± 5c	56 ± 1c	44 ± 2b	**	***	***
Ethyl hexanoate	18.38	88,29,43,27,99	70 ± 3 ab	60 ± 5b	82 ± 8a	54 ± 9b	68 ± 14a	72 ± 2ab	55 ± 7b	70 ± 4 ab	ns	ns	***
Ethyl lactate	23.15	45,29	362 ± 11cde	430 ± 43bcd	345 ± 32de	453 ± 60bc	324 ± 22e	323 ± 18e	620 ± 45a	475 ± 19b	ns	***	***
Ethyl octanoate	27.10	88,101,57,127,60	37 ± 7de	12 ± 1de	112 ± 6b	139 ± 21a	71 ± 3c	14 ± 1e	52 ± 7cd	58 ± 6cd	**	***	***
Isoamyl lactate	32.70	45,43,70,55,71	42 ± 1a	45 ± 6a	44 ± 6a	55 ± 11a	42 ± 3a	43 ± 5a	56 ± 1a	44 ± 2a	ns	ns	*
Diethyl succinate	36.92	101,29,129,27,28	2528 ± 111ab	2856 ± 230a	1753 ± 148c	2235 ± 291b	1188 ± 86d	1182 ± 86d	2940 ± 88a	2593 ± 106 ab	ns	***	**
Butyl ethyl succinate	40.1	101,129,29	8 ± 1b	11 ± 1a	8 ± 0.3b	11 ± 2a	5 ± 0c	5 ± 0c	11 ± 1a	10 ± 1a	***	***	***

(*continued on next page*)

Table 5 (*continued*)

Compounds			Storage time (T, months)								*Tr*	*T*	*Tr*T*
			Bottling (T0)		T3		T6		T12				
			r-C	r-EX	r-C	r-EX	r-C	r-EX	r-C	r-EX			
Phenethyl acetate	42.29	104,43,91	58 ± 2a	67 ± 8a	41 ± 3b	56 ± 6a	16 ± 2d	26 ± 3cd	35 ± 3bc	44 ± 2b	***	***	***
Ethyl isopentyl succinate	45.14	101,129,71	51 ± 2bc	62 ± 5ab	55 ± 7ab	65 ± 9a	40 ± 4cd	33 ± 2d	59 ± 3 ab	57 ± 4 ab	ns	***	*
Total			*3818 ± 116bc*	*4379 ± 326ab*	*3051 ± 208d*	*3577 ±458cd*	*2227 ± 126e*	*2075 ± 96e*	*4783 ± 63a*	*4094 ± 157bc*	ns	***	***
Aldehydes													
Acetaldehyde	4.79	29,44,43,15,42	4 ± 1c	26 ± 3a	8±1c	8 ± 2c	14 ± 2b	7 ± 0.4c	13 ± 1b	7 ± 0.4c	**	***	***
Nonanal	25.41	57,41,43,56,44,55	58 ± 7b	51 ± 5b	154 ± 18a	22 ± 3c	50 ± 4b	58 ± 2b	10 ± 1c	5 ± 0.3c	***	***	***
Furfural	28.59	96,95,39,38,29	16 ± 1c	7 ± 1d	14 ± 1c	8 ± 2d	14 ± 2c	7 ± 0.3d	64 ± 2a	41 ± 1b	***	***	***
Total			*78 ± 8b*	*84 ± 7b*	*176 ± 19a*	*38 ± 7d*	*78 ± 1b*	*72 ± 2bc*	*87 ± 1b*	*54 ± 1cd*	***	***	***
Terpenes													
Linalool	31.66	71,93,55,43,41,80	43 ± 1ab	47 ± 2a	35 ± 4bcd	39 ± 6abc	29 ± 2de	25 ± 2e	37 ± 1bcd	31 ± 1cde	ns	***	*
α-Terpineol	37.71	59,93,121,136,67	31 ± 1bc	36 ± 4 ab	24 ± 2c	33 ± 4b	16 ± 2d	25±2d	37 ± 1a	31 ± 1ab	ns	***	**
Total			*74 ± 2ab*	*83 ± 3a*	*60± 6c*	*72 ± 10abc*	*45 ± 3d*	*40 ± 3d*	*78 ± 3ab*	*68 ± 2bc*	ns	***	*
Noroisoprenoids													
Damascenone	42.42	69,121,41	7 ± 0.2a	6 ± 1a	4 ± 0.3bc	6 ± 1a	3 ± 1c	3 ± 0.3c	5 ± 0.1b	6 ± 0.1a	***	***	***
Other compounds													
Acetoin	20.77	45,43	16 ± 1b	26 ± 2a	11 ± 1b	5 ± 1c	15 ± 1b	3 ± 0d	26 ± 4a	10 ± 2c	***	***	***
4-ethylphenol	54.29	107,122,77	9 ± 0.1d	12 ± 2d	10 ± 1d	83 ± 4b	8 ± 1d	58 ± 5c	13 ± 2d	108 ± 3a	***	***	***
Total			*25 ± 1e*	*38 ± 2d*	*21 ± 2e*	*88 ± 5b*	*23 ± 2e*	*61 ± 5c*	*39 ± 5d*	*118 ± 3a*	***	***	***
Total VOCs			*22043 ± 660ab*	*24641 ± 1515a*	*16175 ± 1374c*	*19761 ± 2570b*	*12620 ±984cd*	*12084 ± 532d*	*23063 ± 565ab*	*19830 ± 196b*	ns	***	**

*In row, data followed by different letters indicate statistically significant differences at $p \leq 0.05$ according to two-way ANOVA with interaction followed by Tukey's test. Average value ± standard deviation (n = 3). r-C, rosé control wine; r-EX, rosé wine with extract; *Tr*, treatment variable; *T*, storage time variable; ns, not significant; *, **, ***, significant at $p \leq 0.05$, $p \leq 0.01$, $p \leq 0.001$, respectively.

3.3. Volatile compounds

SPME/GC-MS analysis of red and rosé wines allowed to identify 40 and 34 different volatile organic compounds, respectively and grouped in six classes: carboxylic acids, alcohols, esters, aldehydes, terpenes, noroisoprenoids and other compounds (Tables 4 and 5). As shown, storage time was the variable that more influenced the amount of volatiles of the wines, that on overall were comparable to data reported in literature [54]. Immediately after bottling the wines showed the highest total amount of volatile compounds and alcohols were the most abundant group of compounds, followed by esters and carboxylic acids. The extract addition generally significantly influenced minor volatile compounds.

