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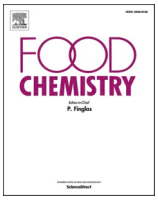

Cold liquid stabulation: Impact on the phenolic, antioxidant, and aroma characteristics of wines from aroma-neutral white grape varieties

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ABSTRACT

Cold liquid stabulation aims to extract valuable compounds from grape lees before juice clarification. In this study, 7, 14, and 21 days of lees contact were tested on aroma-neutral 'Arneis' and 'Cortese' grape juices vs control. Basic parameters, colour, polyphenols, antioxidant capacity, and volatile organic compounds were assessed throughout winemaking. Wine sensory analysis was performed. The produced wines did not differ in terms of colour and show limited differences in polyphenols, not influencing astringency and bitterness. Variety and treatment length influenced free and glycosylated volatile organic compounds. Free terpenes increased in the 21-day treated 'Arneis' wine (+67 %). Lower free esters in 'Arneis' with 14 days of stabulation were found (−10 %). On the contrary, higher values of individual esters were found in 14 and 21-day treated 'Cortese' wines, but these showed lower free C6 (−12 %) and sulphur compounds (−23 % and − 24 %, respectively), and higher overall wine quality with respect to non-stabulated wine.

1. Introduction

Wine primary and secondary metabolites are affected by climate, region, grape variety, ripeness, viticultural and oenological practices (Rienth et al., 2021). Different winemaking techniques help to improve the wine quality modifying the quantity and composition of these compounds (Selli, Canbas, Cabaroglu, Erten and Günata, 2006a).

In white wine production, pre-fermentative maceration on skins, under controlled conditions, low temperature as cold maceration, and usually with the help of exogenous enzymes, improves the extraction of volatile organic compounds (VOCs), as well as some hydrosoluble phenolics (Aleixandre-Tudo et al., 2015; Bestulić et al., 2022; Malićanin et al., 2022; Wang et al., 2016). This aspect results in increased floral and fruity attributes, and in more balanced, round, and full-bodied wines. Alternatively, wine aging in presence of fine lees, produced after alcoholic fermentation, has a significant effect on wine mouthfeel, due to lees being rich in tartaric salts, amino acids, fatty acids, vitamins, and compounds released through yeast autolysis such as mannoproteins, β-glucans, and lipids (Fornairon-Bonnefond et al., 2002).

However, the potentialities of the grape solids corresponding to the grape flesh (*bourbes*) are still scarcely investigated. Usually, this fraction is removed with the juice clarification treatments (e.g. cold settling), applied after pressing and before alcoholic fermentation, and discarded as a winemaking by-product. Only a minor part is kept into juices to increase their lipidic content for yeast growth during fermentation (Casalta, Cervi, Salmon and Sablayrolles, 2013; Guittin et al., 2021). This solid residue is composed mainly of polysaccharides (70 %), lipids (8 %), and in minor part of minerals, pectin, and nitrogen compounds (2.5 %), and lastly of phenolic compounds (Alexandre et al., 1994). Nevertheless, this solid part can influence the wine production process, through a technique called *maceration sur bourbes* or cold liquid stabulation (CLS). In brief, CLS consists, after grape pressing, in maintaining the grape juice on its lees, kept in suspended condition, at a low temperature (0–8 °C) for a variable period (2–26 days). The expected effect of this technique is: *i)* a higher extraction of substances from flesh particles during must-lees contact, mainly VOC precursors such as terpenes, norisoprenoids, and thiols (Philipp et al., 2022; Philipp et al., 2024), and *ii)* an increased content of nutrients for the fermentation development.

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The presence of lipids and nitrogen compounds can impact the fermentation progress, being also a source of fermentative VOCs (Casalta et al., 2013; Guittin et al., 2021), whereas polysaccharides may influence the mouthfeel, as well as phenolic compounds could in turn affect astringency, bitterness, and colour of the produced wines (Hornedo-Ortega et al., 2020). Nevertheless, grape solids may enhance herbaceous notes by increasing the presence of C6 alcohols and aldehydes extracted from skins, or increasing enzymes activities such as polyphenoloxidase or esterase, as well as unwanted microbiological contamination and the risk of pesticide residues into wines due to prolonged skin contact (Casalta et al., 2013).

Few studies on the use of CLS and its impact on finished wines in terms of basic, phenolic, and aroma characteristics are available, showing that in some aromatic varieties, i.e. 'Traminer' or 'Sauvignon blanc', a CLS of 7 days can increase significantly the free monoterpene concentration when compared to a non-stabulated control, or can have a greater impact on the production of thiols, such as 3-mercaptohexanol (3MH). (Philipp et al., 2022; Philipp et al., 2024). Cravero et al. (2012) showed that CLS application on the Italian autochthonous cultivar 'Bombino bianco' resulted in wine with higher golden yellow hue with respect to the control. Among the available studies, to our knowledge no information on the effect of a CLS length greater than 7 days has been published.

The aim of this study was to evaluate the changes in the volatile composition of stabulated grape juices and wines obtained with different CLS lengths, namely 7, 14 and 21 days, and compared to a non-stabulated control, as well as the influence of these changes on sensory profile of resulting wines. At the same time, the assessment of phenolic, colour and the antioxidant characteristics was done. CLS was applied in the winemaking of two *Vitis vinifera* L. varieties, 'Cortese' and 'Arneis'. These varieties are used to produce relevant volumes of white wine in the northern Italy landscape and are involved in several monovarietal Protected Designation of Origin (PDO) wines, mostly from the Piemonte region (Carlin et al., 2022). These two grape varieties are classified neutral in terms of VOCs (Piano et al., 2014; Piergiovanni et al., 2023), therefore an increase in their aroma precursors from the grape and an improvement in fermentative aroma could strongly influence the final wine sensory characteristics.

2. Material and methods

2.1. Winemaking

Approximately 300 kg of grapes *Vitis vinifera* L. cv. 'Arneis' (total soluble solids 23.4 ± 0.3°Brix; total acidity as g/L of tartaric acid 5.9 ± 0.1, pH 3.20 ± 0.02) and 300 kg of grape 'Cortese' (total soluble solids 22.0 ± 0.3°Brix; total acidity as g/L of tartaric acid 5.1 ± 0.1, pH 3.30 ± 0.01) were hand-harvested on the 25th of August and 22nd of September 2022, respectively. Once arrived in the experimental cellar of University of Torino '*Bonafous*' in Chieri (Italy), the intact grapes were stored into a thermo-controlled room at 0 °C for 12 h. The grapes were then destemmed and crushed in a TEMA destemmer–crusher (Enoveneta, Piazzola Sul Brenta, Italy) and pressed using a PMA 4 pneumatic press (Velo SpA, Altivole, Italy), with a pressure program consisting in three cycles with growing pressure (0.6, 0.8, and 1 bar, respectively, for a total of 15 min of pressing for each variety). On the obtained juice, 50 mg/L of SO_2 (potassium metabisulphite, Alea Evolution S.R.L., Molinella, Italy) and 2 g/hL of pectolytic enzyme (Lallzyme cuvée blanc, Lallemand Inc., Montreal, Canada) were added. The enzymatic preparation contains polygalacturonase (≥ 13,000 U/g) and β-glucosidase (≥ 12 U/g) activities, as reported by the manufacturer. The resulting juice was then divided in twelve 15-L glass canisters for each grape variety. Four process lengths were tested in triplicate: 0 (control, no CLS), 7, 14, and 21 days. The control samples, after pressing, undergo a cold static clarification process for 24 h at 0 °C in a temperature-controlled room. Afterwards, they were racked and inoculated for the alcoholic fermentation. The treated samples, instead, during the whole period of CLS were kept in a controlled room at 4 °C with the lees manually suspended twice a day by using a food-grade plastic stirrer. At the end of CLS, a 24-h cold static clarification, in a controlled room at 0 °C, was carried out before racking. The same turbidity value was reached, expressed as nephelometric turbidity units (NTU), for all tests (target of 220 NTU) by adding their respective lees. For the alcoholic fermentation, *Saccharomyces cerevisiae* active dry yeast (Fermol Chardonnay, AEB Group, Brescia, Italy) at 20 g/hL dose were added, following the rehydration procedure from the manufacturer instructions, standardized for all the treatments. Two additions of diammonium phosphate (Agrovin, Ciudad Real, Spain) were done: 50 g/hL were added at the beginning of alcoholic fermentation, and 25 g/hL at one-third of the fermentation. The fermentation was kept at a controlled temperature (18 ± 1 °C), with a daily monitoring of the sugar consumption. At the end of the fermentation, each sample was racked to remove lees and 50 mg/L of SO_2 were added. One month later, the samples were cold stabilised for two weeks in a controlled room at 0 °C and then bottled.

During the experimental procedure, samples were obtained after pressing (juice), after cold liquid stabulation (PS), after alcoholic fermentation (PAF), and one month after bottling (PWI). For each stage, the samples obtained following 7, 14, and 21 days of CLS are indicated as AR07, AR14, AR21 for 'Arneis' and as CO07, CO14, and CO21 for 'Cortese', respectively.

2.2. Chemicals and standards

All chemicals of analytical reagent grade, gallic acid, Folin-Ciocalteu reagent, (−)-epicatechin 92.0 %, gallic acid monohydrate 99.0 %, 2,2-diphenyl-1-picrylhydrazyl (DPPH), 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid 97.0 % (Trolox), disodium phosphate, and HPLC-gradient grade solvents were supplied by Sigma-Aldrich (St Louis, MO, USA). For HPLC analysis, glucose, fructose, and malic, tartaric, citric, succinic, and acetic acids, ethanol and glycerol (purity >98.0 %) and lactic acid (purity 90.0 %) were purchased from VWR International (Milan, Italy). Deionized water was produced by a Milli-Q system (Merck Millipore, Darmstadt, Germany). For the analysis of VOCs, ethanol 99.8 %, 1-heptanol 98.0 %, 2-octanol 98 %, HPLC-grade methanol (MeOH) 99.9 %, anhydrous sodium sulphate 99.0 %, and anhydrous sodium phosphate dibasic 99.0 % were purchased from Sigma-Aldrich. Dichloromethane and citric acid 99.5 % were obtained from Carlo Erba (Rodano, MI, Italy).

2.3. Physical-chemical analysis of 'Arneis' and 'Cortese' grapes, musts, and wines

Samples were collected at different times: grape samples, juice after pressing, after cold liquid stabulation (PS), after alcoholic fermentation (PAF), and one month after bottling (PWI).

2.3.1. Basic parameters

Total soluble solids content (°Brix) was analyzed through a refractometer Atago palette 0–32°Brix with automatic temperature compensation (Atago Corporation, Tokyo, Japan). The pH was measured by potentiometry using an Inolab 730 calibrated pHmeter (WTW, Weilheim, Germany) according to the OIV-MA-AS313–15 method (OIV, 2016). The total acidity was determined by titrimetry following the OIV-MA-AS313–01 method (OIV, 2016). The organic acids, i.e. malic, tartaric, lactic, citric, succinic, and acetic acid, ethanol and glycerol were quantified by HPLC (Agilent 1260, Agilent Technologies, Santa Clara, USA) with a UV detector set to 210 nm and a refractive index detector (Giordano et al., 2009). Turbidity was determined using a turbidimeter (TB1, Velp Scientifica, Usmate, Italy) following the OIV-MA-AS2–08 method and expressed as NTU.

2.3.2. Spectrophometric measurements

The phenolic composition and colour parameters were evaluated using a UV-1800 spectrophotometer (Shimazdu Corporation, Kyoto, Japan). Total phenolic index (TPI) was determined by measuring absorbance at 280 nm of the sample diluted (1:10) in deionized water, and expressed in mg/L of (−)-epicatechin by an external calibration curve (Scalzini et al., 2020).

The CIELab parameters were evaluated according to the OIV-MA-AS2–11 method, namely lightness (L^*), red/green (a^*), and yellow/blue (b^*) colour coordinates (OIV, 2016). The sample spectrum in the region 370–700 nm was recorded using 10 mm pathway plastic cuvette, and the CIELab values were calculated as reported in OIV-MA-AS2–11 (OIV, 2016). These values were converted to RGB values for visualization purposes. The total colour difference (ΔE*) between one CLS-treated sample and the respective control was calculated as follows: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ (OIV, 2016). Total sulphur dioxide (total SO_2) was determined using an Hyperlab Smart automatic analyser (Steroglass, San Marino in Campo, Italy) through a colorimetric method based on the reaction between total sulphur dioxide and a disulfide chromogenic compound with an absorption maximum at 416 nm (Total SO_2 Kit, SQPE060413, Steroglass). With the same instrument, through enzymatic analysis, acetaldehyde was determined (Acetaldehyde Kit, SQPE059576, Steroglass). The antioxidant potential was investigated on the samples (either juice or wine) diluted 1:10 with deionized water, following the Brand-Williams et al. (1995) method, modified by Romanet et al. (2019). Briefly, 100 μL of diluted sample were added with 2 mL of DPPH solution (25 mg/L) prepared daily. The DPPH radical was dissolved in a solution composed of methanol and buffer (60:40 *v*/v). The buffer solution contained 0.1 M citric acid and 0.2 M disodium phosphate, and the pH value was adjusted to 3.6. The absorbance of the samples and the reaction blanks (prepared by replacing the sample with water) was measured at 515 nm absorbance after 240 min. The results (sample absorbance – blank absorbance) were converted as mmol Trolox equivalents/L using a Trolox-based calibration curve.

On wine (PWI samples), the total phenolic content was determined additionally by the Folin-Ciocalteu method (FC) after purification on 1-g Sep-Pak C_{18} solid phase extraction cartridge (Waters Corporation, Milford, MA, USA). After 70 min of reaction, the resulting absorbance was measured at 750 nm and the results were expressed as mg/L of gallic acid through an external calibration curve (OIV-MA-AS2–10 method; OIV, 2016; Scalzini et al., 2020). Total polysaccharides were evaluated in PWI samples, after reaction through a spectrophotometric analysis, measuring the absorbance at 490 nm, according to Marassi et al. (2021) method. The final results were expressed as mg/L of glucose using an external calibration curve.

2.4. Free and glycosylated volatile organic compounds extraction and determination in 'Arneis' and 'Cortese' musts and wines

Free and glycosylated volatile organic compounds (VOCs) were investigated for both varieties on the freshly-pressed juice and at two different times during the winemaking process, i.e. after the stabulation (PS) and at the end of alcoholic fermentation (PAF). Moreover, free VOCs were determined one month after bottling (PWI).

