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ORIGINAL PAPER

Effect of oxidation on color parameters, tannins, and sensory characteristics of Sangiovese wines

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Abstract

The oxidation of two premium wines, Chianti Classico and Brunello di Montalcino, obtained from Sangiovese grapes in 2017 and 2018 vintages, is simulated through oxygen exposure by applying three consecutive saturation/consumption cycles. After each oxidation cycle, wines are analyzed for color intensity, hue, CIElab coordinates, polymeric pigments, monomeric anthocyanins, acetaldehyde, tannins, and flavans and compared to control wines. By increasing the oxygen supply, monomeric anthocyanins decrease faster in the younger wines, acetaldehyde is highly produced in the older ones, while the formation of polymeric pigments depends on the wine type. The color intensity, the yellow and blue tint increase in all wines. The effect of oxidation on main phenolic compounds is more affected by the wine age than the type. Oxidation also significantly affects wine sensory characteristics: the astringency intensity decreases, whereas the subqualities of silk and velvet increase. The percentage increase of the silky sensation is from four to sevenfold higher in young wines. The silkiness is correlated with some anthocyanins decrease [malvidin 3-(6^{II}-acetyl)-monoglucoside, delphinidin 3-monoglucoside, petunidin 3-monoglucoside, malvidin 3-monoglucoside]; the velvet sensation with polymeric pigments formation, acetaldehyde production, and malvidin 3-(6^{II}-acetyl)-monoglucoside decrease. Moreover, after oxidation, aged wines are characterized by an enhanced balsamic odor and aroma.

Keywords Oxidation · Polymeric pigments · Color · Astringency · Subquality · Aging

Introduction

Some high-quality red wines undergo a period of aging, more or less long, depending on the wine type and market. Sangiovese grape variety is at the basis of renowned Italian wines such as Chianti Classico (produced in the provinces of Siena and Florence, Tuscany) and Brunello di Montalcino (produced in the province of Siena, Tuscany), and represents a percentage between 80 and 100% for Chianti Classico and 100% for Brunello di Montalcino. These wines must follow an aging period of a minimum of 24 months before their commercialization. In Chianti Classico's case, the wine can be sold only 2 years after the winemaking, for the "Riserva" specification (http://www.chianticlassico.com). Whereas for Brunello di Montalcino wine, it takes 5 years to go on the market (http://www.consorziobrunellodimontalcino.it). A period of barrel and bottle aging is necessary depending on the production disciplinary for the Chianti Classico DOCG (Denominazione di Origine Controllata e Garantita) and Brunello di Montalcino DOCG wines. Such premium Sangiovese wines can age for a long time. However, Sangiovese-based wines show a sensitive pigment profile due to a high percentage of unstable dihydroxy pigments [1]. Therefore, before deciding on the Sangiovese wines market position, it is advantageous to use tests to simulate wine aging to avoid a detrimental evolution over time. One of the most important phenomena occurring during wine aging is oxidation. Artificial Neural Networks models have been used to predict the aging potential using wines' oxygen consumption rates [2]. Several studies also showed that consecutive

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oxygen saturation and consumption cycles constitute a suitable approach to simulate wine aging under moderate oxygen exposure. Oxygen is readily consumed during the first saturation cycles, and the average oxygen consumption rate (OCR) depends on phenolic compounds, free SO_2, pH, metals traces, and acetaldehyde production [3–5]. In particular, the higher is the flavans/monomeric anthocyanins and tannins/anthocyanins ratio in wines, obtained by increasing marc pressing after maceration, the faster is the OCRs of the wines [5]. Saturation occurs when around 6–7 mg oxygen/L is supplied to wine in each cycle, but the speed of consumption depends on the wine typology [6, 7]. A total amount of oxygen from 7 to 53 mg/L has been provided to Spanish and Italian wines to simulate the aging conditions when the wine passes from the winery to the market [4, 8–11]. The degree of oxidation obtained by supplying the wine with different oxygen concentrations can be associated with the barrel's annual oxygen entry from 2 to 45 mL/L/year, varying the barrel and trial conditions [12].

Concerning the effect of oxygen on wine evolution, several reports show that a moderate exposure of red wines to oxygen can enhance color through the formation of polymeric pigments [5, 10, 13], reduce astringency for variations in tannins content and polymerization degree [14, 15], change the mouthfeel sensations towards positive attributes [16], and improve aroma increasing fruity notes and decreasing vegetal characteristics [17]. On the other hand, high oxygen levels can be detrimental to wine color and sensory characteristics. Oxygen induces phenolic oxidation and polymerization of quinones contributing a yellow–brown color to wines [18]. The red color of the wine decreases while increasing its yellow tint due to a decrease in the concentration of the flavylium cations and increased xanthylium pigments present in wines, formed from the conversion of glyoxylic acid into a xanthylium structure with flavanol units [19]. Overall astringency can also increase depending on the newly formed compounds by oxidation [10, 20]. Regarding mouthfeel, the hard and puckering astringency subqualities can characterize oxidated wines [16, 21, 22]. Besides, the formation of oxidative off-odors [23] such as sensory-detected acetaldehyde [24] and heterocyclic acetals [25] occurs.

Although it is widely studied and considering the great complexity of the wine, the evolution during moderate oxygen exposure and the effect on sensory wine attributes is still a complex and not well-understood phenomenon. In this scenario, the oxygen supply can be crucial for the correct red wine evolution over time. Moreover, the maximum oxygen uptake inducing positive attributes for each wine is difficult to individuate even within the same variety. For example, two Sangiovese-based wines as Chianti Classico and Brunello di Montalcino belong to two different denominations, which provide different aging periods and marketing timing. Generally, these wines are highly rich in phenolics to guarantee long aging. However, aging does not necessarily guarantee the quality of the wine. For Sangiovese and other wine types, the saturation tests can thus provide information on the possible evolution of wine during aging and avoid wrong product commercialization.

In this work, we evaluate the effect of moderate oxidation on the color and sensory characteristics of 100% Sangiovese wines. Chianti Classico (CC) and Brunello di Montalcino (BM) wines from two consecutive vintages (2017 and 2018) are saturated with a total amount of oxygen of around 18 mg/L, as the result of three cycles of oxygen saturation, and the variation between not-oxidated wines (zero time) and wines after each saturation cycle in color intensity, hue, CIElab coordinates, polymeric pigments, acetaldehyde, monomeric anthocyanins, tannins, flavans, and total phenolics is evaluated. Regarding the sensory analysis, differences in astringency intensity, astringency subqualities, odor, and aroma of Sangiovese wines are considered between the zero time and the last saturation cycle.