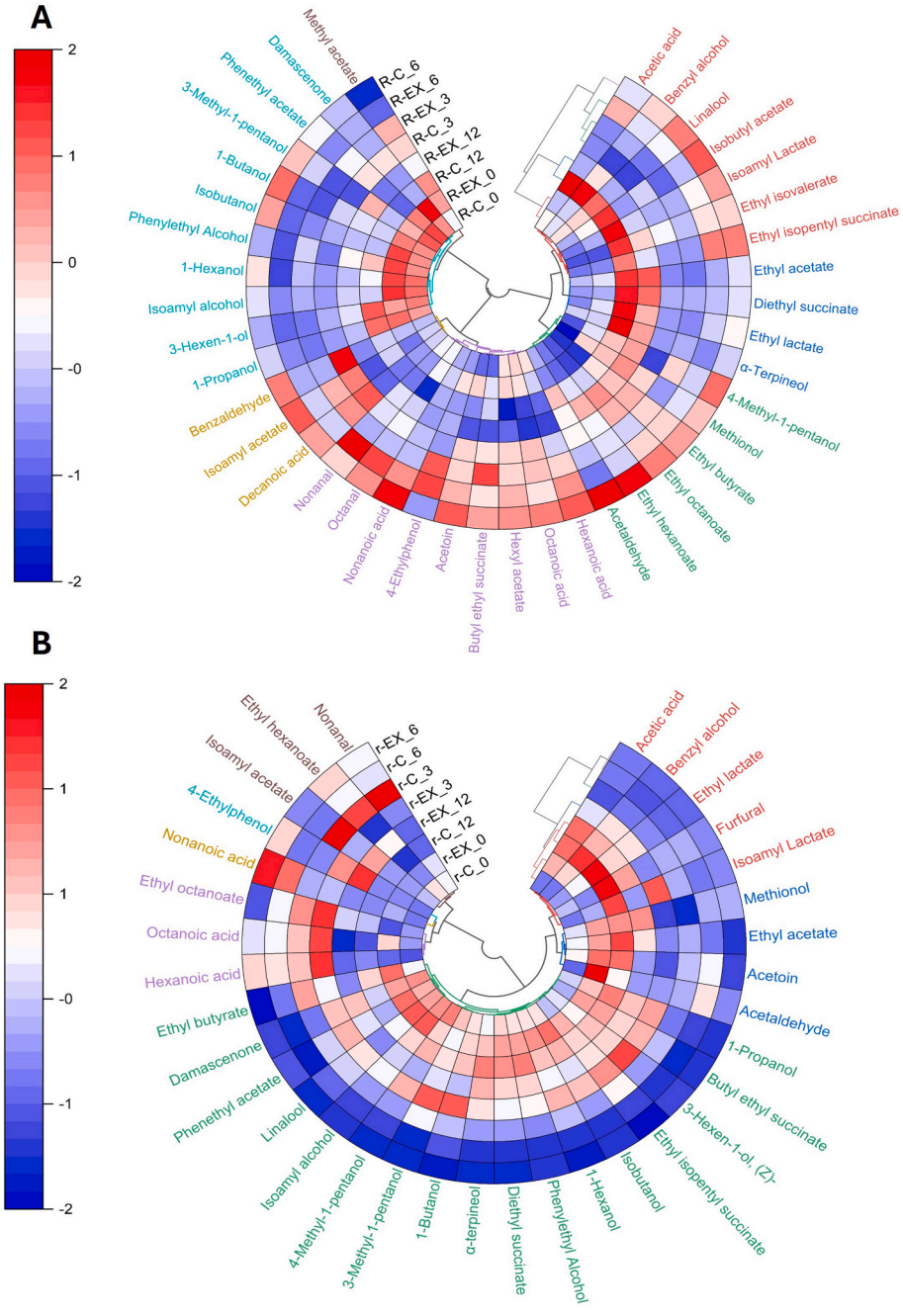


Fig. 2. Polar heatmap with a circular dendrogram derived from hierarchical clustering of the volatile profile of red cv. Negramaro (**A**) and rosé cv. Sangiovese (**B**) wines. Abbreviations: R–C, red control wine; R-EX, red wine with extract; r-C, rosé control wine; r-EX, rosé wine with extract; 0, 3, 6, and 12, months of storage.

A polar heatmap with a circular dendrogram derived from a hierarchical cluster analysis is shown in Fig. 2A, to evaluate the effects of time and extract on the volatile patterns of the red wine cv. Negramaro samples. In fact, the wines were clustered into four homogeneous groups: i) R–C0 and R-EX0; ii) R–C12 and R-EX12; iii) R–C3, R-EX3 and R-EX6; iv) R–C6. The volatile compounds were clustered into seven groups characterized in function of their distribution in the different samples. The first two clusters (analysing the polar heatmap clockwise) particularly characterized R–C and R-EX after 12 months of storage and R–C after 6 months including: seven esters such as lactate and succinate esters, typically formed during aging [36], ethyl acetate (sweet and fruity notes) [55] and diethyl succinate (fruity and cooked apple notes) [56]; one carboxylic acid (acetic acid), two terpenes such as linalool (citrus and floral notes) [56], benzyl alcohol (sweet and floral notes) [57]; acetic acid, with the sharp, pungent, sour vinegar notes that it could determine in wine [56]. As shown in Table 4, no statistically significant difference was found between R–C and R-EX, except for wines obtained after 12 months of storage. In this case, the concentration of acetic acid was lower in R-EX than in R–C.

The third, fourth and fifth clusters were composed of four aldehydes, six esters, four carboxylic acid such as nonanoic acid (pungent or fatty aromas) [58], two alcohols, 4-ethylphenol and acetoin. In general, most of these compounds were particularly present in all wine after 3, 6, and 12 months of storage. Among the aldehydes, acetaldehyde that is the product of ethanol oxidation (Fenton reaction) [59] characterized control wines during storage; benzaldehyde, which was particularly present in R–C wines after 3 and 6 months of storage, could impart a sweet and almond flavour to the wine [57]. Both aldehydes are oxidation markers [3]. Their apparent decrease after 12 months could be explained with the reactivity of aldehydes, with insufficient turnover due to the consumption of dissolved oxygen. Therefore, the vine-shoot extract would have acted either slowing down oxidation or reacting with aldehydes. In addition, higher concentrations of two aldehydes (nonanal and octanal) responsible for fruity and citrus notes were found in wine samples after 6 months of storage. In general, all control wines and both R–C and R-EX wines immediately after bottling were characterized by a higher concentration of alcohols such as phenylethyl alcohol (floral, rose and honey notes) [60] or isoamyl alcohol.

As regards rosé wine, the polar heatmap with circular dendrogram derived from the hierarchical cluster analysis is shown in Fig. 2B. The wines were clustered into four homogeneous groups, while the volatile compounds were clustered into seven groups. The first two clusters of volatiles consisted of two aldehydes (acetaldehyde and furfural), three esters (ethyl lactate, isoamyl lactate, ethyl acetate), two alcohols (methionol and benzyl alcohol), one carboxylic acid (acetic acid) and acetoin. In general, these compounds characterized the wines at bottling and after 12 months of storage and the wine r-EX after 3 months of storage. Specifically, the addition of EX resulted in a slight increase of acetic acid at bottling and after 3 months of storage, although statistically significant. However, no statistically significant difference was found after 6 and 12 moths of storage. Acetoin, described with attributes such as "buttery" or "creamy", is one of the volatile compounds produced during fermentation, and its concentration can change considerably during aging due to oxidative phenomena [61]. The third cluster included seventeen compounds mostly present in all wines considered, except in wines analyzed at bottling: nine alcohols; five esters important for the fruity and floral aroma of wine [62]; two terpenes, linalool which can determine citrus notes in wine and α-terpineol and β-damascenone which can determine floral and honey notes [63]. The fourth and fifth clusters contained compounds particularly present in wines after 3 and six months of storage: hexanoic, octanoic and nonanoic acids that impart vegetal aromas and vinous character to the wine [56]. The sixth cluster was represented by 4-ethylphenol, particularly present in all wines with added extract after 3, 6 and 12 months of storage. This volatile phenol typically derives from the metabolism of the yeast *Brettanomyces bruxellensis*, and, when in sufficiently high amounts, determine the onset of the Brett defect. Even though the levels found for these compounds presumably did not negatively affect the sensory profile of the wines (as shown by the sensory analysis discussed in Section 3.5), this effect on the volatile pattern could suggest a lower antimicrobial protection of the extract, at the doses adopted in this research, compared to SO_2.