2.4.1. Extraction of free and glycosylated volatile organic compounds (VOCs)

The VOCs extraction from juices and wines was performed as described by Giacosa et al. (2019). Briefly, a 50 mL-aliquot of sample was diluted with 100 mL of deionized water and 0.5 mL of 1-heptanol (60 mg/L in 10 % *v*/v absolute ethanol) were added as internal standard. Then, samples were loaded onto a 5-g Sep-Pak C_{18} cartridge (Waters Corporation, Milford, MA, USA), previously activated with methanol and washed with deionized water. Free volatile organic compounds were eluted with 30 mL of dichloromethane. The free fraction was dried over anhydrous sodium sulphate and then concentrated to 50 μL under a stream of nitrogen for the direct injection.

Glycosylated compounds were subsequently eluted with 25 mL of methanol and the eluate was evaporated to dryness using a vacuum rotavapor (Buchi R-210, Flawil, Switzerland) set to 30–35 °C. The glycosylated fraction was dissolved in 10 mL of 0.2 M citrate-phosphate buffer at pH 5 and enzymatic hydrolysis was performed with 50 mg of glycosidase enzyme (Rapidase Revelation Aroma, Corimpex, Romans d'Isonzo, Italy) and incubation at 40 °C for 21 h. After the hydrolysis, two internal standards, 0.5 mL of 1-heptanol and 0.5 mL of 2-octanol (60 mg/L in 10 % v/v absolute ethanol for each one), were added. Finally, the glycosylated precursors were recovered with dichloromethane, following the SPE method previously described, and the glycosylated fraction was then dried over anhydrous sodium sulphate and concentrated to 50 μL under a stream of nitrogen for injection.

1.1.1. Determination of free and glycosylated volatile organic compounds

GC/MS analysis was performed through a GC Agilent 7890A (Agilent Technologies, Santa Clara, CA, USA), equipped with an autosampler Gerstel MultiPurpose Sampler MPS 2 (Gerstel GmbH & Co., Mülheim an der Ruhr, Germany) and a DB-WAX capillary column (30 m × 0.25 mm × 0.5 μm, Agilent Technologies, Santa Clara, CA, USA). Injections of 1 μL were performed in split mode (split ratio 0.9:1) setting the injector temperature to 250 °C. The carrier gas (He) flow rate was 1 mL/min. The VOCs' detection was carried out by the MS Agilent 5975C (Agilent Technologies, Santa Clara, CA, USA) system, using a positive ionization energy of 70 eV and the acquisition range of 30–350 *m/z*. The elaboration of GS/MS data was performed by the Software Agilent G1701EA MSD Productivity ChemStation. Where applicable, the identification of volatile organic compounds was confirmed by comparison with the mass spectra of their respective standards, retention indices (**Table S1**) calculated for each volatile compound using a C_7-C_{30} n-alkanes certified reference material (Sigma-Aldrich, Milan, Italy) or MS data reported in literature and NIST database (www.webbook.nist.gov/chemistry). Semi-quantitative data were obtained by measuring the relative peak area of each identified compound in relation to that of the added internal standard (μg/L of 1-heptanol).

2.5. Sensory analysis of 'Arneis' and 'Cortese' wines

The sensory analysis has been organised in two phases: the preliminary training step and the formal tasting sessions of the wine samples that were conducted in two different vinification points, i.e. at the end of alcoholic fermentation (PAF) and one month after bottling (PWI).

2.5.1. Training sessions

The training sessions consisted of seven half-hour sessions over four weeks. Twelve judges (7 women, 5 men) were selected among university personnel already involved in previous white wine evaluations following the same procedure and able to sensory recognize the selected attributes in water. The panel was further trained on tastes, mouthfeel perceptions, and aromas in white wines, as well as at the use of the scale for this study. Ethical permission, to conduct a human sensory study, was granted by University of Torino Ethics Committee (protocol number 0194129). The participants acknowledge an informed consent statement to participate in the study prior to the sensory sessions. They were informed that they would participate in the sensory survey about wine production, all data will be de-identified and only reported in the aggregate, they were able to withdraw from the survey at any time without giving a reason and that the products tested were safe for consumption.

The sensory descriptors were selected on the basis of the most cited attributes found in literature among neutral varietal white wines (Campo et al., 2008; Fracassetti et al., 2020) and on 'Arneis' and 'Cortese' wines (Piano et al., 2014). In training, the identification technique

(for aroma, taste, and tactile perceptions), ordination task, and unstructured line scale tools (for tastes and tactile perceptions) were adopted on selected aroma descriptors belonging to fruity (*pineapple, lime, lemon, grapefruit, green apple, peach, pear, banana*), floral (*rose, jasmine*), and complex (*almond* and *honey*) perception classes. The selected reference standards (**Table S2**) were dissolved in commercial white wine (Caviro, Faenza, Italy). For taste and mouthfeel (*bitterness*, *astringency*, *acidity*, and *body*), the 1st session was dedicated for the identification of the stimuli in white wines, the 2nd and 3rd to ordination task, the 4th and 5th for scale alignment training. Furthermore, in all sessions (1–7), the aroma training was performed, firstly asking assessors to associate the aromatic stimuli to a descriptor from a given list (1–5), and then without the list (6–7). At the end of each session, the judges were asked to discuss with the panel leader the results and to assess the standard sample again if needed. Before formal session, two sensory sessions were performed with the same tasting sheets and with three neutral varietal wines to assess the panel performance.

2.5.2. Formal sessions

In the official wine sessions, general descriptive analysis (DA) with 10-cm unstructured line scale and Check-All-That-Apply (CATA) methods were adopted for in-mouth descriptors (*bitterness*, *astringency*, *acidity*, and *body*) and aroma descriptors, respectively (Lawless & Heymann, 2010; Valentin et al., 2012). A 10-cm unstructured scale was used also for rating the sample *overall wine quality*. Twenty mL of wine were poured in three-digit randomly coded standard ISO 3591 glasses (International Organization for Standardization, 1977) covered with a petri dish and served at room temperature (18 °C). Mineral water and unsalted crackers were provided as cleanser between samples.

A monadic samples evaluation at PAF (equal volume mixed of the three replications, 12 judges, obtaining 12 answers, for each variety) was proposed to the panel according to different fermentation ends that were related to the CLS time. The formal sample's evaluation of the final wines was performed one months after bottling for each variety separately accordingly to the harvest date (formal session date: 14th December 2022 and 25th January 2023, for 'Arneis' and 'Cortese', respectively). All the samples produced for each variety were tested in one session (4 treatments in duplicate for each variety, panel of 10 and 7 judges for 'Arneis' and 'Cortese', respectively, obtaining a total of 20 and 14 answers).

2.6. Statistical analysis

Statistical analyses were performed using R statistic software (R Foundation for Statistical Computing, Vienna, Austria). For physical-chemical and VOCs analysis, for each variable, one-way analysis of variance (ANOVA), with Tukey HSD post hoc test, was used to evaluate the significant differences ($p < 0.05$) among treatments. In case of heteroscedasticity, the ANOVA with Welch's correction was used, followed by Games Howell test as post-hoc.

For the sensory analysis and multivariate elaboration, the data analysis was performed using FactoMineR (Lê & Husson, 2008) and SensoMineR (Lê et al., 2008) packages. In the bottled wine tasting, the panel performance was evaluated for CATA tasks through the reproducibility index (*Ri*) proposed by Campo et al. (2008). The panel performance was evaluated for each sample set ('Arneis' and 'Cortese', $Ri = 0.37$ and $Ri = 0.48$, respectively), respecting the repeatability requirement ($Ri > 0.20$). For the aroma frequencies from the CATA questionnaire, Correspondence Analysis (CA) was performed, and significant attributes were assessed with Cochran's Q test (Varela & Ares, 2012). For DA's panel evaluation, SensoMineR package was used to assess the agreement and repeatability of the panel, with a three-way ANOVA ("replicates" * "sample" * "judge" as fixed factors and their interaction). The performance was considered adequate when there were no significant differences ($p > 0.05$) in "judges" * "replicates" and "judges" * "sample" interactions, and panel consensus was monitored by principal component analysis (PCA) for each attribute. Samples significant differences were then evaluated by three-way ANOVA with sample and replicates as fixed effect and judges as random effect. For descriptors with significant differences ($p < 0.05$) Tukey HSD was applied.

PCA with free VOCs detected in bottled wines and sensory aroma descriptors (with citation frequency greater than 20 %) was performed. VOCs data was standardized as *z-scores* within each variety to minimize the varietal effects. Aroma descriptors were projected as supplementary quantitative variables after standardization in the same way.

3. Results and discussion

3.1. Impact of CLS treatment on physical-chemical parameters

Physical-chemical parameters of 'Arneis' and 'Cortese', after CLS, alcoholic fermentation, and one month of bottling are shown in the Tables 1 and 2, respectively. For 'Arneis', the results showed that after the CLS all the samples undergo a reduction of the total acidity down to −8 % at AR21 with respect to the control. The same trend was followed by pH values at AR21, although the variation was minimal (0.02 pH units). As well, in 'Cortese' CLS samples both total acidity and pH values decreased (−6 % and −1 % at CO21, respectively, compared to control). The low temperature in both varieties led to a significant decrease of tartaric acid contents, that was evident already after 7 days of CLS, due to tartaric salts precipitation, with the consequence of a reduced acidity. It has been reported that pre-fermentative techniques involving the skin contact can influence the pH of the must, increasing the release of cations from the skin cell wall, and therefore increasing the pH values (Aleixandre-Tudo et al., 2015). Nevertheless, this depends on the varieties and conditions applied (Aleixandre-Tudo et al., 2015; Alti-Palacios et al., 2023; Wang et al., 2016). In the case of CLS, this phenomenon was not observed. In both varieties, ethanol, glycerol, and other organic acids have been investigated but no changes at the end of CLS were found in their contents with respect to control under our experimental conditions, with the exception of a slight decrease of malic acid in 'Arneis' throughout CLS. Overall, these results demonstrate that no microbiological activity affected the juices and no relevant spontaneous fermentations occurred.

After 7 days of CLS, the total polyphenolic index (TPI) decreased in both 'Cortese' and 'Arneis'. In 'Arneis', the lowest TPI value was reached at AR14 (−17 %), but at AR21 the index grew up. Indeed, in 'Cortese' the value of TPI decreased with increasing CLS length (down to −6 % at CO14 with respect to control). Accordingly, the antioxidant capacity (DPPH) decreased with the CLS, but no significant differences were found among different CLS length for 'Arneis' samples. Although polyphenols are well-correlated with the antioxidant capacity of must and wines, other sulphur-containing compounds, such as glutathione and cysteine, can influence the antioxidant capacity (Romanet et al., 2019). In 'Cortese' samples, the antioxidant capacity strongly decreased after 21 days of CLS, which was in line with the TPI value, possibly due to grape flesh cell wall material adsorption of polyphenolic compounds, in particular tannins with a higher molecular mass (Bindon et al., 2010). Previously, 'Cortese' skins were found to be richer in condensed tannins and they were of higher molar mass with respect to 'Arneis' ones (Guaita et al., 2023). In contrast, 'Arneis' grapes have been reported to contain up to two-folds the content of flavonols and hydroxycinnamic acids compared to 'Cortese' ones (Ferrandino et al., 2012). These polyphenols are oxidized faster than condensed tannins, probably leading to a decrease in DPPH already at AR07 in 'Arneis', whereas 'Cortese' could have been affected by both oxidation and adsorption of condensed tannins in a time-dependant manner. During prolonged maceration, secondary oxidation reactions, involving *o*-quinones (yellow-brown colour), can also occur giving colourless or less yellowish pigments, or producing polymer pigments that precipitate (Carbone & Fiordiponti, 2016; Gómez-Míguez et al., 2007). This could be connected also to the fact that the juice resulted clearer (higher L^*) and with a lower yellow

Table 1
Chemical-physical parameters of Arneis juice after cold liquid stabulation (PS), after alcoholic fermentation (PAF), and one month after bottling (PWI).