Material and methods

Experimental wines

Sangiovese wines (100%) are produced in two wineries located in the province of Siena (Tuscany, Italy) during the vintage 2017 and 2018. The vinification is based on the following protocol: grapes (18 tons) from vineyards located in the DOCG area of Chianti Classico (CC) and Brunello di Montalcino (BM) wines are destemmed and crushed; the must is treated with potassium metabisulfite (40 mg/kg) and inoculated with 20 g/hL of yeast (F83 Laffort, Bordeaux, France); the fermentation/maceration lasted 12 days at 25 °C, during which yeast assimilable nitrogen (YAN), in the form of diammonium phosphate (containing ≈ 0.12% of thiamine hydrochloride), is added with the inoculum and then again on the third and sixth day of fermentation to a total concentration of 30 g/hL. The wines are devatted and transferred to 53 L carboys. After the addition of pectolytic enzymes (3 g/hL), wines are inoculated with lactic bacteria (LF16 Direct, Laffort) at 1 g/hL. Potassium metabisulfite (6 g/hL) is then added to the wines (CC-17, CC-18, BM-17, BM-18), which are subsequently fulfilled with N_2, sealed, and shipped in 20 L to the Division of Sciences of Vine and Wine, Department of Agriculture, University of Naples Federico II, in Avellino (Italy). The wines are then racked and transferred into hermetic stainless-steel kegs (15 L) and stored under nitrogen at cellar temperatures (12–18 °C). Before the saturation experiments in August 2019, wines are filtered with 0.45 µm filters to avoid microbiological spoilage consuming oxygen. A total amount of around 18 mg

oxygen/L is provided to the Sangiovese wines, as described below. This oxygen amount can be associated with an aging period of 1 year in new barrels of Limousin (wild grain) [12].

Saturation cycles

The oxidation test consists of three consecutive air saturation cycles (sat1-sat2-sat3). Zero time represents the not-oxidated wine. The procedure used is the same as [5]. Briefly: two 1 L bottles of each wine containing PSt3 oxygen sensors (Nomacorc SA, Thimister-Clermont, Belgium) are saturated with air by adding a gentle flow of oxygen throw a mini-compressor for 15 min until the oxygen level of the wine reached 6.6 mg/L. The oxygen for the second and the third cycle is infused after the oxygen levels are dropped. The oxidation cycle is considered finished once O_2 levels dropped to 10% of the initial concentration. Wines are stored in an incubator in the dark at 25 °C, and dissolved oxygen level is monitored at least once a day with a Nomasense oxygen analyzer from Nomacorc S.A. (Thimister-Clermont, Belgium).

Spectrophotometric analyses

Chromatic characteristics and spectrophotometric measures are determined using a spectrophotometer (Jenway 7305 Spectrophotometer). Color intensity (CI), Abs 420, Abs 520, Abs 620 nm, hue, and CIElab coordinates are evaluated according to the OIV methods [26]. Polymeric pigments, total phenolics, and bovine serum albumin (SIGMA Life Science, USA) BSA reactive tannins are determined by the Harbertson-Adams assay [27]. To determine vanillin reactive flavans (VRF), the method described by Di Stefano and Guidoni [28] is scaled-down, and volumes are adjusted to decrease the consumption of organic solvents. All analyses are conducted through two experimental replicas and two analytical replicas.

High-performance liquid chromatography analyses of acetaldehyde

Acetaldehyde is analyzed using the official OIV method [26]. Briefly, wine sample aliquots (100 µL) are dispensed to a vial, followed by the addition of 20 µL of freshly prepared 1120 mg/L SO_2 solution. Next, 20 µL of 25% sulfuric acid (Carlo Erba reagent 96%) is added, followed by 140 µL of 2 g/L 2,4-dinitrophenylhydrazine reagent (Aldrich chemistry). After mixing, the solution is allowed to react for 15 min at 65 °C and then promptly cooled to room temperature. Analysis of carbonyl hydrazones is conducted by HPLC (HPLC Shimadzu LC10 ADVP apparatus (Shimadzu Italy, Milan), consisting of a SCL-10AVP system controller, two LC-10ADVP pumps, a SPD-M 10 AVP detector, and an injection system full Rheodyne model 7725 (Rheodyne, Cotati, CA) equipped with a 50 µL loop. A Waters Spherisorb column (250 × 4.6 mm, 4 µm particles diameter) is used for separation. Eluted peaks are compared with derivatized acetaldehyde standard. All analyses are conducted through two experimental replicas and two analytical replicas.

High-performance liquid chromatography analyses of anthocyanins

The separation of anthocyanins is carried out according to the OIV Compendium of International Methods of Analysis of Wine and Musts [26]. Analyses are performed in an HPLC Shimadzu LC10 ADVP apparatus (Shimadzu Italy, Milan), consisting of an SCL-10AVP system controller, two LC-10ADVP pumps, an SPD-M 10 AVP detector, and an injection system full Rheodyne model 7725 (Rheodyne, Cotati, CA) equipped with a 50 µL loop. A Waters Spherisorb column (250 × 4.6 mm, 4 µm particles diameter) with pre-column was used. Fifty µL of wine or calibration standards are injected onto the column. For calibration, the external standard method is used: the calibration curve was plotted for the malvidin-3-monoglucoside (Extrasynthese, Lyon, France) on the basis of peak area, and the concentration is expressed as mg/L of malvidin-3-monoglucoside equivalents. All analyses are conducted through two experimental replicas and two analytical replicas.