3.4. Covariate and correlation analysis

Fig. 3 reports the results of point biserial partial correlation analysis of the chemical data with the *treatment* variable for both red (A) and rosé wine (B). Point biserial correlation allows to correlate quantitative variables with a binomial variable (control/extract, in the present case). The figure reports the top-25 correlated variables. Correlation parameters for all the chemical variables, are reported in Supplementary Tables S3 and S4.

As regards the red wine (Fig. 3A), the addition of the extract was positively correlated in particular with the phenolic classes represented in the extract itself. Besides this expected finding, other indices related to phenolic compounds were related to the addition of the vine-shoot extract. In particular, procyanidin dimers was positively correlated, suggesting increased polymerization reactions, as pointed out also by the positive correlation of hue. The positive correlation of 4-ethylphenol, instead, could indicate lower antimicrobial protection compared to SO_2. On the other hand, individual anthocyanins and oxidation aldehydes were negatively related with the use of the extract. This highlights a possible shift of the oxidative protection when replacing SO_2 with the vine-shoot extract, with a lower protection towards anthocyanin either degradation or condensation and higher protection towards carbonyl formation.

A similar pattern of correlation was observed for the rosé wine (Fig. 3B), with some further relevant aspects. Caftaric acid was positively correlated with the use of the extract. Considering that caftaric acid is readily oxidized in wines, this could be considered another indicator of the antioxidant activity of the extract. Skroza et al. [64] showed that the interaction of resveratrol with the oxidation phenomena of wine phenolic compounds strongly depends on the phenolic compounds involved in the interaction and could

have opposite effects. The present results seem to confirm the findings. Moreover, the combined addition of the vine-shoot extract and of SO_2 in the rosé wine was at the same time positively correlated with the color intensity and negatively correlated with individual anthocyanins. This could be explained in different ways. First of all, the decolorizing effect of SO_2 in control wine should not be disregarded in the control rosé wine, compared to the same wine with reduced SO_2. This effect of sulfur dioxide, in fact, is more evident in rosé wines [5]. On the other hand, hyperchromic interactions of stilbenes with anthocyanins have been suggested [46] and could be responsible of an increase of CI in the rosé wine with the combination of SO_2 and vine shoot extract.

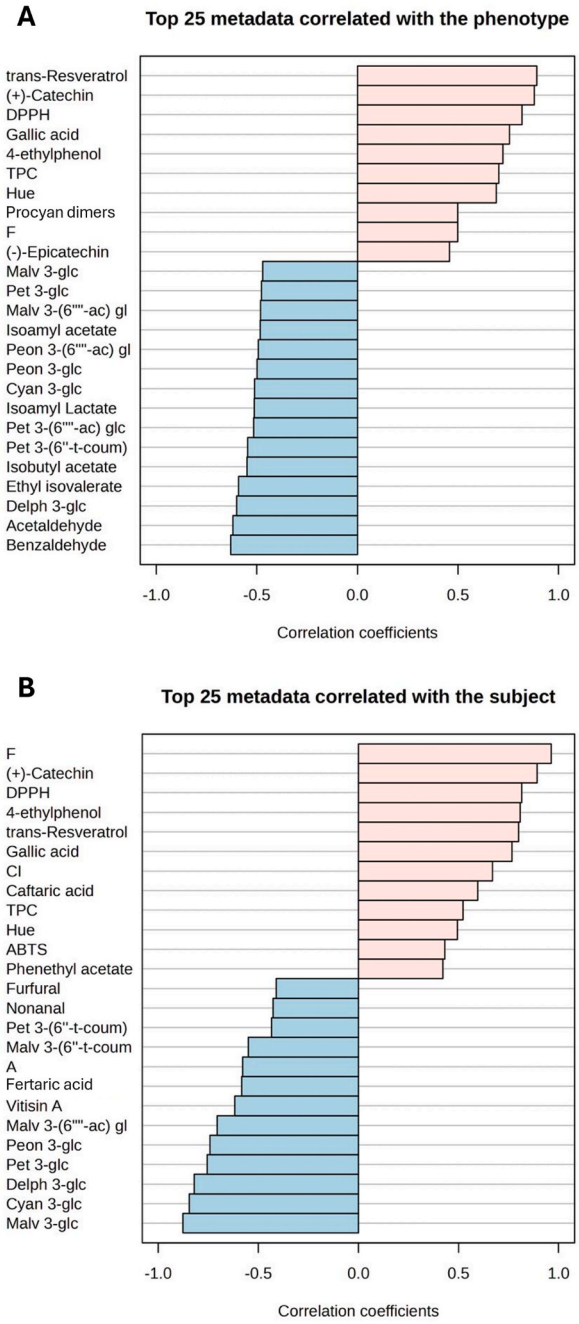


Fig. 3. Results of point biserial partial correlations of chemical parameters with the treatment variable (levels: control wine, wine with extract) for the red (**A**) and the rosé wine (**B**). Abbreviations: Delph 3-glc, Delphinidin 3-glucoside; Cyan, 3-glc, Cyanidin 3-glucoside; Pet 3-glc, Petunidin 3-glucoside; Peon 3-glc, Peonidin 3-glucoside; Malv 3-glc, Malvidin 3-glucoside; Pet 3-(6″-ac) glc, Petunidin-3-(6″-acetyl)-glucoside; Peon 3-(6″-ac) gl, Peonidin-3-(6″-acetyl)-glucoside; Malv 3-(6″-ac) gl, Malvidin 3-(6″-acetyl)-glucoside; Pet 3-(6″-*t*-coum), Petunidin 3-(6″-*t*-coumaroyl)-glucoside; Malv 3-(6″-*t*-coum, Malvidin 3-(6″-*t*-coumaroyl)-glucoside.

Table 6
Results of sensory analysis and overall judgment of wines during storage.