	Arneis														
		PS					PAF					PWI				
	Juice	AR-Control	AR07	AR14	AR21	*Sign*	AR-Control	AR07	AR14	AR21	*Sign.*	AR-Control	AR07	AR14	AR21	*Sign*
Parameter																
Sugars (g/L)	246±0	248±2 a	246±1 ab	246±1 a	243±1 b	**	8±3 a	<1 b	<1 b	<1 b	**	<1	<1	<1	<1	ns
Total acidity (g/L of tartaric acid)	3.8±0.0	3.6±0.1 a	3.4±0.0 b	3.4±0.0 b	3.3±0.0 b	**	6.3±0.1 a	6.0±0.0 b	5.9±0.0 b	6.2±0.2 ab	**	5.2±0.0 b	5.1±0.1 b	5.3±0.1 b	5.6±0.2 a	**
pH	3.20±0.05	3.18±0.01ab	3.19±0.00 a	3.18±0.00 b	3.16±0.00 c	***	3.20±0.00 a	3.18±0.01 ab	3.16±0.01 b	3.22±0.02 a	**	3.01±0.01bc	3.02±0.01 b	2.99±0.00 c	3.05±0.01 a	***
Tartaric acid (g/L)	4.74±0.05	4.40±0.03 a	4.08±0.02 b	3.98±0.04 c	3.98±0.02 c	***	4.35±0.02 a	4.11±0.03 b	4.10±0.02bc	3.99±0.06 c	***	2.60±0.04	2.56±0.01	2.63±0.05	2.65±0.16	ns
Malic acid (g/L)	0.59±0.00	0.63±0.03 a	0.59±0.01 ab	0.56±0.01 b	0.60±0.01 ab	**	0.77±0.02 b	0.79±0.03 b	0.74±0.00 b	0.94±0.02 a	***	0.73±0.02 b	0.82±0.04 b	0.76±0.02 b	0.93±0.05 a	**
Lactic acid (g/L)	nd	nd	nd	nd	nd		0.22±0.01 b	0.23±0.01 ab	0.25±0.01 a	0.22±0.01 b	*	0.19±0.01	0.19±0.01	0.20±0.01	0.18±0.01	ns
Citric acid (g/L)	nd	nd	nd	nd	nd		0.11±0.01 a	0.10±0.01 ab	0.06±0.00 c	0.09±0.00 b	***	0.13±0.00	0.13±.0.01	0.09±0.04	0.12±0.00	ns
Succinic acid (g/L)	nd	nd	nd	nd	nd		1.23±0.02 ab	1.15±0.03 c	1.19±0.03bc	1.26±0.01 a	**	1.15±0.02ab	1.09±0.03 c	1.13±0.03 bc	1.20±0.01 a	**
Acetic acid (g/L)	nd	nd	nd	nd	nd		0.07±0.00 b	0.08±0.00 b	0.10±0.01 a	0.11±0.00 a	***	0.07±0.00 b	0.08±0.00 b	0.10±0.01 a	0.11±0.00 a	***
Glycerol (g/L)	nd	nd	nd	nd	nd		8.11±0.07 a	7.79±0.01 c	8.01±0.02ab	7.94±0.05 b	***	8.11±0.08 a	7.84±0.04 b	8.09±0.00 a	8.06±0.04 a	**
Ethanol (%Vol)	nd	nd	nd	nd	nd		15.03±0.02bc	15.13±0.03 a	15.00±0.04c	15.09±0.03ab	**	15.05±0.06	15.08±0.08	14.97±0.05	15.04±0.07	ns
Acetaldehyde (mg/L)	0±0	0±0 b	1±0 a	1±0 a	1±0 a	***	16±0	16±1	16±0	18±2	ns	19±1	18±1	18±0	20±2	ns
Total sulfur dioxide (mg/L)	38±1	36±1	36±1	35±1	34±1	ns	31±1 a	31±0 a	24±0 c	27±1 b	***	71±1	71±1	68±3	69±1	ns
TPI (A.u.)	10.1±0.0	9.8±0.1 a	8.3±0.1 bc	8.2±0.0 c	8.4±0.1 b	***	6.4±0.0 b	6.5±0.2 b	6.7±0.1 ab	7.0±0.1 a	**	6.2±0.0 c	6.1±0.0 c	6.4±0.0 b	6.6±0.1 a	***
EC (mg/L)	789±3	764±5 a	647±4 bc	636±0 c	653±7 b	***	496±3 b	506±14 b	519±10 ab	543±9 a	**	482±3 c	477±2 c	499±1 b	518±6 a	***
DPPH (mmol Trolox/L)	2.00±0.08	1.96±0.02 a	1.83±0.03 b	1.80±0.03 b	1.79±0.01 b	***	1.05±0.01	1.02±0.06	1.13±0.05	1.07±0.04	ns	1.30±0.01bc	1.28±0.01 c	1.34±0.01 ab	1.38±0.03 a	**
FC (mg/L of gallic acid)		-	-	-	-		-	-	-	-		143	174	173	167	ns
*L**	83.03±0.29	76.46±0.27c	86.02±0.38 a	85.29±0.26ab	84.06±0.89 b	***	98.16±0.12 a	97.47±0.30 b	96.82±0.02c	97.30±0.15bc	***	98.98±0.01a	98.83±0.08a	98.25±0.07 b	98.09±0.46 b	**
*a**	2.41±0.11	3.91±0.11 a	1.44±0.07 b	1.58±0.29 b	1.87±0.28 b	***	-1.43±0.01 c	-1.19±0.07 b	-0.75±0.11 a	-0.91±0.05 a	***	-0.85±0.03 b	-0.80±0.03 b	-0.65±0.04 a	-0.77±0.03 b	**
*b**	29.39±0.24	33.79±0.13a	26.01±0.23 b	24.88±0.01 c	25.21±0.60 bc	***	8.28±0.22 b	9.86±1.28 ab	11.06±0.84a	11.05±0.85 a	*	5.51±0.10 b	5.56±0.10 b	6.60±0.09 ab	7.63±1.06 a	**
Color and ΔE*			12.6	12.8	11.6			1.7	3.2	2.9			0.2	1.3	2.3	
Total polysaccharides (mg/L glucose)		-	-	-	-		-	-	-	-		319±21	302±28	305±23	364±50	ns

All data are expressed as average value ± standard deviation ($n = 3$). Sign: *, **, ***, and ns indicate significance at $p < 0.05$, 0.01, 0.001, and not significant, respectively, according to ANOVA test. Different lowercase letters within the same row refer to the existence of significant differences between different samples, for each variety and sampling point, according to Tukey's test. nd indicate not detected. TPI (A.u.): total phenolic index (in absorbance units), EC: (−)-epicatechin, DPPH: Antioxidant capacity, FC: Folin–Ciocalteu index, *L**: lightness; *a**: red/green colour coordinate; *b**: yellow/blue colour coordinate, ΔE*: total colour difference *vs* control. Colour was acquired by spectrophotometry, expressed in CIEL*a*b* coordinates and then converted into RGB values for visualization. "AR- Control", "AR07", "AR14" and "AR21" indicate the non-stabulated and the three treatment periods, respectively 7, 14 and 21 days for 'Arneis' at the different vinification stages.

Table 2
Chemical-physical parameters of Cortese juice after cold liquid stabulation (PS), after alcoholic fermentation (PAF), and one month after bottling (PWI).

	Cortese															
		PS					PAF					PWI				
	Juice	CO-Control	CO07	CO14	CO21	*Sign.*	CO-Control	CO07	CO14	CO21	*Sign.*	CO-Control	CO07	CO14	CO21	*Sign.*
Parameter																
Sugars (g/L)	229±1	229±1	230±0	230±0	230±0	ns	<1	<1	<1	<1	ns	<1	<1	<1	<1	ns
Total acidity (g/L of tartaric acid)	3.9±0.0	3.6±0.0	3.1±0.0	3.2±0.7	3.4±0.0	ns	6.9±0.1 a	6.4±0.1 b	6.3±0.0 b	6.4±0.0 b	***	5.7±0.1 b	5.7±0.0 ab	5.8±0.1 ab	5.9±0.0 a	*
pH	3.22±0.00	3.19±0.00 a	3.17±0.00 b	3.16±0.00 c	3.16±0.00 c	ns	3.25±0.00 a	3.19±0.01 b	3.12±0.00 c	3.10±0.01 c	***	3.02±0.01 a	2.99±0.01 b	2.99±0.00 b	2.98±0.02 b	*
Tartaric acid (g/L)	4.73±0.01	4.55±0.03 a	3.72±0.02 c	3.83±0.04 b	3.58±0.00 d	***	4.49±0.02 a	4.00±0.01 b	3.85±0.06 c	3.82±0.01 c	***	2.56±0.03 b	2.64±0.01 ab	2.67±0.02 ab	2.72±0.08 a	**
Malic acid (g/L)	0.71±0.03	0.71±0.03	0.68±0.02	0.67±0.01	0.67±0.04	ns	0.68±0.01 a	0.60±0.00 b	0.57±0.01 c	0.54±0.01 d	***	0.68±0.01 a	0.58±0.02 b	0.53±0.00 b	0.54±0.04 b	***
Lactic acid (g/L)	nd	nd	nd	nd	nd		0.24±0.01 b	0.26±0.00 a	0.25±0.01 a	0.24±0.00 ab	**	0.24±0.01 b	0.26±0.00 a	0.26±0.00 a	0.25±0.01 a	**
Citric acid (g/L)	nd	nd	nd	nd	nd		0.18±0.00 a	0.18±0.00 ab	0.18±0.00 a	0.17±0.00 b	**	0.23±0.00 b	0.26±0.01 a	0.23±0.00 b	0.22±0.01 b	**
Succinic acid (g/L)	nd	nd	nd	nd	nd		1.29±0.01 a	1.18±0.02 b	1.16±0.01 bc	1.14±0.01 c	***	1.25±0.01 a	1.15±0.03 b	1.13±0.01 b	1.12±0.04 b	***
Acetic acid (g/L)	nd	nd	nd	nd	nd		0.15±0.01 c	0.21±0.01 a	0.17±0.00 b	0.23±0.01 a	***	0.15±0.01 c	0.22±0.01 a	0.18±0.01 b	0.23±0.02 a	***
Glycerol (g/L)	nd	nd	nd	nd	nd		8.89±0.11 a	8.90±0.05 a	8.69±0.06 b	8.80±0.03 ab	*	8.95±0.10 a	8.97±0.07 a	8.73±0.05 b	8.93±0.10 ab	*
Ethanol (%Vol)	nd	nd	nd	nd	nd		13.95±0.10	13.98±0.04	14.04±0.03	14.03±0.05	ns	14.03±0.05	14.06±0.02	14.05±0.02	14.06±0.04	ns
Acetaldehyde (mg/L)	1±0	1±0 b	1±0 b	2±0 a	2±0 a	***	18±1 a	18±1 a	15±1 b	17±1 ab	**	22±2	21±1	20±2	20±2	ns
Total sulfur dioxide (mg/L)	36±1	31±1 a	31±2 ab	32±1 a	28±1 b	*	33±1 a	28±1 b	29±1 b	22±1 c	***	66±1 a	64±1 ab	64±1 ab	60±1 b	*
TPI (A.u. x dil)	7.1±0.0	6.5±0.1 a	6.3±0.0 b	6.1±0.0 c	6.2±0.0 bc	***	6.6±0.1 a	6.4±0.1 a	6.0±0.0 b	5.8±0.0 c	***	6.3±0.1 a	6.2±0.1 a	5.9±0.0 b	5.8±0.0 b	***
EC (mg/L)	556±1	508±6 a	493±3 b	478±4 c	482±2 bc	***	512±5 a	502±5 a	470±3 b	451±3 c	***	492±6 a	486±4 a	462±2 b	452±1 b	***
DPPH (mmol Trolox/L)	1.56±0.01	1.35±0.01 a	1.21±0.02 b	1.35±0.01 a	1.12±0.01 c	***	0.95±0.03 a	0.91±0.02 a	0.90±0.01 a	0.78±0.01 b	***	1.99±0.02	1.95±0.01	1.96±0.02	1.91±0.05	ns
FC (mg/L of gallic acid)	-	-	-	-	-		-	-	-	-		157 ab	147 bc	135 c	166 a	**
*L**	90.28±0.55	93.34±0.56 c	95.68±0.24 b	97.63±0.08 a	97.48±0.21 a	***	98.26±0.04	98.16±0.11	98.13±0.38	98.26±0.06	ns	98.84±0.03 a	98.52±0.14 b	98.68±0.10 ab	98.57±0.14 ab	*
*a**	0.04±0.12	-0.47±0.07 a	-0.65±0.01 b	-0.83±0.01 c	-0.62±0.02 b	***	-1.11±0.05 b	-0.87±0.03 a	-0.89±0.11 a	-0.87±0.03 a	**	-0.53±0.05 c	-0.31±0.00 a	-0.42±0.02 b	-0.44±0.03 b	***
*b**	16.85±0.41	13.39±0.38 a	9.89±0.23 b	8.07±0.07 c	9.37±0.27 b	***	6.32±0.09 b	6.70±0.04 a	6.00±0.21 b	5.51±0.05 c	***	4.92±0.09 b	5.13±0.03 a	4.83±0.05 b	4.81±0.05 b	***
Color and ΔE*			4.2	6.8	5.8			0.5	0.4	0.8			0.4	0.2	0.3	
Total polysaccharides (mg/L glucose)		-	-	-	-		-	-	-	-		315±48	299±75	370±83	338±19	ns

All data are expressed as average value ± standard deviation (n = 3). Sign: *, **, ***, and ns indicate significance at $p < 0.05$, 0.01, 0.001, and not significant, respectively, according to ANOVA test. Different lowercase letters within the same row refer to the existence of significant differences between different samples, for each variety and sampling point, according to Tukey's test. nd indicate not detected. TPI (A.u.): total phenolic index (in absorbance units), EC: (−)-epicatechin, DPPH: Antioxidant capacity, FC: Folin–Ciocalteu index, *L**: lightness; *a**: red/green colour coordinate; *b**: yellow/blue colour coordinate. ΔE*: total colour difference (ΔE*) *vs* control. Colour was acquired by spectrophotometry, expressed in CIEL*a*b* coordinates and then converted to RGB values for visualization. "CO-Control", "CO07", "CO14" and "CO21" indicate the non-stabulated and the three treatment periods, respectively 7, 14 and 21 days for 'Cortese' at the different vinification stages.

hue (b^*) as long as the CLS length increased in 'Cortese' except for CO21, whereas in 'Arneis' it was already significantly clearer at AR07 (+13 %) and less yellow (−23 %) and then remained steady.

At PAF, for 'Arneis' wines total acidity values were still lower in AR07 and AR14 (but not significantly for AR21) than control due to the yeast contribution in the production of other organic acids. In the case of AR21 samples, higher contents of succinic acid were found with respect to other CLS samples while those of tartaric acid decreased according to CLS length. For 'Cortese' wines, total acidity and tartaric acid content were lower in stabulated samples, compared to the control, independently on CLS length. At PWI, in both varieties total acidity values were significantly higher in AR21 for 'Arneis' than control, AR07, and AR14 samples ($p < 0.01$) and for 'Cortese' only than control ($p < 0.05$). This may be linked to a higher stability of tartaric acid salts already achieved with the CLS. At PAF, in CLS samples, an increase of acetic acid content was found with respect to control (achieving 0.23 g/L in 'Cortese' and 0.11 g/L in 'Arneis', both at 21 days CLS samples) and it remained unchanged also in bottled wines (PWI) for both the varieties.

'Arneis' wines at PAF, TPI tends to be higher as the CLS length increases, although it does significantly only for AR21 when compared to control. Furthermore, no significant differences were found in the antioxidant capacity. In bottled wines, AR14 and AR21 samples had higher values of TPI and antioxidant capacity, up to +7 % and + 6 %, respectively, with respect to control. In contrast with 'Arneis', for 'Cortese' wines at PAF, TPI decreased ($p < 0.001$), especially in the CO21 sample (−12 % with respect to control). The same applies for the antioxidant capacity (−18 % at CO21). For TPI in bottled wines, the values in wines produced after 14 and 21-day CLS were significantly lower with respect to the other samples, although no significant differences were observed in the antioxidant capacity. In line, also Folin-Ciocalteu results for 'Cortese' CO14 wines were significantly lower than control ($p < 0.01$). Those differences are probably due to the grape phenolic composition (Guaita et al., 2023; Motta et al., 2014): the stabilization of 'Arneis' wines, being the grapes richer in hydroxycinnamoyl tartrates (HCTAs), flavonols, and monomeric flavanols than Cortese ones (Ferrandino et al., 2012), was faster through CLS by the oxidation and removal of these phenolic compounds with racking. In fact, the TPI values are relatively less variable from juice to PWI for 'Cortese' (decrease of 11–18 %) throughout winemaking with respect to 'Arneis', the latter showing a decrease of 35–40 % from juice to PWI.