Sensory evaluation of wines

The sensory panel from the Division of Sciences of Vine and Wine, Department of Agriculture, University of Naples Federico II, in Avellino (Italy), with a long experience in wine evaluation and trained for mouthfeel sensations [21], is composed by13 trained assessors (comprising five women between the age of 35–50 and 8 men between the age of 25–44 years). They have been previously trained trough six stages: (i) a selection stage, during which solutions of sucrose (10.0 g/L for sweetness), tartaric acid (1.0 g/L for acidity), caffeine (1.0 g/L for bitterness), and tannic acid (2.0 g/L for astringency) are presented in water and white wine; (ii) a taste recognition stage, during which solutions of sucrose (5.0 g/L for sweetness), tartaric acid (0.8 g/L for acidity), caffeine (0.5 g/L for bitterness) and tannic acid (1.0 g/L for astringency) are presented in water, white and red wine; (iii) a binary-mixtures stage, in which mixed solutions are presented in white wine at lower concentration; (iv) a rating stage, in which scaling solutions of caffeine (0.1–0.8 g/L) and five enological tannins (0.2–1.5 g/L) are presented in water and white wine; (v) a subqualities familiarization phase, during which the panel familiarized with astringency terms and selected the most appropriate

descriptors to use in the check-all-that-apply (CATA) questionnaire (Supplementary Table 1); and (vi) a training stage for subqualities, during which the panel tested six commercial red wines spiked with five enological tannins (from 0.2 to 0.5 g/L), and used the CATA questionnaire in association with touch standards as described in [21, 29]. During the subsequent tasting sessions, the panel selected and rated the sensations they perceived (rate-all-all-that-apply, RATA) using the same list of 16 astringency subqualities (silk, velvet, dry, corduroy, adhesive, aggressive, hard, soft, mouthcoat, rich, full-body, green, grainy, satin, pucker, persistent) (Supplementary Table 1) and a 0–7 points scale with end-point anchors 1 = 'slightly applicable' and 7 = 'very applicable'.

In this way, the panel evaluates the control (zero time) and oxidated Sangiovese wines (after sat3) in duplicate. In each session, two tasting brackets of four anonymous samples are performed. They are presented in balanced, random order at a temperature of 18 ± 2 °C, in black tulip-shaped glasses coded with 3-digit random numbers. The panel is instructed to pour the whole sample in their mouth, hold it for 8 s, expectorate, and evaluate the astringency intensity. Then, they select and rate the perceived astringency subqualities. The order in which the RATA terms are presented is different for each wine and each assessor, in accordance with a Williams Latin Square experimental design. Judges wait for 2 min before rinsing with mineral water for 10 s twice and then wait at least 30 s before the following sample. The panel also evaluates the intensity of fruity, floral, herbaceous, spicy, balsamic descriptors relating to odor (the olfactory sensation felt directly by the nose) and aroma (the olfactory sensation felt retronasally upon in the mouth) of wines using a 0–5 points scale.

Statistical analysis

Analytical data are compared using Fisher's least significant differences (LSD) procedure between samples and saturation cycles when the variance resulted homogeneous over four replicates ($p < 0.05$). The same test is applied to sensory data (intensity and RATA) over two replicates. The effect of oxidation is evaluated by the analysis of the variance (ANOVA) and Fisher's LSD test on sensory characteristics (astringency, subquality, odor, and aroma) between control (zero time) and oxidated Sangiovese wines (sat3), with a confidence level of $p < 0.05$. Pearson's correlation was performed between sensory (RATA) and analytical data with $p < 0.05$. Analyses are carried out by the XLSTAT software package (Addinsoft, XLSTAT 2021, Paris, France).

Results and discussion

In this work, we perform three consecutive cycles of oxygen saturation and consumption on two Sangiovese wines from two successive vintages (CC-17, BM-17, CC-18, BM-18) to simulate wine aging under moderate oxygen exposure. The oxygen consumption rates for all wines at the beginning of the first saturation cycle are the highest in the cycles (Fig. 1), in agreement with previous studies [3–5, 30].

This behavior is probably due to the chemical structure of the compounds formed following the consumption of

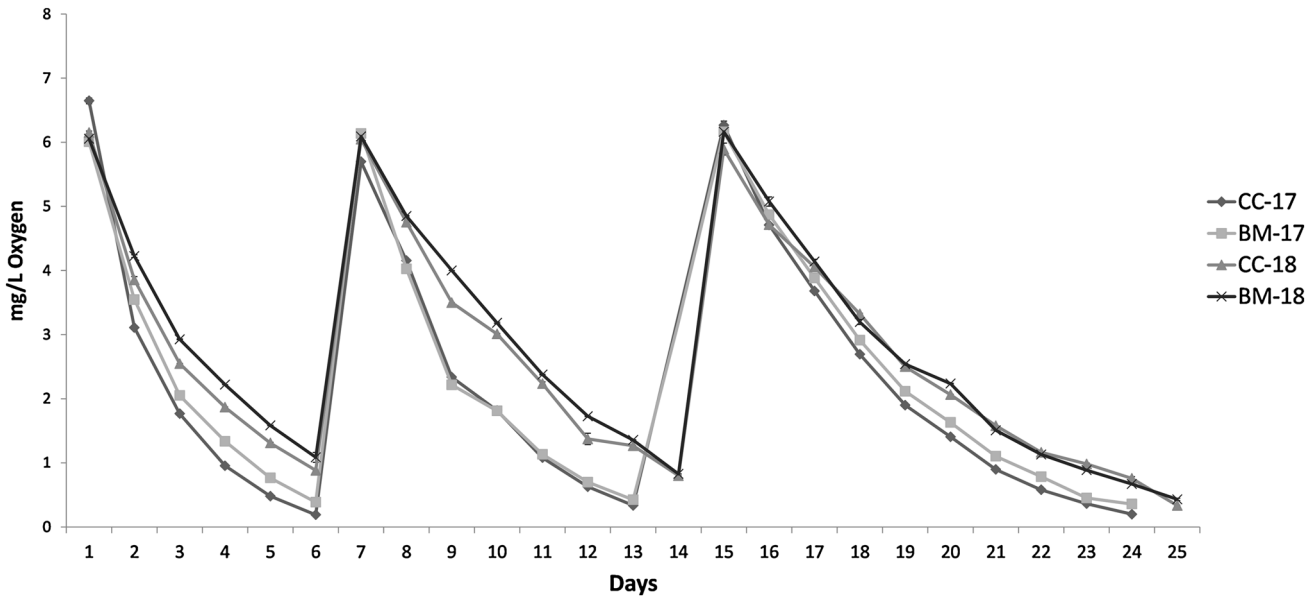

Fig. 1 Average oxygen concentrations measured in each wine sample throughout the experiment

oxygen after the first saturation cycle. This hypothesis can be easily supported by the fact that when oxidation is triggered in a solution containing grape proanthocyanidins, intermolecular and intramolecular rearrangements can create new bonds, and the newly formed structures can probably show a decreased reactivity owing to the lower presence of active sites on the molecule [31]. Previous studies on red wines with added tannins [9] also show that the oxygen consumption related to the first saturation cycle determines the production of new compounds with a lower capacity to react in the oxidation cascade subsequent cycles. Figure 1 shows a significant difference in oxygen consumption between 2017 and 2018 Sangiovese wines, being the younger wines slower in consuming oxygen. This result is not a surprise because young wines are richer in antioxidants, especially monomeric anthocyanins (Fig. 2a), which negatively correlate to OCR [5].