Phase	Descriptors	Samples	Storage time (T, months)				*Tr*	*T*	*Tr*T*
			Bottling (T0)	T3	T6	T12			
Visual	Color Intensity	R–C	*8.6 ± 0.2ab	8.7 ± 0.3ab	8.4 ± 0.3ab	8.4 ± 0.3ab	ns	*	ns
		R-EX	8.9 ± 0.2a	8.1 ± 0.2ab	7.9 ± 0.4b	8.3 ± 0.3ab			
		r-C	5.6 ± 0.2c	7.3 ± 0.6ab	6.9 ± 0.5b	7.1 ± 0.2b	***	***	*
		r-EX	6.6 ± 0.5bc	7.3 ± 0.3ab	8.3 ± 0.3a	8.4 ± 0.1a			
	Viscosity	R–C	7.4 ± 0.4a	8.1 ± 0.4a	7.9 ± 0.3a	7.8 ± 0.3a	ns	ns	ns
		R-EX	7.7 ± 0.3a	7.7 ± 0.3a	7.6 ± 0.3a	7.4 ± 0.3a			
		r-C	5.1 ± 0.2d	7.0 ± 0.3a	6.9 ± 0.2 ab	5.2 ± 0.3cd	*	***	*
		r-EX	4.9 ± 0.4d	7.4 ± 0.5a	7.8 ± 0.4a	6.1 ± 0.4bc			
Olfactory	Intensity	R–C	8.0 ± 0.3a	7.9 ± 0.4a	7.3 ± 0.8a	7.7 ± 0.3a	ns	ns	ns
		R-EX	8.3 ± 0.6a	7.3 ± 0.3a	7.4 ± 0.3a	7.9 ± 0.4a			
		r-C	7.1 ± 0.2b	7.2 ± 0.2ab	7.4 ± 0.5ab	7.2 ± 0.7ab	ns	*	*
		r-EX	6.9 ± 0.2b	7.1 ± 0.2ab	7.2 ± 0.3 ab	7.9 ± 1.1a			
	Persistency	R–C	7.6 ± 0.2ab	8.2 ± 0.2a	7.0 ± 0.5b	7.5 ± 0.5ab	ns	*	ns
		R-EX	7.6 ± 0.5ab	7.2 ± 0.4ab	7.1 ± 0.2b	7.1 ± 0.4b			
		r-C	6.2 ± 0.2c	6.9 ± 0.4bc	7.6 ± 0.3 ab	6.9 ± 0.2bc	ns	***	ns
		r-EX	6.3 ± 0.3c	7.1 ± 0.4bc	8.0 ± 0.5a	7.0 ± 0.5abc			
	Balance	R–C	7.6 ± 0.2ab	7.8 ± 0.2a	7.1 ± 0.2abc	6.4 ± 0.3c	ns	***	ns
		R-EX	7.4 ± 0.4ab	7.2 ± 0.5abc	7.2 ± 0.3abc	6.7 ± 0.3bc			
		r-C	7.7 ± 0.3a	7.3 ± 0.3ab	7.7 ± 0.3a	7.3 ± 0.3ab	ns	ns	**
		r-EX	6.6 ± 0.5b	6.8 ± 0.5ab	7.3 ± 0.3ab	7.9 ± 0.4a			
Gustatory	Intensity	R–C	7.8 ± 0.4a	7.6 ± 0.5abc	6.6 ± 0.3c	6.9 ± 0.5abc	ns	*	**
		R-EX	7.7 ± 0.3abc	6.7 ± 0.3bc	7.6 ± 0.3abc	7.7 ± 0.3 ab			
		r-C	7.0 ± 0.3ab	6.9 ± 0.4b	6.4 ± 0.1b	6.2 ± 0.3b	**	*	**
		r-EX	6.8 ± 0.4b	7.3 ± 0.3b	7.8 ± 0.3a	6.8 ± 0.3b			
	Persistency	R–C	7.7 ± 0.3a	7.3 ± 0.6 ab	6.3 ± 0.3c	6.4 ± 0.3bc	**	**	**
		R-EX	7.7 ± 0.1a	6.9 ± 0.2abc	7.8 ± 0.4a	7.2 ± 0.2abc			
		r-C	6.7 ± 0.3ab	7.1 ± 0.5ab	6.3 ± 0.3b	6.8 ± 0.3ab	*	*	*
		r-EX	6.3 ± 0.3b	7.1 ± 0.2ab	7.5 ± 0.5a	7.6 ± 0.3a			
	Balance	R–C	6.6 ± 0.2a	6.9 ± 0.2a	3.4 ± 0.3c	4.1 ± 0.2bc	ns	***	*
		R-EX	6.2 ± 0.4a	6.4 ± 0.4a	4.1 ± 0.5bc	4.6 ± 0.3b			
		r-C	6.6 ± 0.2a	6.2 ± 0.5a	4.2 ± 0.3c	4.7 ± 0.3bc	*	***	ns
		r-EX	5.7 ± 0.3ab	6.0 ± 0.3a	3.9 ± 0.6c	4.3 ± 0.3c			
	Tannicity	R–C	5.6 ± 0.4ab	6.0 ± 0.3a	3.7 ± 0.3c	5.4 ± 0.1 ab	ns	***	ns
		R-EX	5.3 ± 0.6ab	5.9 ± 0.4a	3.8 ± 0.3c	4.5 ± 0.5bc			
		r-C	3.6 ± 0.2d	3.9 ± 0.2cd	4.5 ± 0.5c	5.4 ± 1.0b	*	***	**
		r-EX	4.0 ± 0.3cd	3.8 ± 0.2cd	4.1 ± 0.2cd	6.4 ± 1.1a			
	Body	R–C	6.6 ± 0.4ab	6.9 ± 0.4a	3.1 ± 0.2d	5.7 ± 0.3bc	*	***	***
		R-EX	6.8 ± 0.5a	6.3 ± 0.3ab	4.9 ± 0.5c	6.6 ± 0.1ab			
		r-C	4.6 ± 0.2bcd	5.2 ± 0.2 ab	4.1 ± 0.4cd	3.7 ± 0.3d	***	***	**
		r-EX	4.7 ± 0.3bc	5.2 ± 0.4ab	5.6 ± 0.5a	4.6 ± 0.1bc			
Overall judgment		R–C	7.6 ± 0.2ab	7.8 ± 0.2a	6.5 ± 0.3c	6.4 ± 0.1c	ns	**	**
		R-EX	6.9 ± 0.5abc	7.3 ± 0.5abc	6.7 ± 0.3bc	7.4 ± 0.3abc			
		r-C	6.6 ± 0.2cd	6.8 ± 0.2bc	6.2 ± 0.3d	6.4 ± 0.1cd	***	***	***
		r-EX	6.1 ± 0.2d	6.7 ± 0.3bc	7.3 ± 0.3ab	7.6 ± 0.1a			

*Data followed by different letters indicate statistically significant differences at $p \leq 0.05$ according to two-way ANOVA with interaction followed by Tukey's test. Average value ± standard deviation (n = 3). Abbreviations: R–C, red control wine; R-EX, red wine with extract; r-C, rosé control wine; r-EX, rosé wine with extract. Tr, treatment variable; T, storage time variable; ns, not significant; *, **, ***, significant at $p \leq 0.05$, $p \leq 0.01$, $p \leq 0.001$, respectively.