The trends of L^*, a^*, and b^* with the increase of the stabulation length at PAF changed with respect to PS. Thereby, CLS 'Arneis' samples at PAF had lower L^* values, while significant differences were not found in 'Cortese'. The a^* and b^* values, contrary to what happens after the CLS, increased with 7 days of treatment in 'Arneis' with respect to control. The same applies for the bottled wines but the differences were significant at longer CLS. In particular, 'Arneis' had a higher b^* value, and therefore more intense yellow hue in AR21 wines after bottling. In 'Cortese' stabulated samples at PWI, L^* value was lower ($p < 0.05$) only in CO07, where also a higher b^* was reported with respect to the other samples. The opposite behaviour of b^* in the two varieties agrees with the differences found in TPI values. ΔE^* highlights that just at the end of CLS all 'Arneis' samples showed a visually perceived different colour with respect to the control ($\Delta E^* > 6$). However, the colours of AR14 and AR21 at PAF ($\Delta E^* = 3.2$ and 2.9, respectively) and AR21 at PWI ($\Delta E^* = 2.3$) were also markedly different compared to the control, while in contrast AR07 had a minimum difference ($\Delta E^* < 2$). A ΔE^* of 2.3 was reported as a minimum value to clearly discriminate the colour of white wines in a glass (Sáenz-Gamasa et al., 2009), and therefore from instrumental data no potential sensory impact of CLS was found at PWI, except for 'Arneis' wines with 21 days of CLS. Similarly, the colour differences among 'Cortese' treatments were perceivable in juice after CLS, but they become negligible after alcoholic fermentation and bottling ($0.2 < \Delta E^* < 0.8$). During skin-contact prefermentative treatments, the extraction of water-soluble phenolic compounds occurs, which can be also more easily oxidized giving yellow pigments (Gómez-Míguez et al., 2007; Carbone and Fiordiponti, 2016). Nevertheless, the CLS treatment applied in this study allowed to obtain wines whose colour cannot be easily differentiated from the control probably due to fast oxidative processes, adsorption on fine lees, and also polymerization and precipitation.

3.2. Impact of CLS treatment on free and glycosylated volatile organic compounds

Glycosylated VOCs were determined in the just pressed juice used for this study. From the aromatic point of view, both 'Arneis' and 'Cortese' are considered neutral grape varieties, although the analysis of grape juices' VOCs showed differences in their glycosylated volatile compositions (**Table S3**). The concentration of terpenes in 'Cortese' must was almost double than the one in 'Arneis' (467 vs 273 μg/L), and it was remarkably higher in volatile phenols (1145 vs 703 μg/L). Instead, the juice obtained from 'Arneis' grapes had more than twice glycosylated norisoprenoids (530 vs 196 μg/L). Nevertheless, the different flavour of their wines is due to both the varietal characteristics and the formation of fermentative volatile organic compounds. Free VOCs (Figs. 1 and 2, for 'Arneis' and 'Cortese', respectively) and their glycosylated precursors (Fig. 3) have been investigated in different steps of the winemaking process with the aim of evaluating their evolution and possible correlation with the sensory analysis.

3.2.1. Free and glycosylated volatile organic compounds at the end of CLS

After the CLS treatment, 44 free VOCs (**Table S4**) and 53 glycosylated VOCs (**Table S5**) were identified in the juice. In 'Arneis' juice, significant differences were found in the total free ester content, although these compounds were present as expected before alcoholic fermentation in limited quantity in both varieties (0–3.26 μg/L for 'Arneis' and 8.29–19.53 μg/L for 'Cortese'). The same applies for total free higher alcohol content (59.81–82.15 μg/L in 'Arneis' and 228.48–268.26 μg/L in 'Cortese'), with 2-phenylethanol representing the most abundant compound, particularly for 'Cortese'. The free fraction content of 2-phenylethanol was not significantly different among the treated samples for each variety while that of the glycosylated form decreased significantly for 'Arneis' juice with respect to control. Other free and glycosylated higher alcohols such as 2-ethyl-1-hexanol, 1-octanol, and 1-octen-3-ol decreased with CLS in 'Arneis', and the first two also in 'Cortese' only as glycosylated precursors at CO14 and CO21. Therefore, a significant decrease in the total content of glycosylated higher alcohols was observed for CLS treated 'Arneis' juices.

Generally, free volatile acids were significantly lower in all treated samples for 'Arneis' ($p < 0.01$, −36-44 % with respect to control) whereas they were not affected in 'Cortese'. Phenomena of adsorption have been found between fatty acids and macromolecules deriving from skin contact in Chardonnay variety (Ferreira et al., 1995). Except for free dodecanoic acid whose content increased significantly ($p < 0.001$) in CLS-treated 'Arneis' juices, the other individual compounds followed a decreasing trend. A lower concentration after CLS treatment was found also on glycosylated volatile acids for 'Arneis' ($p < 0.01$, −18-27 % with respect to control), whereas in 'Cortese' there was an increase up to two-times in CO14 and CO21 ($p < 0.001$). It was reported previously an increase in bound fatty acids as consequence of skin maceration (15 °C, 24 h) in cv. 'Narince' (Selli, Canbas, Cabaroglu, Erten and Günata, 2006a).

For free sulphur compounds, a decrease in the total content with CLS was found in both Arneis (down to −84 %, $p < 0.01$), with benzothiaziole decreasing significantly in 'Arneis', while also methionol showed a significant decrease in CO14 and CO21 for 'Cortese' juice compared to control.

'Arneis' and 'Cortese' free C6-compounds were not significantly affected by the CLS with the exception of a decrease in 2-hexenal for 'Cortese' ($p < 0.01$, −24-55 % with respect to control). It has been reported that skin contact increases the release of C6-compounds (Selli,

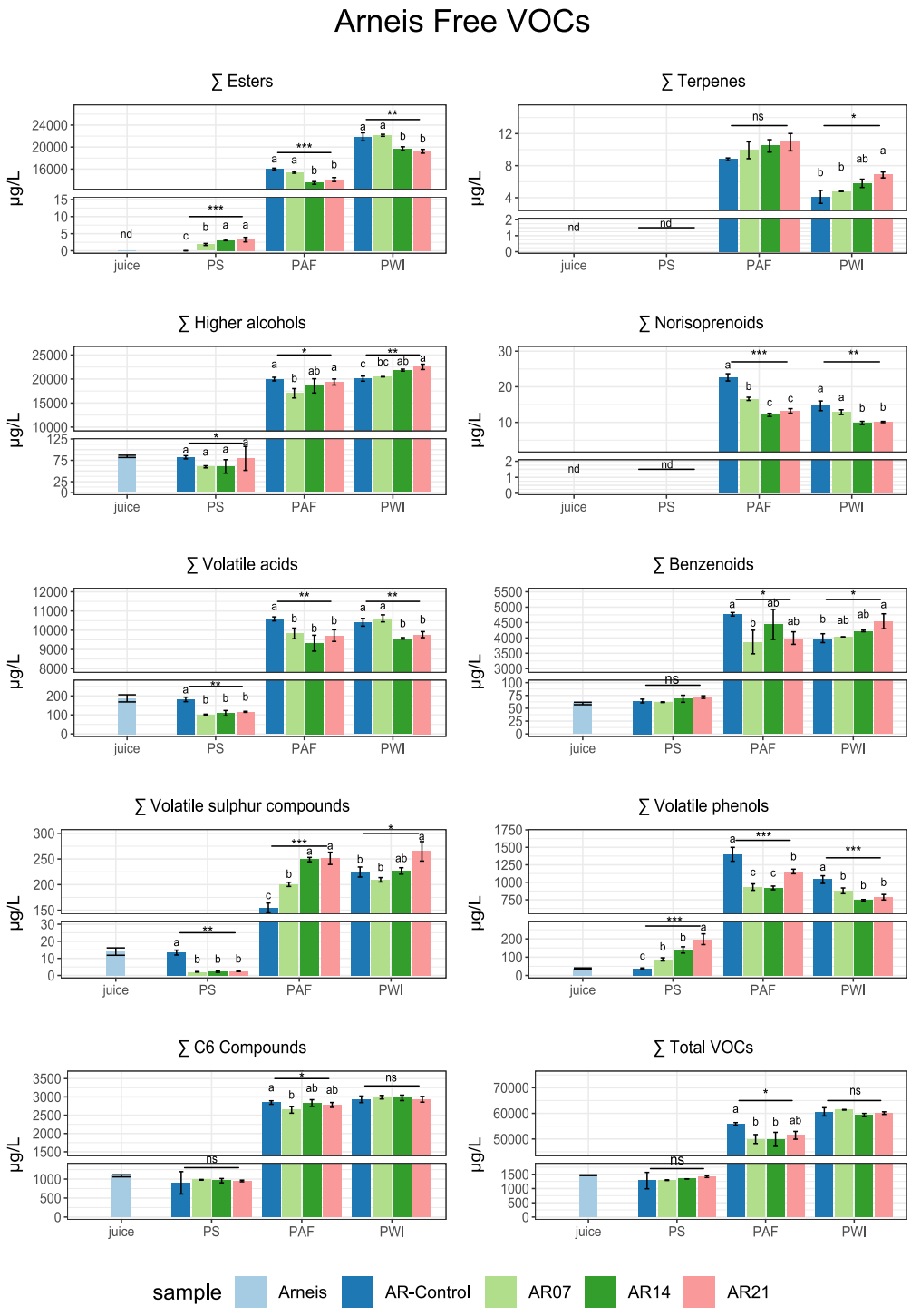

Fig. 1. Free VOCs of 'Arneis' juice, after CLS (PS), after the alcoholic fermentation (PAF), and one month after bottling (PWI). Sign: *, **, ***, and ns indicate significance at $p < 0.05$, 0.01, 0.001, and not significant, respectively, according to ANOVA test. Different lowercase letters within the same sampling point refer to the existence of a significant difference among different samples according to Tukey's HSD test. "AR- Control", "AR07", "AR14" and "AR21" indicate the non-stabulated and the three treatment lengths 7, 14 and 21 days, respectively, for 'Arneis' at the different winemaking stages.

Canbas, Cabaroglu, Erten and Günata, 2006a). Nevertheless, the lower temperature applied with respect to skin maceration may have limited the activity of lipoxygenase (LOX) involved in lipid oxidation that causes the formation of C6-compounds (Costantini et al., 2006). Furthermore, losses of C6-compounds and of their precursors can occur by adsorption on macromolecules and skin components (Ferreira et al., 1995). A decrease (−9–15 %) was found for 'Arneis' with the lowest content detected at AR14 in the glycosylated fraction, in particular related to 1-hexanol. In 'Cortese', C6 compounds did not show significant differences in the glycosylated fraction.

Concerning varietal compounds (Figs. 1 and 2), the only free VOCs' class significantly affected by CLS in both the varieties under evaluation were volatile phenols. Increasing CLS length, their concentrations in 'Arneis' increased ($p < 0.001$) with the highest value achieved in AR21 sample (up to 5-fold the control content), whereas the differences in 'Cortese' between control and CLS treated samples were independent of

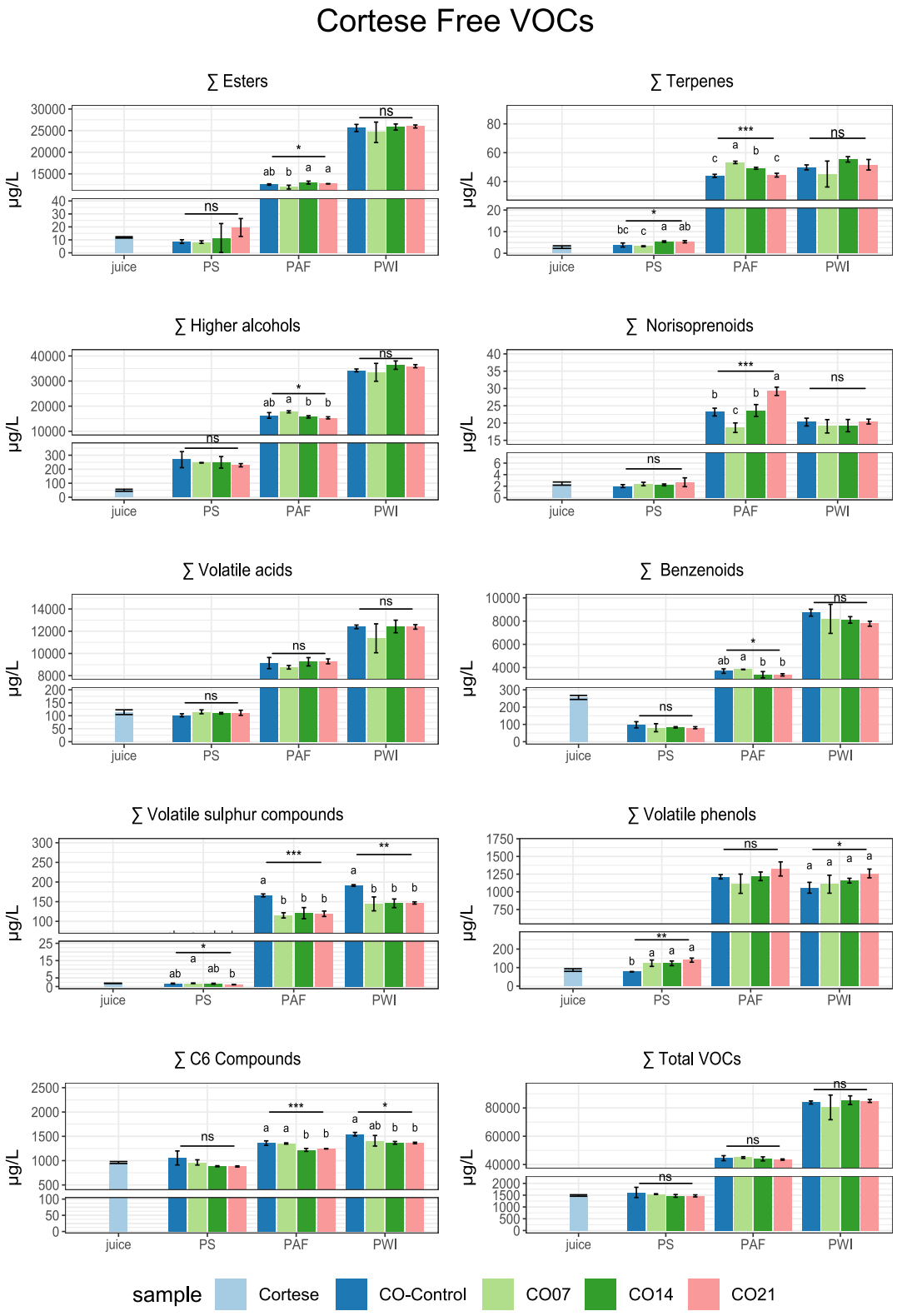

Fig. 2. Free VOCs of 'Cortese' juice, after CLS (PS), after the alcoholic fermentation (PAF), and one month after bottling (PWI). "P07", "P14" and "P21" indicate the three treatment periods, respectively 7, 14 and 21 days. Sign: *, **, ***, and ns indicate significance at $p < 0.05$, 0.01, 0.001, and not significant, respectively, according to ANOVA test. Different lowercase letters within the same sampling point refer to the existence of a significant difference among different samples according to Tukey's HSD test. "CO-Control", "CO07", "CO14" and "CO21" indicate the non-stabulated and the three treatment lengths 7, 14 and 21 days, respectively, for 'Cortese' at the different winemaking stages.