Pigments and chromatic characteristics variation

After oxygen saturation, as expected, a loss of monomeric anthocyanins and a concomitant production of new pigments are detected for all wines. In Fig. 2, the concentration of monomeric anthocyanins (Fig. 2a) and polymeric pigments (Fig. 2b) in not-oxidated wines (zero time) and each saturation cycle (sat1-2-3) is shown. The richer the wine is in anthocyanins, the greater their loss after the three saturation cycles (− 29%, − 35%, − 38%, and − 38%, respectively, for CC-17, BM-17, CC-18, and BM-18). The decline of anthocyanins is due to the several reactions triggered by oxygen; some are degradative [32, 33], and others can generate new pigmented structures [13]. Mild-oxidative conditions promoted the loss of monomeric anthocyanins and the formation of colored pigments also in aged micro-oxygenated wines [34, 35]. In Fig. 2b, the concomitant increase of polymeric pigments seems correlated to

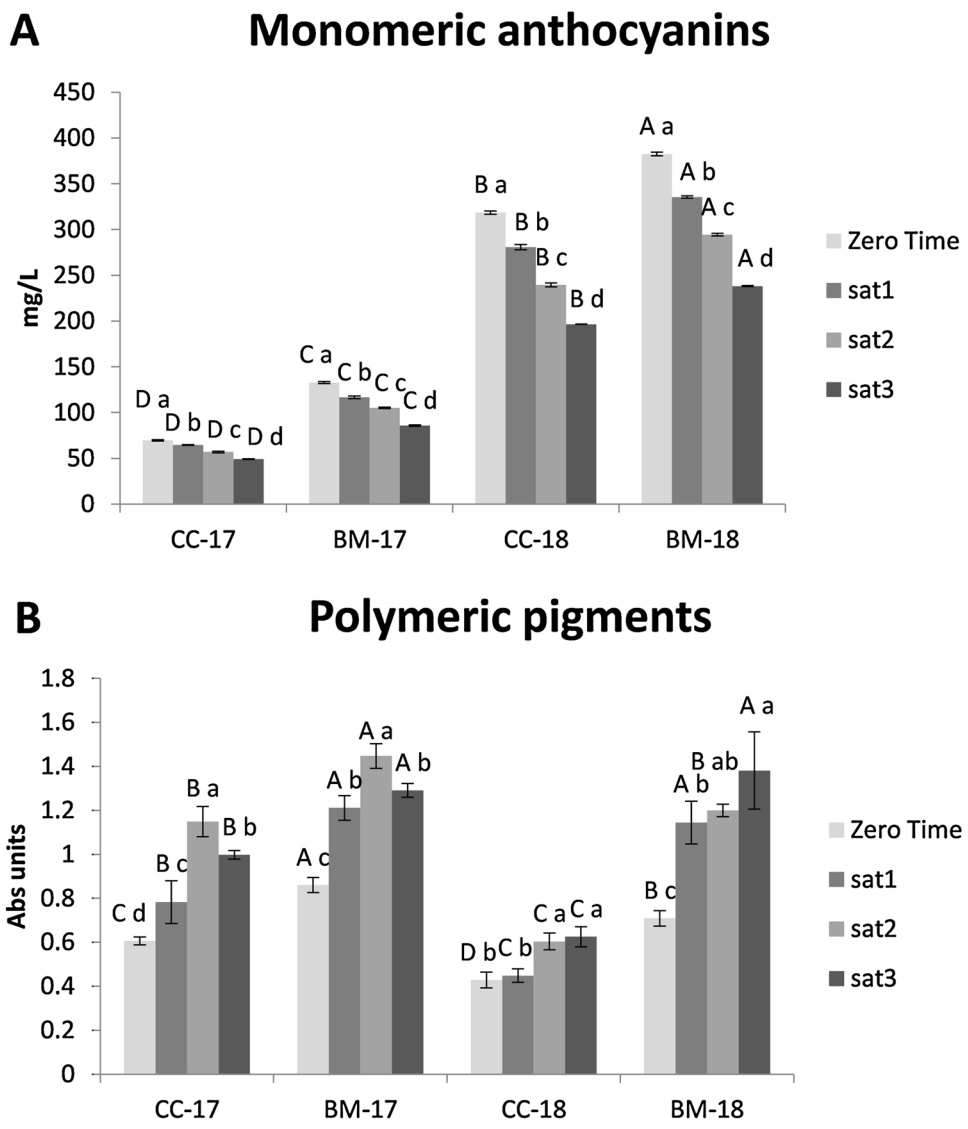

Fig. 2 Total monomeric anthocyanins **a** and polymeric pigments **b** of Sangiovese wines before (zero time) and after each saturation cycle. Histograms with different letters differ according to Fisher LSD analysis ($p < 0.05$). Capital letters refer to differences between wines; lowercase letters refer to saturation cycles

anthocyanins' loss until the second saturation (sat2) and with less evidence in 2018 wines. The formation of polymeric pigments is higher in BM than CC, independently from the vintage. Several factors such as the anthocyanins/tannins ratio, the presence of acetaldehyde and other reactive carbonyls and phenolic acids [13, 14, 36, 37] may determine a different reactivity of monomeric anthocyanins in forming the new polymeric pigments. Concerning the individual monomeric anthocyanins (Table 1), the percentage of loss for all wines follows the trend: malvidin 3-(6^{II}-acetyl)-monoglucoside (Mal-Ac) > delphinidin 3-monoglucoside (Del-3 mg) > petunidin 3-monoglucoside (Pet-3 mg) > malvidin 3-monoglucoside (Mal-3 mg). In all wines, significant losses of anthocyanins are detected after each saturation cycle. During aging, the acylated anthocyanins are quickly degraded in agreement with previous results [37–39]. Concerning the not acylated monomeric anthocyanins, their resistance to oxygen injury depends on the B ring substitution pattern: the more hydroxylation (Pet-3 mg and Del-3 mg), the higher degradation during oxidation.