3.5. Sensory profile of wines during storage

The results of sensory analysis of both wines were reported in Table 6. Two-way ANOVA results showed that, unlike the treatment and the wine, the storage time significantly influenced most of the considered descriptors. Particularly, significant difference ($p \leq 0.001$) was found for gustatory balance, tannicity and body that significant decreased after six months of storage. Moreover, only for rosé wines, storage time significantly increased color intensity, in accordance with the instrumental analysis although only for control wines, as well as viscosity, olfactory persistency. The treatment variables, instead, determined lower differences of the scores: at T6 the EX addition significantly improved the gustatory persistency, gustatory body, and color intensity, the latter only in rosé wines. As regards the overall judgment, storage time significantly influenced the scores of R–C and r-EX and the wines added with extract at T12 showed values significantly higher than control ones.

The olfactory profile was evaluated using the check-all-that-apply (CATA) approach, and the assessment of the selected aroma descriptors was performed via smell. Only the most frequently perceived descriptors by tasters (>3) were selected and represented by correspondence analysis. Fig. 4A and B shows the results of correspondence analysis applied on red and rosé wines, respectively. About red wine (Fig. 4A), the most frequently perceived descriptors in all samples and at each time considered were cherry, black cherry in

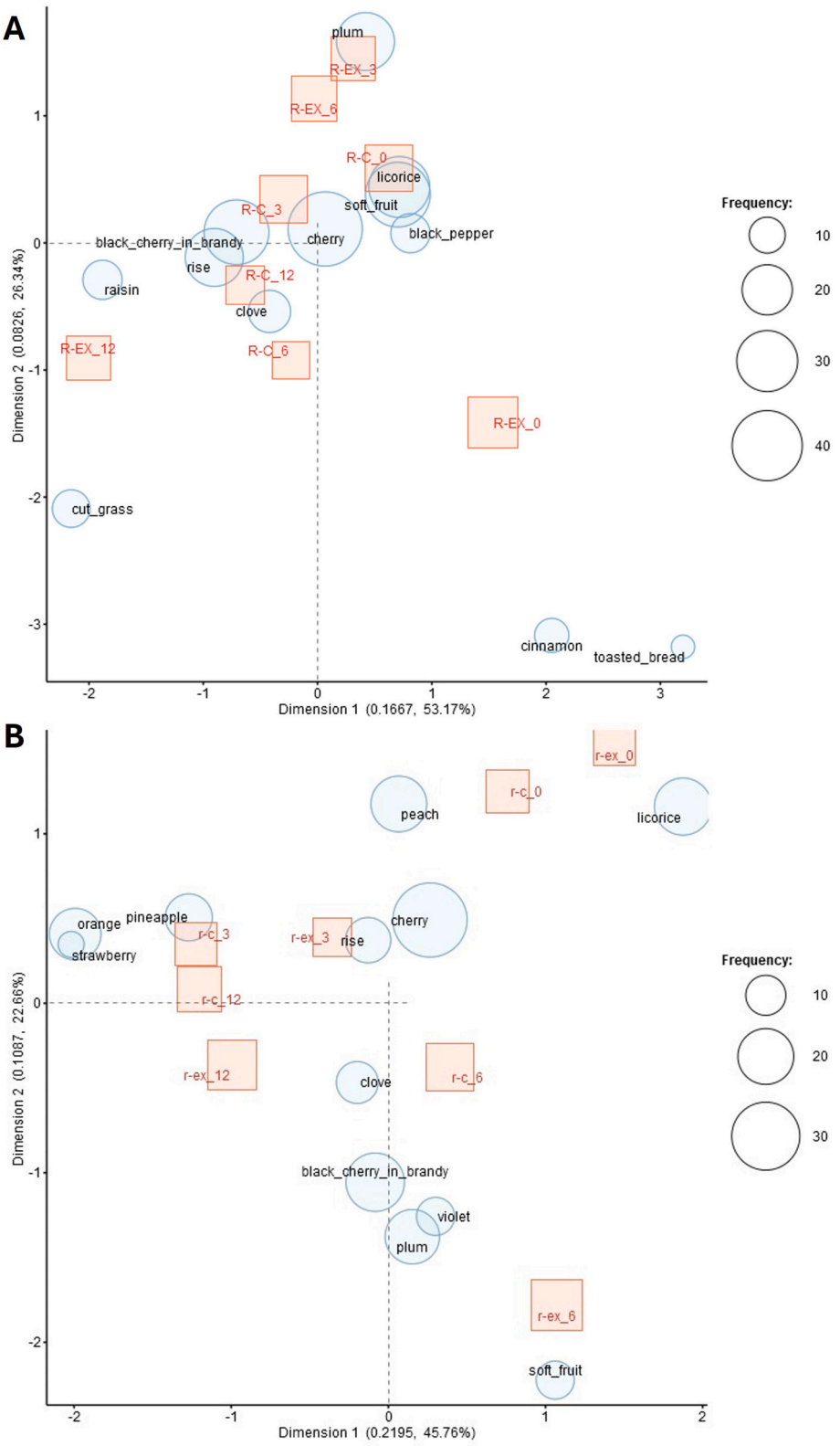


Fig. 4. Correspondence analysis of the highest frequency descriptors of red cv. Negramaro (**A**) and rosé cv. Sangiovese (**B**) wines. Abbreviations: R–C, red control wine; R-EX, red wine with extract; r-C, rosé control wine; r-EX, rosé wine with extract; 0, 3, 6, and 12, months of storage.

brandy, soft fruit, plum, rose and licorice. Specifically, the addition of the stilbene extract changed the sensory profile of the wine over time in agreement with what was reported by Raposo et al. [16]. In fact, in the wine R-EX specific notes were perceived: notes of toasted bread and cinnamon, immediately after bottling; up to 6 months of storage, notes of plum; notes of raisin and cut grass after 12 months of storage.

Regarding rosé wine, Fig. 4B showed that plum, cherry, orange, licorice, and black cherry in brandy were the most frequently perceived descriptors in all samples and at each time considered. In particular, the addition of the extract resulted in licorice notes particularly perceived immediately after bottling. After 3 months of storage, notes of rose and cherry particularly characterized the wines r-EX which was poorer than fruity notes (orange, pineapple, and strawberry) respect to r-C. The wines after 6 months of storage were characterized by notes of black cherry in brandy, plum, violet and soft fruit (particularly in r-EX). After 12 months, no particular difference was found between the two wines.

4. Conclusion

The results obtained show that the addition of the vine-shoot extract in red and rosé wines led for partial or total replacement of SO_2 can be considered a strategy for the reduction of this allergen. However, the effect of the extract was partially shifted compared to SO_2. In fact, the vine-shoot extract granted an increased antioxidant activity, that was exerted towards carbonyl formation rather than towards anthocyanin degradation. The more rapid anthocyanin decrease was partially compensated, in rosé wine, by the lower decoloring effect related of SO_2, present at lower levels. A possible increase of tannin polymerization and anthocyanin condensation should be confirmed by further studies. On the other hand, the increased levels of ethyl-phenol suggest a lower level of antimicrobial protection compared to SO_2 alone. Finally, the sensory analysis showed that the addition of the extract improved the sensory profile of both wines, which were found to be the most liked by tasters after 12 months of storage.