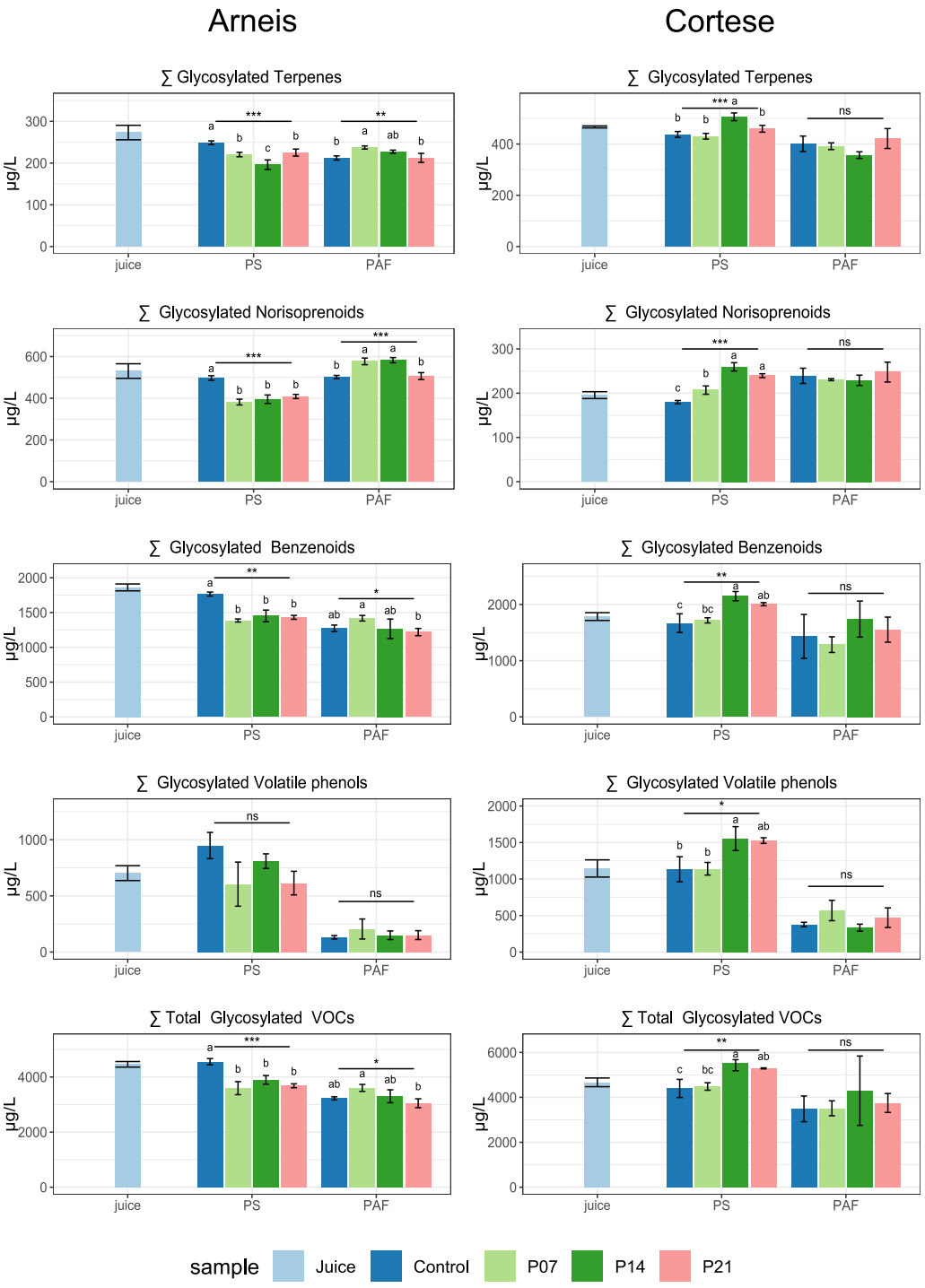

Fig. 3. Glycosylated VOCs of 'Arneis' and 'Cortese' juices, after CLS (PS), and after the alcoholic fermentation (PAF). Sign: *, **, ***, and ns indicate significance at $p < 0.05$, 0.01, 0.001, and not significant, respectively, according to ANOVA test. Different lowercase letters, for each variety, within the same sampling point refer to the existence of a significant difference among different samples according to Tukey's HSD test. "Control", "P07", "P14" and "P21" indicate the non-stabulated and the three treatment lengths 7, 14 and 21 days, respectively, for 'Cortese' and 'Arneis' at different winemaking stages.

the treatment length ($p < 0.01$, +58–80 %). On both the varieties, free 4-vinylphenol, 4-vinylguayacol, and syringol increased significantly with all CLS lengths tested. In 'Arneis', the content of glycosylated volatile phenols decreased in different extent with the CLS treatment, oppositely to the free ones. The most relevant compound was 4-vinylguaicol, in terms of quantity and decrease, followed by 4-vinylphenol, even though the differences with respect to control were only significant for the former ($p < 0.05$, −11-42 %). Glycosylated 3,4,5-trimethoxyphenol and *p*-cresol also decreased significantly with the CLS treatment. The reduction is not in line with the free VOC release, therefore some differences related to adsorption and extraction may have occurred. The opposite behaviour was found in 'Cortese', with the highest content found of glycosylated 4-vinylphenol, 4-vinylguayacol, phenol, syringol, and 3,4,5-trimethoxyphenol at CO14 and CO21 ($p < 0.05$, up to 37 % higher than control and CO07 samples for total glycosylated volatile phenols). This highlights a varietal effect of the CLS technique, strongly related to the varietal VOCs profile and content. In other studies, prolonged contact with grape solid parts (skin contact) also caused

increased quantities of free 4-vinylphenol and decreased ones of both 4-vinylphenol and 4-vinylguaicol in cv. 'Narince' (Selli, Canbas, Cabaroglu, Erten and Günata, 2006a).

As regards free terpenes, in 'Cortese' juices the highest content was found in CO14 and CO21 samples mainly due to the increase of linalool, whereas free terpenes were not detected in 'Arneis'. Terpenes are influenced by several factors that occur during the winemaking process, including the extraction from grape skin as well as the hydrolysis of the bound precursors that can be favoured by some pre-fermentative techniques, such as the presence of enzymes. Pre-fermentative treatments influence the total concentration of terpenes, but different trends can be observed depending on the grape variety and also the vintage (Alti-Palacios et al., 2023). Anyway, the increase observed in 'Cortese' stabulated juices agrees with the results previously reported for free monoterpenes in 'Traminer' grape must after 7 days of stabulation at 2 °C (Philipp et al., 2022). More compounds were found as bound fraction (8 terpenes), with higher total concentration in 'Cortese' than 'Arneis'. In the latter, the CLS treatment led to a significant decrease of total bound terpenes ($p < 0.001$, −9-21 %) as occurred for geranic acid, whereas in 'Cortese' the concentration increased at CO14 with respect to control ($p < 0.001$, +16 %) in agreement with most of individual glycosylated terpenes, in particular 8-hydroxylinalool. Several studies reported a decrease of different extent on free and bound terpenes during skin contact (Aleixandre-Tudo et al., 2015; Alti-Palacios et al., 2023; Selli, Canbas, Cabaroglu, Erten and Günata, 2006a).

In contrast, 'Arneis' showed higher contents of bound norisoprenoids with respect to 'Cortese', which decreased significantly down to −23 % in the first variety by the treatment ($p < 0.001$, −18-23 %). Among them, 3-oxo-α-ionol decreased in all the CLS samples with respect to control. Instead, total content of glycosylated norisoprenoids increased in all CLS samples of 'Cortese' ($p < 0.001$, up to +44 % with respect to the control). Free norisoprenoids were not detected in 'Arneis', and their low concentrations were not affected by CLS in 'Cortese'.

Free benzenoids were not affected by the CLS treatment as well, apart from increased contents of homovanillinic acid in 'Arneis' juice and acetovanillone in 'Cortese' with increasing the CLS length. The bound fraction changed significantly (for both varieties $p < 0.01$) with CLS in a variety-dependant way: in 'Arneis', total content of bound compounds decreased down to −22 % in all CLS samples, decreasing significantly 11 of the 12 detected glycosylated compounds with respect to the control; in 'Cortese', total glycosylated benzenoid content in CO14 and CO21 samples was higher than control (+29 % and + 20 %, respectively). The major benzenoids in the two fractions were benzyl alcohol in 'Arneis' and both homovanillyl alcohol and benzyl alcohol in 'Cortese' juices.

In general, pre-fermentative macerated musts show higher contents of mostly free varietal compounds and glycosylated aroma precursors. However, a longer contact time does not always lead to a greater presence of these compounds due to a balance between extraction, adsorption, and interaction with other compounds or medium components (Alti-Palacios et al., 2023).

Overall, no significant differences were observed in total VOCs (representing the sum of the concentrations corresponding to the different chemical classes) in the free fraction for both the varieties with the CLS treatments. In contrast, the total glycosylated compounds were affected by CLS even if in different extent depending on the variety: in 'Cortese' samples, at the end of CLS, total content of glycosylated VOCs was significantly higher already in CO14 (+26 %) and CO21 (+20 %), whereas in 'Arneis' the CLS caused a decrease in the total content of glycosylated VOCs even at AR07 (from −14 % to −21 % for the different treatments with respect to control).

3.2.2. Free and glycosylated volatile organic compounds in wines

At the end of alcoholic fermentation (PAF), free fermentative volatile organic compounds (**Table S6**), e.g. esters, higher alcohols, volatile acids, sulphur compounds, were affected in different extent by the CLS. Esters and higher alcohols are the major chemical classes in young wines produced from neutral varieties. Esters impact on the final fruity aroma of white wines. Instead, higher alcohols can contribute to a positive note when their concentration is less than 300 μg/L but they are related with pungent notes above this threshold (Alti-Palacios et al., 2023). A varietal effect was evident concerning fermentative volatile organic compounds, which were differently influenced by CLS depending on the chemical class and the variety. According to Fig. 2, 'Cortese' PAF and PWI wines did not show a clear trend for free esters, higher alcohols, and volatile acids with some few exceptions, such as 3-methyl-1-pentanol, 2-phenylethanol, and hexanoic acid, whose contents decreased in CLS treated samples at PAF (**Table S5**). In 'Arneis' PAF wines, both esters and higher alcohols decreased significantly with respect to control ($p < 0.001$ and $p < 0.05$, respectively), reaching the lowest value in AR14 and AR07 samples, respectively (−15–16 %). Particularly, AR14 and AR21 samples, and in minor extent AR07, had a significant reduction of the concentration of isoamyl acetate, responsible of *banana* flavour, being the most abundant ester detected. The same trend was maintained in 'Arneis' for free esters also after bottling (PWI, Fig. 1), with AR14 and AR21 samples having significantly lower contents of esters ($p < 0.01$), but being those of significantly higher contents of higher alcohols ($p < 0.01$). Among higher alcohols, 2-phenylethanol has a pleasant aroma descriptor giving *rose* notes and it can contribute to all 'Cortese' wines whereas only to 'Arneis' wine produced from juice stabulated for 21 days whose content is very close to odour threshold (Table 3). The increase in higher alcohols is often reported when pre-fermentative maceration techniques are applied (Aleixandre-Tudo et al., 2015; Alti-Palacios et al., 2023; Wang et al., 2016), whereas esters are more influenced by variety and treatments (Philipp et al., 2024).

At PAF, the total content of free volatile acids in 'Arneis' CLS samples was lower with respect to control and this behaviour was confirmed after bottling for AR14 and AR21 samples ($p < 0.01$, Fig. 1).

All these fermentative compounds are usually related to the fermentation kinetics and to the yeast strain (Furdíková et al., 2017). A low quantity of yeast assimilable nitrogen can have significant implications in decreasing the production of higher alcohols via Ehrlich pathway and consequently of esters (Aleixandre-Tudo et al., 2015; Casalta et al., 2013; Guittin et al., 2021). For volatile acids, the lower concentration may be related to a loss of precursors due to adsorption on macromolecules and skin components extracted during CLS (Ferreira et al., 1995). The increase of free sulphur compounds, in all 'Arneis' stabulated samples, was significant after alcoholic fermentation ($p < 0.001$). The contents of methionol and 3-ethylmercapto-1-propanol increased in wines with increasing the CLS length, whereas those of benzothiazole increased significantly in AR07 and AR14 samples when compared to control (**Table S6**). This increasing trend was maintained after bottling in 'Arneis' only in AR21 sample (Table 3). Despite this increase due to the CLS, no wine reached values that exceeded the olfactory threshold. The opposite trend was found for 'Cortese' wines at both PAF and PWI (all CLS samples showing significantly lower contents of free sulphur compounds, $p < 0.001$ and $p < 0.01$ for PAF and PWI, respectively, with respect to control, Fig. 2).

A significant decrease in total free C6-compounds was observed also in 'Cortese' PAF and PWI wines produced with CLS lengths of 14 and 21 days when compared to control ($p < 0.001$ and $p < 0.05$ for PAF and PWI, respectively, around −10 %, Fig. 2) whereas the lowest content in 'Arneis' wines corresponded to AR07 sample after alcoholic fermentation, but no significant differences after bottling (Fig. 1). These trends corresponded mainly to the variations in 1-hexanol contents whereas 3-hexen-1-ol was barely affected in both varieties, although were reported concentrations above odour threshold in 'Arneis' that give *green and herbaceous* aromatic notes (Tables 3 **and S6**). This decrease in the concentration of C6-compounds is valuable with respect to skin contact strategies. Usually, the wines resulting from pre-fermentative skin contact show higher hexanol contents because of the formation of C6-aldehydes and C6-alcohols by the enzymatic and chemical oxidation

Table 3
Free volatile compounds (μg/L of 1-heptanol) of control and CLS-treated Arneis and Cortese wines analyzed one month after bottling (PWI).