Changes in pigment structures determine a variation in chromatic wine characteristics (Table 2 and Table 3). After the oxidation cycles, an increase of CI (from 12 to 23%) and hue (from 13 to 19%) is detected as a consequence of a rise in the yellow tint (Abs 420 nm) from 23 to 32%, and of the blue tint (Abs 620 nm) from 19 to 45%. The increase in the colorant intensity and 420 nm absorbance values has also been observed in wines aged after micro-oxygenation treatments and was attributed to the formation of pyranoanthocyanins [35]. Also, for chromatic characteristics, the most significant changes are detected in 2018 Sangiovese wines. Similar variations were observed in red wines after long aging; in particular, a hue increase of about 19% was

Table 1 Monomeric anthocyanins of Sangiovese wines before and after each saturation cycle

	Zero time		sat1	sat2	sat3	[b]Final decrease (%)
CC-17[a]	Del-3 mg	10.11 ± 0.20 D a	9.00 ± 0.11 D b	7.16 ± 0.05 D c	5.32 ± 0.09 D d	− 47
	Cya-3 mg	7.60 ± 0.20 D a	7.79 ± 0.15 D a	7.12 ± 0.08 D b	6.04 ± 0.12 D c	− 21
	Pet-3 mg	12.22 ± 0.21 D a	11.25 ± 0.42 D b	9.92 ± 0.05 D c	8.04 ± 0.10 D d	− 34
	Peo-3 mg	6.70 ± 0.14 D a	6.29 ± 0.06 D b	5.44 ± 0.07 D c	5.47 ± 0.07 D c	− 18
	Mal-3 mg	32.22 ± 0.27 D a	29.40 ± 0.23 D b	27.03 ± 0.69 D c	24.10 ± 0.14 D d	− 25
	Mal-Ac	0.93 ± 0.13 C a	0.88 ± 0.16 C a	0.24 ± 0.06 D b	0.28 ± 0.10 D b	− 70
BM-17	Del-3 mg	19.96 ± 0.58 C a	17.11 ± 0.25 C b	14.31 ± 0.18 C c	10.42 ± 0.07 C d	− 48
	Cya-3 mg	14.10 ± 0.06 C a	13.34 ± 0.15 C b	12.19 ± 0.11 C c	10.10 ± 0.07 C d	− 28
	Pet-3 mg	23.65 ± 0.29 C a	21.67 ± 0.35 C b	18.92 ± 0.24 C c	14.93 ± 0.05 C d	− 37
	Peo-3 mg	11.43 ± 0.20 C a	10.37 ± 0.20 C b	9.25 ± 0.06 C c	8.49 ± 0.12 C d	− 26
	Mal-3 mg	62.74 ± 0.53 C a	53.29 ± 0.58 C b	49.89 ± 0.39 C c	41.30 ± 0.80 C d	− 34
	Mal-Ac	1.06 ± 0.11 C a	0.91 ± 0.03 C b	0.54 ± 0.10 C c	0.53 ± 0.10 C c	− 50
CC-18	Del-3 mg	29.17 ± 0.44 B a	25.41 ± 0.28 B b	20.76 ± 0.17 B c	15.60 ± 0.21 B d	− 47
	Cya-3 mg	16.16 ± 0.45 B a	16.11 ± 0.16 B a	14.64 ± 0.21 B b	12.41 ± 0.03 B d	− 23
	Pet-3 mg	49.60 ± 0.26 B a	45.01 ± 0.15 B b	38.02 ± 0.47 B c	30.25 ± 0.03 B d	− 39
	Peo-3 mg	29.42 ± 0.35 B a	28.18 ± 1.01 B b	23.09 ± 0.26 B c	20.37 ± 0.07 B d	− 31
	Mal-3 mg	191.39 ± 0.49 B a	163.73 ± 1.58 B b	141.02 ± 1.25 B c	116.45 ± 0.10 B d	− 39
	Mal-Ac	2.88 ± 0.32 B a	2.31 ± 0.17 B b	1.88 ± 0.22 B c	1.39 ± 0.08 B d	− 52
BM-18	Del-3 mg	43.20 ± 0.20 A a	36.41 ± 0.22 A b	31.19 ± 0.22 A c	23.61 ± 0.28 A d	− 45
	Cya-3 mg	31.98 ± 0.32 A a	30.13 ± 0.21 A b	28.11 ± 0.07 A c	23.08 ± 0.14 A d	− 28
	Pet-3 mg	63.32 ± 0.97 A a	57.50 ± 0.38 A b	49.61 ± 0.26 A c	38.92 ± 0.15 A d	− 39
	Peo-3 mg	41.89 ± 0.41 A a	37.02 ± 0.12 A b	32.61 ± 0.25 A c	28.18 ± 0.02 A d	− 33
	Mal-3 mg	198.56 ± 0.61 A a	170.94 ± 1.39 A b	150.31 ± 1.00 A c	122.48 ± 0.11 A d	− 38
	Mal-Ac	3.62 ± 0.25 A a	3.40 ± 0.18 A a	2.54 ± 0.19 A b	1.89 ± 0.25 A c	− 48

Different capital letters indicate a statistically significant difference between samples within the same saturation cycle; lowercase letters indicate a statistically significant difference between saturation cycles, according to the Fisher LSD test ($p < 0.05$)

Del-3 mg delphinidin 3-monoglucoside, *Cya-3 mg* cyanidin 3-monoglucoside, *Pet-3 mg* petunidin 3-monoglucoside, *Peo-3 mg* peonidin 3-monoglucoside, *Mal-3 mg* malvidin 3-monoglucoside, *Mal-Ac* malvidin 3-(6^{II}-acetyl)-monoglucoside

[a]The values represent the mean ± standard deviation over four replicates

[b]Final decrease represents the mean reduction percentage between time zero and sat3

Table 2 Color parameter values of the Sangiovese wines subjected to three saturation cycles