Therefore, this extract could potentially be used in combination with reduced doses of SO_2 in wines. Such use would not only reduce the problems associated with the use of SO_2, but would also represent a possible sustainable alternative use of a wine waste. However, further studies should be carried out to evaluate different doses and proportions with SO_2, addition at different stages of the winemaking process and the possible microbiological changes it could cause in wines.

Ethical statement

Participants 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.

Data availability

Data will be made available on request.

CRediT authorship contribution statement

Mirella Noviello: Writing – original draft, Visualization, Methodology, Investigation. **Claudia Antonino:** Writing – original draft, Visualization, Methodology, Investigation. **Giuseppe Gambacorta:** Writing – review & editing. **Vito Michele Paradiso:** Writing – original draft, Visualization, Supervision, Methodology, Conceptualization. **Francesco Caponio:** Writing – original draft, Visualization, Supervision, Methodology, Conceptualization.

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.

Acknowledgements

This research was funded by the Ministero dell’Istruzione, dell’Università e della Ricerca, programmi di ricerca 2017 (2017JTNK78) and “GOOD-BY-WASTE. Obtain GOOD products-exploit BY-products-reduce WASTE” (CUP H98D19000940006).

The authors grateful thank wineries Azienda Agricola Conti Zecca (Leverano, Puglia, Italy) and Divella s.r.l. (Santeramo in Colle, Puglia, Italy).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.heliyon.2024.e34310.

References

[1] S. Giacosa, S. Río Segade, E. Cagnasso, A. Caudana, L. Rolle, V. Gerbi, SO2 in wines, in: Red Wine Technol, Elsevier, 2019, pp. 309–321, https://doi.org/10.1016/B978-0-12-814399-5.00021-9.

[2] M.T. Lisanti, G. Blaiotta, C. Nioi, L. Moio, Alternative methods to SO 2 for microbiological stabilization of wine, Compr. Rev. Food Sci. Food Saf. 18 (2019) 455–479, https://doi.org/10.1111/1541-4337.12422.

[3] W.J. du Toit, J. Marais, I.S. Pretorius, M. du Toit, Substrates for oxidation in wine, S. Afr. J. Enol. Vitic. 27 (2006) 76–94.

[4] A. Capece, R. Pietrafesa, G. Siesto, P. Romano, Biotechnological approach based on selected Saccharomyces cerevisiae Starters for reducing the use of sulfur dioxide in wine, Microorganisms 8 (2020) 738, https://doi.org/10.3390/microorganisms8050738.

[5] P. Ribéreau-Gayon, D. Dubourdieu, B. Donèche, A. Lonvaud, Handbook of Enology, ume 1, The microbiology of wine and vinifications, 2018.

[6] R. Vecchio, E. Parga-Dans, P. Alonso González, A. Annunziata, Why consumers drink natural wine? Consumer perception and information about natural wine, Agric. Food Econ. 9 (2021) 22, https://doi.org/10.1186/s40100-021-00197-1.

[7] S. Giacosa, S. Río Segade, E. Cagnasso, A. Caudana, L. Rolle, V. Gerbi, SO2 in Wines: Rational Use and Possible Alternatives, Red Wine Technol, 2018, pp. 309–321, https://doi.org/10.1016/B978-0-12-814399-5.00021-9.

[8] F.V.M. Silva, S. van Wyk, Emerging non-thermal technologies as alternative to SO2 for the production of wine, Foods 10 (2021) 2175, https://doi.org/10.3390/foods10092175.

[9] M. Lebleux, H. Alexandre, R. Romanet, J. Ballester, V. David-Vaizant, M. Adrian, R. Tourdot-Maréchal, C. Rouiller-Gall, Must protection, sulfites versus bioprotection: a metabolomic study, Food Res. Int. 173 (2023), https://doi.org/10.1016/j.foodres.2023.113383.

[10] E. Kontaxakis, E. Trantas, F. Ververidis, Resveratrol: a fair race towards replacing sulfites in wines, Molecules 25 (2020) 2378, https://doi.org/10.3390/molecules25102378.

[11] A. Valletta, L.M. Iozia, F. Leonelli, Impact of environmental factors on stilbene biosynthesis, Plants 10 (2021) 90, https://doi.org/10.3390/plants10010090.

[12] I. Dávila, E. Robles, I. Egüés, J. Labidi, P. Gullón, The biorefinery concept for the industrial valorization of grape processing by-products, in: Handb. Grape Process. By-Products, Elsevier, 2017, pp. 29–53, https://doi.org/10.1016/B978-0-12-809870-7.00002-8.

[13] M. Troilo, G. Difonzo, V.M. Paradiso, C. Summo, Bioactive compounds from vine shoots , grape stalks , and wine lees : their potential use in agro-food chains, 1–16, https://doi.org/10.3390/foods10020342, 2021.

[14] R.F. Guerrero, M.J. Aliaño-González, B. Puertas, T. Richard, E. Cantos-villar, Industrial Crops & Products Comparative analysis of stilbene concentration in grapevine shoots of thirteen Vitis during a three-year study, Ind. Crop. Prod. 156 (2020) 112852, https://doi.org/10.1016/j.indcrop.2020.112852.

[15] M. Noviello, A.F. Caputi, G. Squeo, V.M. Paradiso, G. Gambacorta, F. Caponio, Vine shoots as a source of trans-resveratrol and ε-viniferin: a study of 23 Italian varieties, Foods 11 (2022) 553, https://doi.org/10.3390/foods11040553.

[16] R. Raposo, M.J. Ruiz-moreno, T. Garde-cerdán, B. Puertas, J.M. Moreno-rojas, A. Gonzalo-diago, R. Guerrero, V. Ortíz, E. Cantos-villar, Grapevine-shoot stilbene extract as a preservative in red wine, Food Chem. 197 (2016) 1102–1111, https://doi.org/10.1016/j.foodchem.2015.11.102.

[17] S. Cruz, R. Raposo, M.J. Ruiz-moreno, T. Garde-cerdán, B. Puertas, Grapevine-shoot stilbene extract as a preservative in white wine, Food Packag, Shelf Life 18 (2018) 164–172, https://doi.org/10.1016/j.fpsl.2018.10.008.

[18] R. Gutiérrez-Escobar, M.J. Aliaño-González, I. Le Mao, A. Marrufo-Curtido, M. Carbú, M.J. Jiménez-Hierro, B. Puertas, T. Richard, E. Cantos-Villar, Grapevine shoots extract as an alternative to SO2 in rosé wines. A double approach: classical measurements and 1H NMR metabolomics, Food Control 152 (2023) 109861, https://doi.org/10.1016/j.foodcont.2023.109861.

[19] B. Houillé, S. Besseau, V. Courdavault, A. Oudin, G. Glévarec, G. Delanoue, L. Guérin, A.J. Simkin, N. Papon, M. Clastre, N. Giglioli-Guivarc'h, A. Lanoue, Biosynthetic origin of E -resveratrol accumulation in grape canes during postharvest storage, J. Agric. Food Chem. 63 (2015) 1631–1638, https://doi.org/10.1021/jf505316a.