Chemical class/ compound	Ref.	Descriptors	OT[#] (μg/L)	Arneis					Cortese				
				AR-Control	AR07	AR14	AR21	*Sign*	CO-Control	CO07	CO14	CO21	*Sign*
Acetate esters													
Isoamyl acetate	[1]	Banana, fruity	30	3859.76 ±8.49 a	4019.11 ±67.32 a	3127.68 ±11.28 b	3008.71 ±92.24 b	***	3485.29 ±201.08 a	2863.69 ±355.02 b	3961.5 ±78.55 a	3948.61 ±118.32 a	*
(*E/Z*)-3-Hexen-1-ol acetate	[2]	Fresh green, cut grass	-	60.15±1.19 a	63.40±2.05 a	52.48±0.74 b	48.72±1.61 b	***	nd	nd	nd	nd	
1,3-Propanediol diacetate	[2]	Fruity	-	43.39±3.46	46.67±0.98	38.73±2.39	40.37±1.57	ns	nd	nd	nd	nd	
2-Phenylethyl acetate	[1]	Sweet, honey, floral, rose	250	1311.57 ±53.06 a	1232.09 ±5.60 ab	1170.25 ±4.12 b	1133.75 ±44.31 b	**	1234.43 ±78.14 a	999.72 ±112.68 b	1249.79 ±47.21a	1182.11 ±41.00 ab	*
Hexyl acetate	[1]	Lolly, apple,cherry, pear, sweet floral	115	441.35 ±13.57a	471.55±1.07 a	403.18 ±11.52 b	388.17 ±11.35 b	***	204.55 ±14.81 ab	172.81±16.32 b	217.94 ±16.70a	222.10±5.86 a	*
Ethyl esters													
Ethyl hexanoate	[3]	Green apple, fruity, tropical, floral, strawberry	14	1071.21 ±23.42 b	1157.78 ±30.36 a	920.64±2.41 c	941.09 ±20.29 c	***	973.46 ±61.92	913.80±95.50	1038.65 ±28.11	1014.86 ±14.09	ns
Ethyl octanoate	[3]	Fruity, sweet, waxy	5	1928.94 ±92.25 a	2097.97 ±29.68 a	1546.07 ±36.64 b	1561.66 ±52.28 b	***	1990.41 ±114.32 ab	1791.37 ±168.65 b	2083.85 ±79.77 ab	2101.33 ±50.32 a	*
Ethyl decanoate	[2]	Fruity, grape,pear, apple	200	952.14 ±66.42 a	975.85 ±45.35 a	780.95 ±3.61b	717.33 ±22.93 b	**	870.17 ±69.09 a	865.15±72.35 a	959.60 ±35.10 a	986.66 ±22.39 a	*
Ethyl 4-hydroxybutanoate	[4]	Pineapple, rose, tropical fruit	-	1272.87 ±103.51 bc	1607.88 ±10.42 a	989.09 ±101.46 c	1352.67 ±131.05 ab	**	581.69 ±28.80 b	469.92±67.96 c	681.38 ±33.41 ab	771.75 ±20.85 a	***
Ethyl 3-hydroxybutanoate	[2]	Fruity, grape, green	20	206.36 ±32.23 a	225.92 ±17.88 a	137.50±4.51 b	147.44±8.35 b	**	125.68±6.10 b	108.17±9.86 c	137.29±2.09 b	154.52±2.89 a	***
Ethyl 2-hydroxy-4-methylpentanoate	[2]	Fresh blackberry	-	118.02±4.02 b	127.02±8.50 ab	142.55±6.21 a	128.99±6.61 ab	*	110.64±2.22	108.47±10.34	113.74±3.57	102.43±0.29	ns
Diethyl malate	[1]	Over-ripe, peach, prune	760[§]	526.59 ±15.99 a	437.68±3.04 b	355.20 ±10.50 c	339.72 ±11.56 c	***	498.95±9.93 a	459.73±32.44 a	380.72±1.16 b	345.59±9.46 b	**
Ethyl lactate	[5]	Milk, soap, butter, fruits	150[§]	2489.74 ±74.67 a	2461.04 ±36.47 a	2542.85 ±151.51a	2261.72 ±87.18 a	*	3157.91 ±100.76	3413.64 ±331.49	3387.73 ±251.20	3419.30 ±194.72	ns
Monoethyl succinate	[2]	Caramel, coffee	1,000[§]	6328.95 ±361.54	6164.69 ±22.78	6479.59 ±321.85	6232.93 ±223.60	ns	10912.49 ±511.85	11148.63 ±1232.01	10447.37 ±326.14	10648.96 ±397.37	ns
Diethyl succinate	[6]	Oily, fruity, floral, caramel	200[§]	1020.67 ±7.72 a	845.40±8.31 b	754.23±3.28 c	661.85±6.38 d	***	1181.43 ±20.52 a	1053.87 ±60.07 b	942.48±6.20 c	868.03 ±14.37 c	***
Ethyl phenyllactate	[2]	Spicy peppery, black pepper	-	237.62±3.15 b	208.97±2.19 c	268.62±8.98 a	268.27±6.04 a	***	282.22±9.10 a	243.63±22.50 b	229.92±0.77 b	196.16±5.34 c	***
∑ ***Esters***				21869.32 ±729.03 a	22143.02 ±181.31 a	19709.60 ±343.78 b	19233.38 ±345.01 b	**	25609.34 ±830.19	24612.60 ±2365.15	25831.95 ±704.23	25962.42 ±352.63	ns
Higher alcohols													
Isoamyl alcohol	[1]	Harsh, stale, fusel odour	30[§]	11593.41 ±165.53 b	11969.19 ±40.48 ab	12642.92 ±222.22 a	12505.99 ±297.06 a	**	17794.48 ±456.08 a	17872.30 ±1806.45 a	19872.57 ±791.46 a	19688.55 ±518.22 a	*
3-Methyl-1-pentanol	[6]	Vinous, herbaceous, cocoa	50[§]	207.96±3.81 b	240.04 ±13.84 a	172.61±4.62 c	171.51 ±12.76 c	***	215.23 ±11.19 a	172.92±6.11 b	187.70 ±10.51 b	138.44±7.11 c	***
2,3-Butanediol	[4]	Fruity, fresh	150[§]	46.66±14.55	46.62±13.96	41.48±3.35	35.26±6.30	ns	34.78±6.37	89.24±88.36	54.16±22.26	327.77 ±497.98	ns
2-Phenylethanol	[6]	Floral, rose	10[§]	8229.16 ±399.33 b	8214.94 ±44.89 b	8991.14 ±4.49 ab	9818.64 ±347.99 a	**	16210.79 ±321.33	15348.53 ±1681.72	16235.76 ±904.81	15768.86 ±415.92	ns
∑ ***Higher alcohols***				20077.19 ±520.32 c	20470.79 ±23.39 bc	21848.15 ±209.76 ab	22531.40 ±550.61 a	**	34255.28 ± 566.04	33482.98 ±3562.12	36350.18 ±1671.45	35923.61 ±3628.32	ns
Volatile acids													

(*continued on next page*)

Table 3 (*continued*)

Chemical class/ compound	Ref.	Descriptors	OT[#] (μg/L)	Arneis					Cortese				
				AR-Control	AR07	AR14	AR21	*Sign*	CO-Control	CO07	CO14	CO21	*Sign*
Isobutyric acid	[2]	Cheese, pungent	2300	380.87 ±15.68 b	392.86 ±25.77 b	500.15 ±12.95 a	483.37 ±27.46 a	**	482.77 ±46.29	453.86±48.59	486.28 ±74.97	472.80 ±42.58	ns
Butanoic acid	[5]	Pungent	170	392.32 ±13.11 b	461.12±9.16 a	340.21 ±18.71 c	341.92±7.57 c	***	405.16±6.14	389.96±50.07	396.92 ±46.39	414.11 ±12.09	ns
Isovaleric acid	[5]	Taleggio Cheese, rancid, sweaty, stinky	700	953.31 ±34.72 ab	1014.93 ±9.36 a	952.24 ±29.02 ab	932.41±7.83 b	*	1138.30 ±25.66	1052.17 ±117.50	1103.11 ±26.50	1015.00 ±29.17	ns
Hexanoic acid	[6]	Sour, vinegar, cheese, sweaty, chemical	420	3583.02 ±145.35 a	3757.09 ±10.47 a	3250.61 ±67.36 b	3479.42 ±42.02 ab	**	4511.82 ±146.23	3939.79 ±434.00	4347.31 ±179.53	4313.05 ±79.70	ns
Octanoic acid	[3]	Goat rancid cheese, fatty, oily, acetic	500	3610.39 ±68.87 a	3544.46 ±107.84 ab	3231.47 ±118.11 b	3230.14 ±117.95 b	**	4192.15 ±51.01	3912.82 ±449.23	4375.62 ±247.74	4381.17 ±102.82	ns
Nonanoic acid	[1]	Must, fat	-	7.73±0.61	11.75±2.41	6.54±0.14	7.34±0.86	ns	7.89±1.74 ab	5.53±0.65 b	7.82±1.09 ab	8.67±0.92 a	*
Decanoic acid	[5]	Vinegar, animal, fatty, rancid, citrus, phenolic	1000	1338.47 ±47.97 a	1268.88 ±77.52 ab	1184.84 ±3.39 ab	1146.67 ±15.76 b	**	1546.77 ±35.84	1539±199.69	1652.29 ±56.78	1700.54 ±49.87	ns
9-Decenoic acid	[2]	Waxy, creamy, cheesy	-	84.90±21.58	97.93±16.55	58.37±11.23	87.28±9.94	ns	38.15±1.87 a	12.07±1.09 b	6.79±2.31 c	11.88±1.83 b	***
Dodecanoic acid	[6]	Chemical, fatty, rancid	1000	62.85±4.03	68.11±6.35	53.17±6.90	62.28±9.10	ns	69.10±3.16 a	58.93±9.84 a	59.34±3.11 a	71.86±2.06 a	*
∑ Volatile acids				10413.86 ±201.54 a	10617.14 ±182.58 a	9577.60 ±37.33 b	9770.83 ±155.72 b	**	12392.10 ±163.49	11364.16 ±1301.21	12428.68 ±565.60	12389.08 ±202.67	ns
Volatile sulphur compounds													
3-Ethylmercapto-1-propanol	[7]	Sweat odour, roasted, potato, broth	60	19.02±0.36 c	19.74±0.59 bc	20.68±0.41 b	22.01±0.31 a	***	24.79±0.81 a	19.29±2.13 b	20.07±0.50 b	17.71±0.19 b	***
Methionol	[8]	Vegetables, Boiled potato, Cabbage	500	201.29±9.23 b	185.73±3.40 b	201.29±4.67 b	237.03 ±18.34 a	*	160.36±2.04 a	119.28±15.04 b	118.15 ±10.61 b	123.45±3.89 b	**
Benzothiazole	[7]	Burnt Rubber	350	4.25±0.23 b	3.97±0.28 b	4.71±1.20 ab	5.83±0.81 a	*	5.97±2.29	5.82±0.58	7.65±2.59	5.57±0.97	ns
∑ Volatile sulphur compounds				224.56±9.76 b	209.44±4.26 b	226.68±6.28 ab	264.88 ±19.01 a	*	191.12±2.08 a	144.39±17.74 b	145.87 ±10.98 b	146.73±2.81 b	**
C6 compound													
1-Hexanol	[3]	Green, resin, flower	2500	1711.79 ±53.31	1679.92 ±23.73	1732.37 ±44.89	1701.72 ±38.19	ns	1191.35 ±29.97 a	1079.87 ±79.14 ab	1025.70 ±20.01 b	1023.53 ±21.22 b	**
(*E*/*Z*)-3-Hexen-1-ol	[3]	Green	(400/ 70)	1222.28 ±36.18	1311.33 ±24.25	1240.97 ±29.18	1232.17 ±40.51	ns	353.42±9.88	328.62±29.81	340.59±7.61	339.54±5.92	ns
∑ C6 compounds				2934.07 ±89.47	2991.25 ±47.98	2973.34 ±74.07	2933.89 ±78.42	ns	1544.77 ±35.52 a	1408.49 ±108.86 ab	1366.29 ±27.62 b	1363.08 ±15.58 b	*
Terpenes													
Linalool	[6]	Rose, Citrus	15	nd	nd	nd	nd		49.79±1.73	45.19±9.00	55.37±1.90	51.63±3.65	ns
Geraniol	[6]	Rose, geranium	30	4.10±0.81 b	4.79±0.00 b	5.80±0.52 ab	6.84±0.37 a	**	nd	nd	nd	nd	
∑ Terpenes				4.10±0.81 b	4.79±0.00 b	5.80±0.52 ab	6.84±0.37 a	*	49.79±1.73	45.19±9.00	55.37±1.90	51.63±3.65	ns
Norisoprenoids													
3-Oxo-α-ionol	[2]	Spicy	-	14.64±1.36 a	12.86±0.67 a	9.83±0.45 b	10.10±0.17 b	**	20.28±1.13	19.06±1.93	19.25±1.77	20.42±0.71	ns
Benzenoids													
Vanillin	[6]	Vanilla, sweet pastry	60	3.64±0.30	3.53±0.56	2.73±0.17	2.44±0.12	ns	7.41±0.44	7.74±1.22	8.13±2.59	6.62±0.49	ns
Benzyl Alcohol	[6]	Caramel, fruity, nutty, cherry, rose	200[§]	27.48±2.94	29.54±0.75	26.57±0.79	26.84±1.19	ns	34.30±1.92	31.48±3.68	29.68±0.50	28.97±0.34	ns
Homovanillinic acid				24.12±2.75 a	20.64±1.42 ab	16.93±1.18 b	17.58±0.89 b	*	31.58±2.10 a	28.29±3.57 ab	22.87±2.63 b	23.08±2.01 b	**
Tyrosol	[5]	Bees wax, honey-like	-	3863.48 ±141.85 b	3908.40 ±5.88 b	4120.21 ±28.08 ab	4435.94 ±243.69 a	*	8382.74 ±312.91	7876.82 ±1216.72	7801.10 ±280.96	7467.79 ±209.25	ns
3,4,5-Trimethoxy-benzenemethanol				7.94±0.25 a	8.34±0.30 a	4.87±0.07 b	5.37±0.16 b	***	14.48±1.00	14.6±1.42	13.89±1.29	15.42±0.17	ns

(*continued on next page*)

Table 3 (*continued*)