	Zero time[a]			
	CC-17	BM-17	CC-18	BM-18
Abs 420 nm	2.67 ± 0.02 B d	2.72 ± 0.02 A c	2.51 ± 0.04 C d	2.48 ± 0.01 C d
Abs 520 nm	3.63 ± 0.03 A c	3.40 ± 0.01 C b	3.56 ± 0.03 B b	3.61 ± 0.02 A b
Abs 620 nm	0.64 ± 0.01 A b	0.57 ± 0.01 B b	0.50 ± 0.02 C c	0.47 ± 0.01 D c
CI	6.93 ± 0.06 A b	6.69 ± 0.03 B c	6.57 ± 0.08 C c	6.55 ± 0.04 C c
Hue	0.74 ± 0.00 B d	0.80 ± 0.01 A d	0.71 ± 0.00 C d	0.69 ± 0.00D d
	sat1			
	CC-17	BM-17	CC-18	BM-18
Abs 420 nm	3.06 ± 0.02 B c	3.22 ± 0.07 A b	2.90 ± 0.10 C c	2.84 ± 0.06 C c
Abs 520 nm	3.84 ± 0.02 BC a	3.75 ± 0.08 C a	3.90 ± 0.13 AB a	4.00 ± 0.06 A a
Abs 620 nm	0.77 ± 0.02 A a	0.74 ± 0.02 A a	0.65 ± 0.03 B b	0.63 ± 0.01 B b
CI	7.66 ± 0.03 AB a	7.71 ± 0.16 A b	7.44 ± 0.26 B b	7.47 ± 0.13 AB b
Hue	0.80 ± 0.00 B c	0.86 ± 0.00 A c	0.74 ± 0.00 C c	0.71 ± 0.00 D c
	sat2			
	CC-17	BM-17	CC-18	BM-18
Abs 420 nm	3.19 ± 0.01 B b	3.26 ± 0.02 A b	3.05 ± 0.01 C b	3.00 ± 0.02 D b
Abs 520 nm	3.81 ± 0.01 C ab	3.66 ± 0.02 D a	3.88 ± 0.02 B a	4.01 ± 0.03 A b
Abs 620 nm	0.77 ± 0.02 A a	0.74 ± 0.02 B a	0.69 ± 0.01 C a	0.66 ± 0.02 C a
CI	7.77 ± 0.02 A a	7.66 ± 0.07 B b	7.61 ± 0.04 B ba	7.67 ± 0.06 B a
Hue	0.84 ± 0.00 B b	0.89 ± 0.00 A b	0.79 ± 0.00 C b	0.75 ± 0.00 D b
	sat3			
	CC-17	BM-17	CC-18	BM-18
Abs 420 nm	3.29 ± 0.09 B a	3.59 ± 0.26 A a	3.17 ± 0.06 B a	3.13 ± 0.05 B a
Abs 520 nm	3.74 ± 0.10 B ab	3.84 ± 0.28 AB a	3.87 ± 0.07 AB a	4.04 ± 0.12 A a
Abs 620 nm	0.76 ± 0.03 A a	0.78 ± 0.06 A a	0.68 ± 0.02 B a	0.68 ± 0.02 B a
CI	7.79 ± 0.22 A a	8.21 ± 0.60 A a	7.72 ± 0.15 A a	7.85 ± 0.18 A a
Hue	0.88 ± 0.00 B a	0.93 ± 0.00 A a	0.82 ± 0.00 C a	0.78 ± 0.02 D a

Different capital letters indicate a statistically significant difference between samples within the same saturation cycle; lowercase letters indicate a statistically significant difference between saturation cycles, according to the Fisher LSD test ($p < 0.05$)

[a]The values represent the mean ± standard deviation over four replicates

seen after five years of bottle aging in Cabernet [40] and Casavecchia [22] wines.

About CIElab coordinates (Table 3), the loss of violet hue and the accumulation of tawny tonality during red wine aging are shown by the increase of b* (blueness), and hue (angle), and the decrease of chromatic parameters a* (redness) as already observed in other aged red wines [13, 41]. Consequently, the color variation between the wines before and after the three saturation cycles is higher than three CIElab units ($\Delta E > 3$) for all wines, and it is more pronounced in the Sangiovese 2017 wines. Values > 3 indicate wines show a difference detectable to the human eye with respect to the control. With the increase of the saturation cycles, ΔE increases. The higher effect is observed in the oldest wines, probably because molecules such as pinotins are characterized by more orange tint [13] are preferentially formed.

Acetaldehyde variation

Figure 3 shows the acetaldehyde concentration in wines before (zero time) and after each saturation cycle (sat1-2-3).

The percentage increase of acetaldehyde strongly differs in wines between the 2017 and 2018 vintage, equal to 67% and 65% for CC-17 and BM-17, and only 39% and 26% for CC-18 and BM-18, respectively. This difference could be explained if we consider acetaldehyde's great reactivity towards monomeric anthocyanins showed in model solution in the presence of flavanols [32, 38, 42]. Therefore, it is likely that the higher the content of

Table 3 The main CIELAB values of the Sangiovese wines subjected to three saturation cycles