[20] A. Torreggiani, C. Demarinis, D. Pinto, A. Papale, G. Difonzo, F. Caponio, E. Pontonio, M. Verni, C.G. Rizzello, Up-cycling grape pomace through sourdough fermentation: characterization of phenolic compounds, antioxidant activity, and anti-inflammatory potential, Antioxidants 12 (2023) 1521, https://doi.org/10.3390/antiox12081521.

[21] A. Pasqualone, R. Punzi, A. Trani, C. Summo, V.M. Paradiso, F. Caponio, G. Gambacorta, Enrichment of fresh pasta with antioxidant extracts obtained from artichoke canning by-products by ultrasound-assisted technology and quality characterisation of the end product, Int. J. Food Sci. Technol. 52 (2017) 2078–2087, https://doi.org/10.1111/ijfs.13486.

[22] F.Z. Makhlouf, G. Squeo, M. Barkat, A. Trani, F. Caponio, Antioxidant activity, tocopherols and polyphenols of acornoil obtained from Quercus species grown in Algeria, Food Res. Int. 114 (2018) 208–213, https://doi.org/10.1016/j.foodres.2018.08.010.

[23] P. Goufo, R.K. Singh, I. Cortez, A reference list of phenolic compounds (including stilbenes) in grapevine (Vitis vinifera L.) roots, woods, canes, stems, and leaves, Antioxidants 9 (2020) 398, https://doi.org/10.3390/antiox9050398.

[24] G. Difonzo, A. Aresta, P. Cotugno, R. Ragni, G. Squeo, C. Summo, F. Massari, A. Pasqualone, M. Faccia, C. Zambonin, F. Caponio, Supercritical co2 extraction of phytocompounds from olive pomace subjected to different drying methods, Molecules 26 (2021), https://doi.org/10.3390/molecules26030598.

[25] G. Gambacorta, D. Antonacci, M. La Gatta, M. Faccia, B. La Gatta, S. Pati, A. Coletta, E. La Notte, Phenolic composition of Aglianico and Nero di Troia grapes and wines as affected by cover cropping and irrigation, Ital, J. Food Sci. 23 (2011) 381–394.

[26] A. Tarantino, G. Difonzo, G. Lopriore, G. Disciglio, V.M. Paradiso, G. Gambacorta, F. Caponio, Bioactive compounds and quality evaluation of 'Wonderful' pomegranate fruit and juice as affected by deficit irrigation, J. Sci. Food Agric. 100 (2020) 5539–5545, https://doi.org/10.1002/jsfa.10606.

[27] Y. Glories, Il colore del vino rosso I parte, Connaiss. Vigne Vin (1984) 195–217.

[28] M. Noviello, V.M. Paradiso, G. Natrella, G. Gambacorta, M. Faccia, F. Caponio, Application of toasted vine-shoot chips and ultrasound treatment in the ageing of Primitivo wine, Ultrason. Sonochem. 104 (2024) 106826, https://doi.org/10.1016/j.ultsonch.2024.106826.

[29] J. Pérez-Navarro, P.M. Izquierdo-Cañas, A. Mena-Morales, J. Martínez-Gascueña, J.L. Chacón-Vozmediano, E. García-Romero, I. Hermosín-Gutiérrez, S. Gómez-Alonso, Phenolic compounds profile of different berry parts from novel Vitis vinifera L. red grape genotypes and Tempranillo using HPLC-DAD-ESI-MS/MS: a varietal differentiation tool, Food Chem. 295 (2019) 350–360, https://doi.org/10.1016/j.foodchem.2019.05.137.

[30] T. Püssa, J. Floren, P. Kuldkepp, A. Raal, Survey of grapevine Vitis vinifera stem polyphenols by liquid Chromatography−Diode array Detection−Tandem mass spectrometry, J. Agric. Food Chem. 54 (2006) 7488–7494, https://doi.org/10.1021/jf061155e.

[31] A. Stój, I. Kapusta, D. Domagała, Classification of red wines produced from zweigelt and rondo grape varieties based on the analysis of phenolic compounds by UPLC-PDA-MS/MS, Molecules 25 (2020) 1342, https://doi.org/10.3390/molecules25061342.

[32] A.M. Timperio, A. D'Alessandro, M. Fagioni, P. Magro, L. Zolla, Production of the phytoalexins trans-resveratrol and delta-viniferin in two economy-relevant grape cultivars upon infection with Botrytis cinerea in field conditions, Plant Physiol. Biochem. 50 (2012) 65–71, https://doi.org/10.1016/j.plaphy.2011.07.008.

[33] D. Topi, H. Kelebek, G. Guclu, S. Selli, LC-DAD-ESI-MS/MS characterization of phenolic compounds in wines from Vitis vinifera ' Shesh i bardhë ' and ' Vlosh ' cultivars, J. Food Process. Preserv. 46 (2022) 1–14, https://doi.org/10.1111/jfpp.16157.

[34] X.-K. Zhang, S.-Y. Li, X. Zhao, Q.-H. Pan, Y. Shi, C.-Q. Duan, HPLC-MS/MS-based targeted metabolomic method for profiling of malvidin derivatives in dry red wines, Food Res. Int. 134 (2020) 109226, https://doi.org/10.1016/j.foodres.2020.109226.

[35] A. Wojdyło, J. Samoticha, J. Chmielewska, The influence of different strains of Oenococcus oeni malolactic bacteria on profile of organic acids and phenolic compounds of red wine cultivars Rondo and Regent growing in a cold region, J. Food Sci. 85 (2020) 1070–1081, https://doi.org/10.1111/1750-3841.15061.

[36] I. Prezioso, G. Fioschi, L. Rustioni, M. Mascellani, G. Natrella, P. Venerito, G. Gambacorta, V.M. Paradiso, Influence of prolonged maceration on phenolic compounds, volatile profile and sensory properties of wines from Minutolo and Verdeca, two Apulian white grape varieties, Lwt 192 (2024) 115698, https://doi.org/10.1016/j.lwt.2023.115698.

[37] A. Trani, V. Verrastro, R. Punzi, M. Faccia, G. Gambacorta, Phenols, volatiles and sensory properties of primitivo wines from the "Gioia Del Colle" PDO area, S. Afr. J. Enol. Vitic. 37 (2016) 139–148, https://doi.org/10.21548/37-2-1002.