Chemical class/ compound	Ref.	Descriptors	OT[#] (µg/L)	Arneis					Cortese				
				AR-Control	AR07	AR14	AR21	*Sign*	CO-Control	CO07	CO14	CO21	*Sign*
Homovanillyl alcohol				15.41±0.94 a	15.58±0.34 a	11.71±0.17 b	12.28±0.72 b	**	59.51±2.19	55.89±7.28	53.28±3.12	55.19±2.06	ns
Acetovanillone	[9]	Floral, clove, vanilla	1000	26.77±1.06 a	28.86±0.72 a	22.98±0.06 b	22.48±0.82 b	***	167.04±6.55	156.98±16.90	153.86 ±11.36	157.90±2.48	ns
3-Hydroxy-4-phenyl-2-butanone	[2]	Creamy, sweet fruity	-	23.26±1.56 a	18.44±1.28 b	20.20±1.23 ab	18.77±0.64 b	*	29.06±3.16 a	20.97±1.61 b	19.76±0.82 b	17.48±0.95 b	***
∑ Benzenoids				3992.10 ±144.28 b	4033.34 ±0.51 ab	4226.21 ±25.23 ab	4541.69 ±243.13 a	*	8726.12 ±299.44	8192.77 ±1250.31	8102.57 ±280.81	7772.46 ±214.53	ns
Volatile phenols													
3,5-Di*tert*-butylphenol				31.15±0.15 ab	34.45±2.04 a	28.59±0.03 b	28.53±1.65 b	*	39.96±3.03	37.73±2.71	41.35±6.42	42.05±3.01	ns
4-Vinylguaiacol	[5]	Spicy, smoked, phenolic, curry	440	455.02 ±24.51 a	455.28 ±20.67 a	342.51±6.62 b	346.16 ±10.15 b	***	851.61 ±59.22 b	886.30±88.24 ab	924.81 ±35.04 ab	1004.99 ±24.53 a	*
Phenol	[2]	Sweet, tarry (phenol)	-	25.42±11.53	15.52±0.03	13.93±1.35	13.77±0.53	ns	20.61±4.24	24.14±3.24	22.45±0.77	22.04±2.99	ns
2,6-Dimethoxyphenol (Syringol)	[10]	Smoke, phenolic	570	71.38±23.40 a	48.73±1.55 a	34.15±1.34 a	33.38±1.56 a	*	50.00±10.32	52.67±4.95	49.12±3.44	50.61±5.03	ns
4-Vinylphenol	[5]	Clove, medicinal	770	455.74 ±35.31 a	324.22 ±16.57 b	324.41±0.85 b	364.86 ±42.47 ab	*	95.28±15.51	106.63±29.06 a	119.98 ±10.81	140.03 ±31.74	ns
∑ Volatile phenols				1038.69 ±54.95 a	878.20 ±40.79 b	743.58 ±10.14 b	786.70 ±39.53 b	***	1057.46 ±76.56 a	1107.47 ±127.11 a	1157.70 ±33.37 a	1259.73 ±61.74 a	*
Lactones													
Butyrolactone	[11]	Caramel, sweet	35	35.31±2.24 ab	43.14±3.60 a	23.08±2.96 c	32.59±3.79 b	**	16.98±3.65	21.93±9.82	29.95±4.56	60.04±33.11	ns
Total volatile compounds				60603.87 ±1597.57	61403.97 ±118.91	59343.86 ±628.00	60112.30 ±496.63	ns	83863.22 ±1068.20	80399.03 ±8712.79	85487.81 ±3068.92	84949.19 ±1047.22	ns

[#]Odour threshold; [§] Values expressed in mg/L.

All data are expressed as average value ± standard deviation (n = 3). Sign: *, **, ***, and ns indicate significance at $p < 0.05$, 0.01, 0.001, and not significant, respectively, according to ANOVA test. Different lowercase letters within the same row refer to the existence of significant differences between different samples, for each variety and sampling point, according to Tukey's HSD test. "CO-Control", "CO07", "CO14" and "CO21" indicate the non-stabulated and the three treatment periods, 7, 14 and 21 days, respectively, for 'Cortese' wines and "AR- Control", "AR07", "AR14" and "AR21" indicate the non-stabulated and the three treatment periods, 7, 14 and 21 days, respectively, for 'Arneis' wines.

Sensory descriptors were reported after comparison of literature and sources available online: [1] Fracassetti et al., 2020*; [2] www.thegoodscentscompany.com; [3]* Ferreira et al., 2000*; [4] Scutarașu et al., 2022; [5]* Lambrechts & Pretorius, 2000*; [6] Sánchez-Palomo et al., 2017; [7] Lavigne-Cruège and Dubourdieu, 1996; [8]* Rutan et al., 2014*; [9]* Gambetta et al., 2014*; [10]* Lopez et al., 2002*;[11] Sánchez-Palomo et al., 2010.*

of fatty acids extracted from the grape skins (Aleixandre-Tudo et al., 2015).

Concerning free and glycosylated varietal VOCs (**Tables S6 and S7, respectively**), as already previously reported for aromatic 'Traminer' (Philipp et al., 2024), 'Arneis' wines showed a significant increase in some free terpenes in AR21 samples after bottling, particularly geraniol, even if the concentration was below detection threshold (Table 3). After alcoholic fermentation, the content of free geraniol also increased with increasing the CLS length, but the differences were not significant with respect to control (**Table S6**). Instead, free linalool content increased in CO07 samples for 'Cortese' PAF wines, achieving values over its odour threshold that remained after bottling (Table 3), although significant differences were not found among control and CLS treated samples in 'Cortese' PWI wines (Table 3). Contrarily to the effect observed on CLS musts (**Table S5**), the highest total concentration of glycosylated terpenes in the PAF wines was found in AR07 and AR14 samples for 'Arneis' while in CO21 samples for 'Cortese', although in the latter case the differences were not significant (**Table S7**). In 'Arneis' PAF wines, most of individual glycosylated terpenes followed the same trend of total ones while geraniol showed the opposite trend with the highest content being present in control. Regarding 'Cortese' PAF wines, the same trend was observed for the individual and total glycosylated terpenes, particularly for linalool and 8-hydroxylinalool.

Aleixandre-Tudo et al. (2015) found that pre-fermentative skin contact of crushed 'Chenin blanc' grapes at 4 °C for 12 h led to a decrease in terpene concentrations. This agrees with increased terpene concentrations in 'Muscat of Bornova' wines, reported after a short period of skin contact at 10 °C while longer periods of up to 12 h caused their decrease (Selli et al., 2006b). In the present study, the advantage of contact with the juice lees instead of grape skins is the possibility of increasing stabulation length without reducing but increasing the concentration of free terpenes in the final wines, as found particularly for 'Arneis'.

Free norisoprenoids were scarcely present (3-oxo-α-ionol was the only detected compound) and its concentration decreased significantly up to −47 % ($p < 0.001$) by CLS in 'Arneis' PAF wines, following this same behaviour after bottling, particularly for AR14 and AR21 samples (Fig. 1). The AR07 and AR14 samples had a higher concentration of glycosylated compounds, showing an inverse trend to that previously observed in the treated grape juices (Fig. 3). Instead, after an initial decrease for CO07 sample, an increased content of free 3-oxo-α-ionol was achieved for CO21 sample in 'Cortese' PAF wines ($p < 0.001$, +26 %, **Table S6**). This was in line with a higher content of precursors just after CLS (+30 %) with respect to control, **Table S5**), which were not significantly different in 'Cortese' PAF wines among CLS treated samples and control (**Table S7**). Nevertheless, this difference was not observed in 'Cortese' PWI wines (Table 3).

As regards total free benzenoids, the only difference observed among control and CLS treated samples was for AR07 sample in 'Arneis' PAF wines, showing a significantly lower content of them ($p < 0.05$, −19 %, Figs. 1–2). However, a higher quantity was found at AR21 samples for PWI wines. Significant differences with respect to control were not found in 'Cortese' PAF and PWI wines. For each variety, some individual free compounds increased significantly with stabulation, such as benzyl alcohol and methyl salicylate for 'Arneis', as well as vanillin for 'Cortese' at PAF (**Table S6**), but their concentrations are below their olfactory threshold in final wines (Table 3). Only some small differences in glycosylated benzenoids were observed in 'Arneis' and 'Cortese' wines after alcoholic fermentation when compared to the musts. Particularly for 'Arneis' PAF wines, the highest concentration of glycosylated precursors was found in AR07 samples while non-stabulated musts were richer in glycosylated benzenoids (**Tables S5 and S7**).

In 'Arneis' PAF and PWI wines a significant decrease was found for total content of free volatile phenols for all CLS samples ($p < 0.001$, −15-34 % with respect to control, Fig. 1). 4-vinylguaicol and 4-vinylphenol were the compounds most affected by the CLS technique, reporting a decrease of −25 % and − 29 %, respectively, in bottled 'Arneis' wines (Table 3). This may be relevant since the concentration of 4-vinylguaicol is at threshold level in this variety. Contrarily, in 'Cortese' PAF and PWI wines, no significant differences were found in both free and bound volatile phenols (Figs. 2 and 3). 'Arneis' PAF wines also showed no significant differences in total glycosylated volatile phenols or in main individual compounds (4-vinylguaicol and 4-vinylphenol). Selli et al., 2006b reported an increase in the volatile phenol concentration in the wines when increasing the length of pre-fermentative skin contact till 12 h. In the present study, the use of a lower temperature and juice lees contact allowed to extend the contact time without increasing the presence of these compounds.

3.3. Impact of CLS treatment on wine sensory characteristics and correlation with instrumental data

'Arneis' and 'Cortese' wines have been evaluated through sensory analysis at the end of alcoholic fermentation (PAF) and one month after bottling (PWI). Regarding the first tasting stage, in 'Arneis' no differences were perceived in terms of mouthfeel, whereas AR14 received a lower score in aroma intensity ($p < 0.05$) with respect to control (Table 4), which agreed with total free VOCs content (**Table S6**). As concerns the aromatic descriptors (Fig. 4), *grapefruit* (58.3 %), *jasmine, pear, and lemon* (all 41.7 %) perceptions were recognized in 'Arneis' control wine. In general, the *lemon* descriptor was found in all the samples (41.7–50 %). In contrast, *peach* was able to discriminate wines according to the Cochran's Q-test ($p = 0.053$, **Table S8**), with AR07 sample showing the highest frequency (50 %) in this wine group. In longer stabulated samples, *green apple* (67 % and 50 % for AR14 and AR21, respectively) and *rose* (41.7 % for AR14 and AR21) were recognized. The aroma descriptors used predominantly by judges for AR21 were *pear* (75 %), *lime* and *honey* (both 42 %) (Cochran's Q-test $p = 0.019$ for *honey*, **Table S8**).

At PAF, some significant differences were found in 'Cortese': CLS significantly increased the *body* attribute ($p < 0.001$) and an increasing trend for the *overall wine quality* ($p < 0.01$) was found (Table 4). At PWI polysaccharides content was investigated but neither 'Arneis' nor 'Cortese' wines reported significant differences among treatments (Tables 1 and 2). For the other mouthfeel and taste attributes, no statistical differences were found, indicating that the differences in polyphenolic content were negligible in terms of bitterness-astringency evaluation. Regarding the aromatic descriptors, *peach* (58.3 %), *green apple* (58.3 %), and *banana* (50 %) hints were underlined in control. The last two descriptors were well represented in all the tasted 'Cortese' PAF samples (frequency above 41 %). In contrast with 'Arneis', in 'Cortese' the *rose* descriptor was more frequent in control and CO07 with respect to the longer CLS samples, while *pear* (50 %) and *honey* (33.3–50 %) contributed mainly to CO14 and CO21 wines.

At PWI, for each variety, the differences among samples decreased. 'Arneis' or 'Cortese' wines did not differ for mouthfeel descriptors, aroma intensity, and overall wine quality when considering the CLS treatment (Table 4). 'Arneis' stabulated samples showed no difference in total free VOCs content (Table 3) with respect to the control in agreement with sensory analysis. However, the perceived descriptors changed when compared to PAF (Fig. 4). In control samples there was an increase in frequency of descriptors like *rose* and *honey* (both 50 %). The latter two descriptors were recognized also in AR07, with 50 % and 45 %, respectively. In fact, both descriptors were able to discriminate samples according with the Cochran's Q test ($p = 0.038$ and $p = 0.064$, for *rose* and *honey,* respectively) from longer stabulated samples. Also, in the AR07 sample, a contribution of some tropical fruits, particularly *pineapple* (45 %), was found. In terms of descriptors *green apple, pear,* and *honey* were more perceived in AR14 (40 %). *Green apple* was a descriptor more cited in AR14 and AR21 wines at both PAF and PWI when compared to control and AR07 samples.

In 'Cortese' samples, a significant difference persisted in *overall wine*

Table 4
Results of sensory Descriptive Analysis of wines mouthfeel (bitterness, astringency, acidity, body), aroma intensity, and overall wine quality after alcoholic fermentation (PAF) and one month after bottling (PWI) for Arneis (AR) and Cortese (CO) varieties.

Sample	Bitterness	Astringency	Acidity	Body	Aroma intensity	Overall wine quality
Arneis (PAF)[#]						
AR-Control	3.71 ± 0.54	1.75 ± 0.58	6.53 ± 0.43	5.17 ± 0.54	7.29 ± 0.31 a	6.27 ± 0.68
AR07	3.72 ± 0.62	1.90 ± 0.67	6.48 ± 0.37	4.10 ± 0.51	6.90 ± 0.31 ab	6.56 ± 0.33
AR14	3.84 ± 0.66	2.30 ± 0.74	6.31 ± 0.55	4.20 ± 0.43	6.01 ± 0.52 b	5.38 ± 0.45
AR21	2.73 ± 0.56	2.18 ± 0.67	6.13 ± 0.36	5.01 ± 0.60	7.29 ± 0.27 a	6.24 ± 0.66
p value	0.285	0.667	0.876	0.211	0.015	0.474
Sign.	ns	ns	ns	ns	*	ns
Arneis (PWI)						
AR-Control	3.38 ± 0.77	1.99 ± 0.91	4.76 ± 0.18	4.47 ± 0.57	6.34 ± 1.13	5.45 ± 1.96
AR07	3.58 ± 0.35	1.79 ± 0.53	5.02 ± 0.43	4.66 ± 0.61	7.03 ± 0.14	5.26 ± 0.82
AR14	3.39 ± 0.74	2.03 ± 0.07	4.34 ± 0.39	4.74 ± 0.08	6.04 ± 0.04	5.98 ± 0.36
AR21	3.16 ± 0.04	1.73 ± 0.65	4.99 ± 0.32	5.64 ± 0.35	6.35 ± 0.19	5.91 ± 1.33
p value	0.865	0.945	0.522	0.097	0.245	0.696
Sign	ns	ns	ns	ns	ns	ns
Cortese (PAF)[#]						
CO-Control	2.33 ± 0.51	2.13 ± 0.78	6.18 ± 0.42	3.02 ± 0.48 b	5.83 ± 0.67	5.03 ± 0.55 b
CO07	2.29 ± 0.61	2.08 ± 0.75	5.78 ± 0.53	4.88 ± 0.62 a	6.24 ± 0.53	6.71 ± 0.58 ab
CO14	2.84 ± 0.73	2.04 ± 0.59	5.93 ± 0.41	4.82 ± 0.54 a	6.97 ± 0.58	7.09 ± 0.53 a
CO21	3.47 ± 0.64	2.73 ± 0.68	6.33 ± 0.41	6.37 ± 0.44 a	6.93 ± 0.41	7.37 ± 0.51 a
p value	0.321	0.536	0.587	0.0002	0.225	0.007
Sign.	ns	ns	ns	***	*ns*	**
Cortese (PWI)						
CO-Control	2.99 ± 1.38	2.99 ± 0.14	6.26 ± 0.86	3.66 ± 0.43	5.21 ± 1.10	4.31 ± 0.85 b
CO07	2.39 ± 0.58	1.96 ± 0.80	6.59 ± 1.68	4.11 ± 0.71	6.03 ± 0.41	5.01 ± 1.63 ab
CO14	2.19 ± 0.21	2.91 ± 1.00	6.42 ± 0.82	3.63 ± 0.42	6.00 ± 0.13	6.50 ± 0.49 a
CO21	1.83 ± 0.12	2.23 ± 0.65	6.66 ± 0.41	4.24 ± 0.38	6.29 ± 0.42	6.13 ± 0.11 a
p value	0.255	0.233	0.893	0.618	0.334	0.003
Sign	ns	ns	ns	ns	ns	**