	Zero time[a]			
	CC-17	BM-17	CC-18	BM-18
L*	64.62 ± 0.43 B c	65.85 ± 0.30 A a	64.62 ± 0.63 B b	64.82 ± 0.27 B a
a*	20.17 ± 0.20 B a	19.65 ± 0.09 A ab	20.27 ± 0.05 B c	20.57 ± 0.09 C c
b*	11.12 ± 0.04 D d	11.90 ± 0.11 A d	11.35 ± 0.09 C d	11.65 ± 0.17 B d
C	23.07 ± 0.17 BC b	22.97 ± 0.05 C c	23.22 ± 0.05 B d	23.67 ± 0.12 A c
h	16.55 ± 0.38 B d	19.65 ± 0.29 A d	16.07 ± 0.28 B d	16.07 ± 0.49 B d
	sat1			
	CC-17	BM-17	CC-18	BM-18
L*	64.80 ± 0.40 A bc	65.17 ± 0.12 A ab	65.22 ± 0.91 A ab	65.45 ± 0.96 A a
a*	20.50 ± 0.57 BC a	20.12 ± 0.32 C ab	20.87 ± 0.20 B a	21.55 ± 0.12 A a
b*	13.82 ± 0.18 B c	14.37 ± 0.17 A c	12.97 ± 0.25 C c	12.95 ± 0.23 C c
C	24.70 ± 0.53 AB a	24.72 ± 0.33 AB b	24.55 ± 0.26 B c	25.12 ± 0.09 A b
h	22.80 ± 0.49 B c	25.10 ± 0.27 A c	19.30 ± 0.37 C c	18.05 ± 0.50 D c
ΔE	2.79 ± 0.29 A c	2.62 ± 0.36 A b	1.88 ± 0.33 B c	2.00 ± 0.30 B c
Δhue	6.25 ± 0.19 A c	5.45 ± 0.26 A c	3.22 ± 0.47 B c	1.97 ± 0.87 C c
	sat2			
	CC-17	BM-17	CC-18	BM-18
L*	65.22 ± 0.28 A b	65.60 ± 0.29 A a	65.52 ± 0.25 A a	65.57 ± 0.20 A a
a*	20.05 ± 0.17 C a	19.45 ± 0.05 D b	20.70 ± 0.11 B ab	21.70 ± 0.00 A a
b*	14.80 ± 0.18 B b	15.35 ± 0.05 A b	14.42 ± 0.04 C b	14.30 ± 0.08 C b
C	24.92 ± 0.23 C a	24.77 ± 0.09 C b	25.25 ± 0.05 B b	25.97 ± 0.05 A a
h	25.92 ± 0.20 B b	28.65 ± 0.12 A b	23.17 ± 0.28 C b	21.10 ± 0.08 D b
ΔE	3.73 ± 0.21 A b	3.47 ± 0.93 AB b	3.29 ± 0.29 BC b	2.99 ± 0.17 C b
Δhue	9.37 ± 0.53 A b	9.00 ± 2.77 A b	7.10 ± 0.24 B b	5.02 ± 0.45 C b
	sat3			
	CC-17	BM-17	CC-18	BM-18
L**	65.80 ± 0.21 A a	64.47 ± 0.92 B b	65.80 ± 0.14 A a	65.30 ± 0.31 A a
a*	19.30 ± 0.29 B b	20.45 ± 1.15 A a	20.47 ± 0.26 A bc	21.07 ± 0.17 A b
b*	15.80 ± 0.18 B a	18.02 ± 1.06 A a	15.42 ± 0.14 B a	15.17 ± 0.14 B a
C	24.97 ± 0.38 B a	27.27 ± 1.58 A a	25.65 ± 0.35 B a	25.95 ± 0.19 B a
h	29.77 ± 0.14 B a	32.82 ± 0.09 A a	26.17 ± 0.17 C a	24.25 ± 0.05 D a
ΔE	4.93 ± 0.13 B a	6.43 ± 1.47 A a	4.27 ± 0.30 BC a	3.60 ± 0.32 C a
Δhue	13.22 ± 0.30 A a	13.17 ± 0.35 A a	10.10 ± 0.43 B a	8.17 ± 0.49 C a

Different capital letters indicate a statistically significant difference between samples within the same saturation cycle; lowercase letters indicate a statistically significant difference between saturation cycles, according to the Fisher LSD test ($p < 0.05$)

ΔE and Δhue are calculated with respect to the zero time of the same sample

[a]The values represent the mean ± standard deviation over four replicates

monomeric anthocyanins and flavanols in young wines, the greater the involvement of acetaldehyde in these reactions. A recent study also shows a direct relationship between anthocyanins, condensed flavanols, and acetaldehyde consumptions to produce polymeric pigments [43]. This is evident in BM-18 wine, where the concentration of polymeric pigments is one of the highest. It is not even ruled out that, in young red wines as Sangiovese 2018, the presence of a higher amount of anthocyanins than the more pro-oxidant compounds as the flavanols [5] directly limits the production of acetaldehyde due to the lower activity of orto-diphenols in triggering the Fenton reaction.

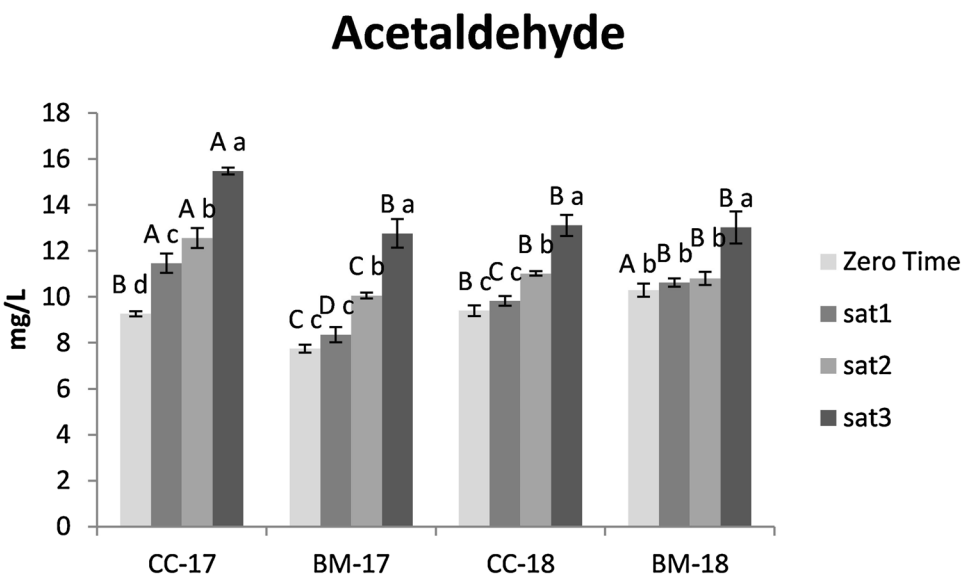

Fig. 3 Acetaldehyde content of Sangiovese wines before and after each saturation cycle. Histograms with different letters differ according to Fisher LSD analysis ($p < 0.05$). Capital letters refer to differences between wines; lowercase letters refer to saturation cycles

Tannins and total phenolics variation

The oxidation also determines a variation in the polymerization degree of proanthocyanidins in wine. The measure of low molecular weight proanthocyanidins and high molecular weight proanthocyanidins (HMWP) is carried out by evaluating their reactivity towards specific reagents. The parameter VRF, which measures tannins reactivity towards vanillin and, indirectly, proanthocyanidins at low molecular weight [44], decreases following oxidation for all wines, around − 12% (Fig. 4a).