[38] V.M. Paradiso, L. Sanarica, I. Zara, C. Pisarra, G. Gambacorta, G. Natrella, M. Cardinale, 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 (2022), https://doi.org/10.3390/foods11213373.
[39] Y. Bai, P. Zhao, Y. Du, J. Lin, F. Han, Effect of postharvest grape dehydration on the phenolic composition of 'Marselan' rose wine during aging, J. Food Compos. Anal. 123 (2023) 105630, https://doi.org/10.1016/j.jfca.2023.105630.
[40] R. Raposo, F. Chinnici, M.J. Ruiz-moreno, B. Puertas, F.J. Cuevas, M. Carbú, R.F. Guerrero, V. Ortíz-somovilla, J.M. Moreno-rojas, E. Cantos-villar, Sulfur free red wines through the use of grapevine shoots : impact on the wine quality, Food Chem. 243 (2018) 453–460, https://doi.org/10.1016/j.foodchem.2017.09.111.
[41] F. He, N.-N. Liang, L. Mu, Q.-H. Pan, J. Wang, M.J. Reeves, C.-Q. Duan, Anthocyanins and their variation in red wines I. Monomeric anthocyanins and their color expression, Molecules 17 (2012) 1571–1601, https://doi.org/10.3390/molecules17021571.
[42] F. He, N.-N. Liang, L. Mu, Q.-H. Pan, J. Wang, M.J. Reeves, C.-Q. Duan, Anthocyanins and their variation in red wines II. Anthocyanin derived pigments and their color evolution, Molecules 17 (2012) 1483–1519, https://doi.org/10.3390/molecules17021483.
[43] R. Gutiérrez-Escobar, M.J. Aliaño-González, E. Cantos-Villar, Wine polyphenol content and its influence on wine quality and properties: a review, Molecules 26 (2021) 718, https://doi.org/10.3390/molecules26030718.
[44] J. Lachman, M. Šulc, M. Schilla, Comparison of the total antioxidant status of Bohemian wines during the wine-making process, Food Chem. 103 (2007) 802–807, https://doi.org/10.1016/j.foodchem.2006.09.024.
[45] A. Floegel, D.O. Kim, S.J. Chung, S.I. Koo, O.K. Chun, Comparison of ABTS/DPPH assays to measure antioxidant capacity in popular antioxidant-rich US foods, J. Food Compos. Anal. 24 (2011) 1043–1048, https://doi.org/10.1016/j.jfca.2011.01.008.
[46] Y.C. Lou, P. Jing, DFT study of the stabilization effect on anthocyanins via secondary interactions, Food Chem. Mol. Sci. 3 (2021) 100057, https://doi.org/10.1016/j.fochms.2021.100057.
[47] M.T. Lisanti, R. Capuano, L. Moio, A. Gambuti, Wood powders of different botanical origin as an alternative to barrel aging for red wine, Eur. Food Res. Technol. 247 (2021) 2309–2320, https://doi.org/10.1007/s00217-021-03791-1.
[48] I. Benucci, Impact of post-bottling storage conditions on colour and sensory profile of a rosé sparkling wine, LWT 118 (2020) 108732, https://doi.org/10.1016/j.lwt.2019.108732.
[49] G. Gambacorta, M. Faccia, G. Natrella, M. Noviello, G. Masi, L. Tarricone, Early basal leaf removal at different sides of the canopy improves the quality of aglianico wine, Foods 11 (2022) 3140, https://doi.org/10.3390/foods11193140.
[50] E. Pelonnier-Magimel, K. Chira, P.L. Teissèdre, M. Jourdes, J.C. Barbe, Color characterization of bordeaux red wines produced without added sulfites, Foods 12 (2023), https://doi.org/10.3390/foods12122358.
[51] A.L. Waterhouse, J. Zhu, A quarter century of wine pigment discovery, J. Sci. Food Agric. 100 (2020) 5093–5101, https://doi.org/10.1002/jsfa.9840.
[52] X. Zhao, F. He, X.K. Zhang, Y. Shi, C.Q. Duan, Impact of three phenolic copigments on the stability and color evolution of five basic anthocyanins in model wine systems, Food Chem. 375 (2022) 131670, https://doi.org/10.1016/j.foodchem.2021.131670.
[53] R.S. Jackson, Wine Science: Principles and Applications, 2008.
[54] M. Tufariello, S. Capone, P. Siciliano, Volatile components of Negroamaro red wines produced in Apulian Salento area, Food Chem. 132 (2012) 2155–2164, https://doi.org/10.1016/j.foodchem.2011.11.122.
[55] W. Cao, N. Shu, J. Wen, Y. Yang, Y. Jin, W. Lu, Characterization of the key aroma volatile compounds in nine different grape varieties wine by headspace gas chromatography–ion mobility spectrometry (HS-GC-IMS), odor activity values (OAV) and sensory analysis, Foods 11 (2022) 2767, https://doi.org/10.3390/foods11182767.
[56] P. Piombino, L. Moio, A. Genovese, Orthonasal vs. retronasal: studying how volatiles' hydrophobicity and matrix composition modulate the release of wine odorants in simulated conditions, Food Res. Int. 116 (2019) 548–558, https://doi.org/10.1016/j.foodres.2018.08.072.
[57] J. Liu, W. Zhao, S. Li, A. Zhang, Y. Zhang, S. Liu, Characterization of the key aroma compounds in proso millet wine using headspace solid-phase microextraction and gas chromatography-mass spectrometry, Molecules 23 (2018) 462, https://doi.org/10.3390/molecules23020462.
[58] J. Ruiz, F. Kiene, I. Belda, D. Fracassetti, D. Marquina, E. Navascués, F. Calderón, A. Benito, D. Rauhut, A. Santos, S. Benito, Effects on varietal aromas during wine making: a review of the impact of varietal aromas on the flavor of wine, Appl. Microbiol. Biotechnol. 103 (2019) 7425–7450, https://doi.org/10.1007/s00253-019-10008-9.
[59] R. Cucciniello, M. Forino, L. Picariello, F. Coppola, L. Moio, A. Gambuti, How acetaldehyde reacts with low molecular weight phenolics in white and red wines, Eur. Food Res. Technol. 247 (2021) 2935–2944, https://doi.org/10.1007/s00217-021-03841-8.
[60] A. Baiano, A. Mentana, M. Quinto, D. Centonze, M.A. Previtali, G. Varva, M.A. Del Nobile, L. De Palma, Volatile composition and sensory profile of wines obtained from partially defoliated vines: the case of Nero di Troia wine, Eur. Food Res. Technol. 243 (2017) 247–261, https://doi.org/10.1007/s00217-016-2740-y.
[61] D. Moreno, C. Falguères, A. Pérez-González, M. Duval, P. Voinchet, A. Benito-Calvo, A.I. Ortega, J.J. Bahain, R. Sala, E. Carbonell, J.M. Bermúdez de Castro, J. L. Arsuaga, ESR chronology of alluvial deposits in the arlanzón valley (atapuerca, Spain): contemporaneity with atapuerca gran dolina site, Quat. Geochronol. 10 (2012) 418–423, https://doi.org/10.1016/j.quageo.2012.04.018.
[62] Y. Yu, L. Li, R. Xue, C. Wang, M. Chen, J. Ramos, S. Zhang, B. Sun, Impact of different oak chips' aging on the volatile compounds and sensory characteristics of vitis amurensis wines, Foods 11 (2022) 1126, https://doi.org/10.3390/foods11081126.
[63] R. Perestrelo, C. Silva, J.S. Câmara, Madeira wine volatile profile. A platform to establish madeira wine aroma descriptors, Molecules 24 (2019) 3028, https://doi.org/10.3390/molecules24173028.
[64] D. Skroza, I. Generalić Mekinić, S. Svilović, V. Šimat, V. Katalinić, Investigation of the potential synergistic effect of resveratrol with other phenolic compounds: a case of binary phenolic mixtures, J. Food Compos. Anal. 38 (2015) 13–18, https://doi.org/10.1016/j.jfca.2014.06.013.