[#] For samples after fermentation two-way ANOVA was performed with sample as fix effect and judges as random effect, and if statistical significance was found ($p < 0.05$), Tukey HSD was used for establishing significant differences among samples. Results are expressed as mean ± s/(n)$^{1/2}$, standard deviation; n, number of panellists) for PAF, whereas for PWI are expressed ad mean ± standard deviation of two independent sensory analysis sessions. "CO-Control", "CO07", "CO14" and "CO21" indicate the non-stabulated and the three treatment periods, 7, 14 and 21 days, respectively, for 'Cortese' wines and "AR- Control", "AR07", "AR14" and "AR21" indicate the non-stabulated and the three treatment periods, 7, 14 and 21 days, respectively, for 'Arneis' wines.

quality, where CO14 and CO21 were preferred than control after bottling ($p < 0.01$; Table 4). At PWI, the most used aroma descriptors for 'Cortese' were *green apple*, *pear*, *rose,* and *jasmine*. Particularly, *green apple* descriptor was more used for control (50 %) and CO21 (57 %), *pear* in CO14 and CO21 (50–57 %), as well as *rose* (57.1 % for CO14 and 42.9 % for CO21). Furthermore, *jasmine* was used in the 35.7 % of cases to describe the control and by 42.9 % for CO14. Nevertheless, the total free VOCs of 'Cortese' wines had no significant difference among samples (Table 3), confirming the sensory results that showed no perceived differences in aroma intensity at wine tasting.

Although in this case the treatment did not affect neither the total concentration of VOCs nor the overall aroma intensity, some individual VOCs may have influenced the final *overall wine quality*. With this aim, a PCA was performed considering, for both varieties studied, the results of the wines after bottling (Fig. 5). The first dimension accounted for the 41.6 % of the explained variance whereas the second dimension for the 22.7 %, with a total of 64.3 %. Some trends can be underlined: control and short CLS (AR07 and CO07) samples are in the upper side of the graph, whereas the longest CLS samples (AR14, CO14 and AR21, CO21) are in the lower. The first dimension was positively correlated with four esters: ethyl 3-hydroxybutanoate, ethyl octanoate, isoamyl acetate, and hexyl acetate ($R = 0.938$, 0.920, 0.910, and 0.906, respectively, all $p < 0.01$), instead it was negatively correlated with geraniol ($R = -0.765$) and ethyl phenyllactate ($R = -0.770$), both $p < 0.05$. In fact, 'Arneis' esters were lower in AR14 and AR21 compared to control and AR07 wines ($p < 0.01$). The presence of compounds, such as isoamyl acetate, 2-phenylethyl acetate, hexyl acetate or ethyl 3-hydroxybutanoate, in control and 7-day CLS samples conferred to those wines fruity and floral characters, like *pineapple*, *banana*, and *rose*, which were recognized by the judges during sensory analysis. In contrast, 14 and 21-day CLS samples were found in the opposite side of the graph, being characterised by significant higher contents of isoamyl alcohol and 2-phenylethanol, and in general, in higher alcohols. Overall, these two last compounds may have been related to *jasmine* aroma descriptor and, more generally, to the floral- sensation perceived in these wines. In addition, in 'Arneis' wines, geraniol content was significantly higher in AR21, although lower than its detection threshold.

Dimension 2 was positively correlated ($p < 0.05$) mainly with benzyl alcohol, 3-methyl-1-pentanol, diethyl malate, homovanillic acid, and diethyl succinate ($R > 0.8$), and negatively with 1-hexanol ($R = -0.753$, $p < 0.05$). C6-compounds may have differentiated the 'Cortese' wines by the CLS treatment, with CO14 and CO21 samples resulting in a lower content of this chemical class responsible for herbaceous hint. This could be linked to the higher *overall wine quality* scores (Table 4). Moreover, the *rose* and *jasmine* aroma descriptors, mostly perceived in CO14 samples (Fig. 4, **Table S8**), were associated with higher contents of linalool, even though not significantly, above its odour threshold. The similarity among the control and AR07 samples for 'Arneis' with the longer stabulated samples for 'Cortese' (CO14 and CO21) found in the PCA is mainly due to the opposite behaviour of volatile phenols (mainly, 4-vinylphenol and 4-vinylguaiacol) in the two varieties.

4. Conclusions

The cold liquid stabulation (CLS) technique gave contrasting results in the winemaking of 'Arneis' and 'Cortese' in terms of polyphenolic and

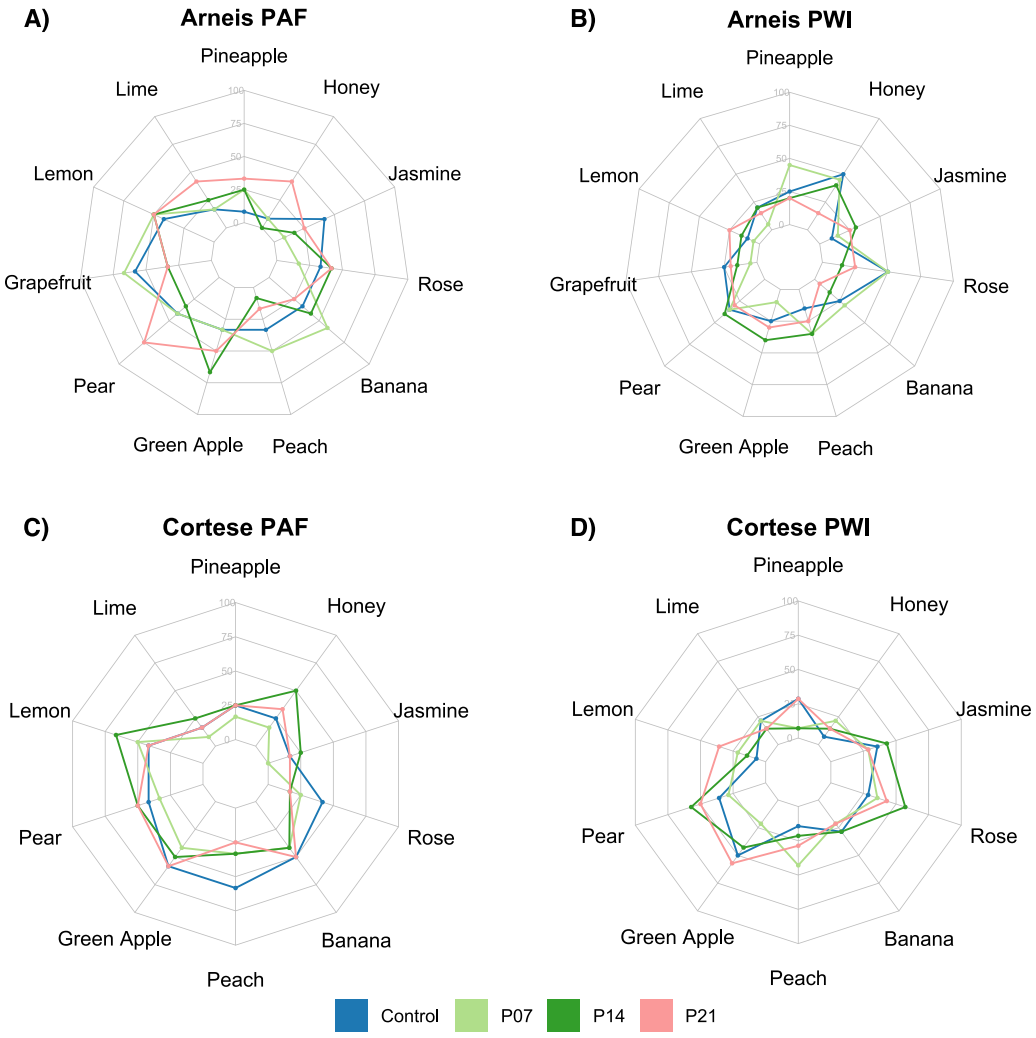

Fig. 4. Sensory analysis results (percentage frequency) of aroma descriptors evaluated by Check-All-That-Apply method after the alcoholic fermentation (PAF) and one month after bottling (PWI). "Control", "P07", "P14" and "P21" indicate the non-stabulated and the three treatment lengths 7, 14 and 21 days, respectively, for A) 'Arneis' after alcoholic fermentation, B) 'Arneis' one month after bottling, C) 'Cortese' after alcoholic fermentation, and D) 'Cortese' one month after bottling.

aroma composition. Some chemical-physical parameters, such as total acidity, pH, and colour parameters of wines after one month of bottling changed similarly in the two varieties according to treatments, without affecting the related sensory features of mouthfeel and colour.

A major role of the variety on the effect of this oenological technique was highlighted for secondary metabolites. TPI increased in stabulated 'Arneis', while it decreased in 'Cortese', but these differences were limited and not perceivable by wine sensory analysis. Regarding VOCs, 'Arneis' non-treated control and a short CLS treatment (7 days) led to a higher accumulation of esters in wines whereas longer CLS (14 and 21 days) produced a major quantity of higher alcohols, and geraniol and benzenoids for the longest treatment. Instead, in 'Cortese', 14 and 21-day CLS led to wines with less C6-compounds but higher linalool after 14 days of CLS. Cortese overall wine quality rating increased in stabulated samples for 14 and 21 days.

This technique may be worth to be considered when starting from healthy white grapes but involves increased energy costs (due to refrigeration), and a continuous process control is necessary. Future research may concern the linkage between these results and the grape composition, as well an in-depth characterization of the solid residue – grape lees or *bourbes* – in terms of nitrogen-containing compounds, lipids, and polysaccharides, in connection with their extraction-adsorption phenomena.

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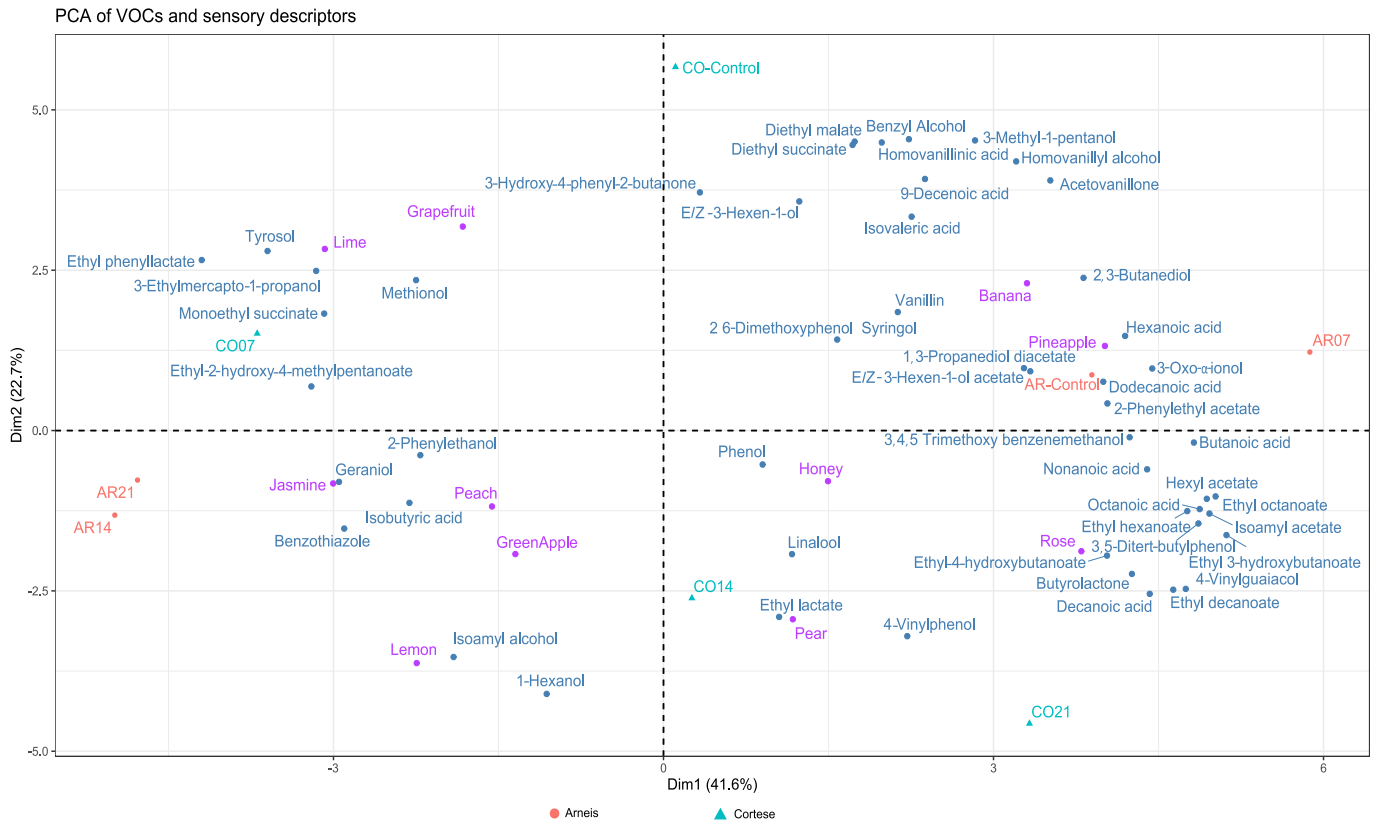

Fig. 5. Principal Component Analysis of individual significant free VOCs (active variables, reported the 25 with higher contribution) and aroma sensory descriptors (supplementary variables). "CO-Control", "CO07", "CO14" and "CO21" indicate the non-stabulated and the three treatment lengths 7, 14 and 21 days, respectively, for 'Cortese' wines and "AR- Control", "AR07", "AR14" and "AR21" indicate the non-stabulated and the three treatment lengths 7, 14 and 21 days, respectively, for 'Arneis' wines.

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.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2024.142058.

Data availability

Data will be made available on request.

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