While VRF change does not differ between wines, changes in tannins reactive towards BSA depend on wine type (Fig. 4b): in BM the decrease is between 8 and 9%, while for CC the reduction is almost three times higher (25–29%). Since the BSA reactive tannins are mainly the HMWP with a polymerization degree higher than three [45], the variation in their content could be significant for wine quality. Recently, some of these complex structures in aged red wines have been partially elucidated by combining a purification step and different chemical degradation methods [46]. However, given the diversity and structural complexity of red wines, the compositional characterization and possible correlation with sensory attributes remain difficult. For this reason, in this study, the sensory evaluation of Sangiovese wine mouthfeel is also carried out to understand the possible sensory implication of the observed variations due to oxidation (Sensory characteristics variation paragraph). Concerning the total phenolics variation (Fig. 4c), determined by measuring their reactivity towards iron, a higher loss is detected after the oxidation of 2017 wines (− 15% in 2017 against − 10% in 2018 wines), confirming that the global reactivity of wine phenolics is more dependent on the year than the wine type [47].

Sensory characteristics variation

The sensory evaluation of Sangiovese wines CC-17, BM-17, CC-18, and BM-18 is made before (zero time) and after the last saturation cycle (sat3). Table 4 shows the mean values of the sensory characteristics influenced by oxidation: the intensity of astringency ($p = 0.001$), balsamic odor ($p = 0.001$) and aroma ($p < 0.000$), and the RATA of the astringency subqualities such as silk ($p = 0.000$) and velvet ($p = 0.001$). The percentage variation due to oxidation of these sensory attributes for each Sangiovese wine is shown in Fig. 5.

The overall astringency intensity decreases after oxidation in all wines (Table 4). The oxidation effect is enhanced in younger wines with a decrease of − 23% (2018), while the 2017 wines CC-17 and BM-17 show decreases of − 7% to − 18%, respectively (Fig. 5a). The reduction of astringency with time has been shown to depend on the reduced concentration of tannins due to precipitation [14, 48], but not strictly related to the BSA-tannins, as shown in Fig. 4a. The astringency of red wine decreases during aging also because of the changes in the structure of tannins due to numerous reactions as those generating low molecular weight species [14], the polymerization and subsequent precipitation [15], and direct or indirect condensation with anthocyanins [49]. The decrease in astringency is also related to the decline in monomeric anthocyanins during aging.

We find a high Pearson correlation between astringency and monomeric anthocyanins ($R > 0.68$; $p < 0.05$). The implication of anthocyanins in astringency perception is also recently investigated by sensory analysis [50] and interaction with oral constituents, namely oral epithelium cells and salivary proteins [51]. As the astringency decreases, the mouthfeel sensation of silk increases in

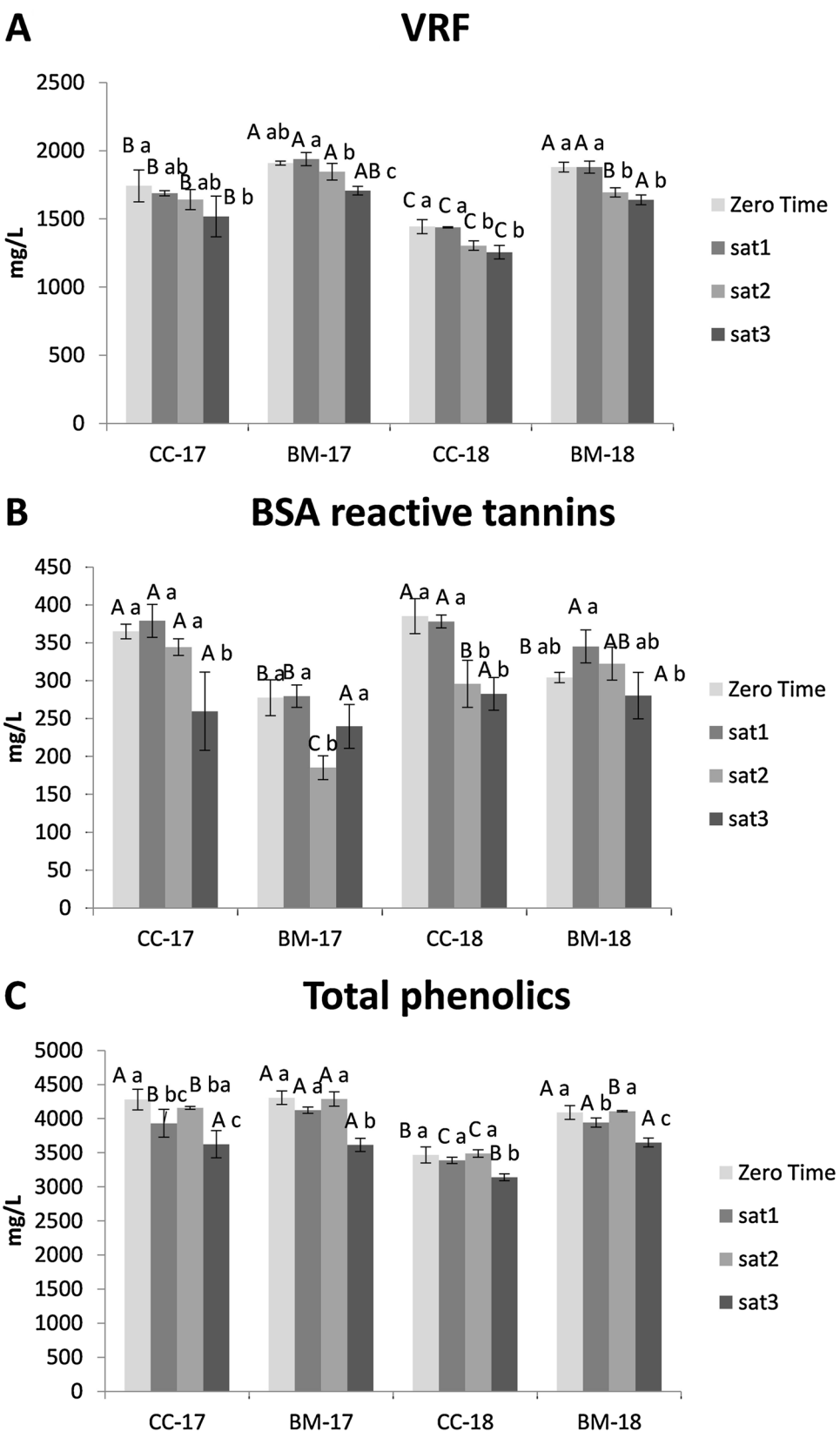

Fig. 4 **a** Vanilline Reactive Flavans (VRF); **b** BSA reactive tannins; and **c** total phenolics of Sangiovese wines before and after each saturation cycle. Histograms with different letters differ according to Fisher LSD analysis ($p < 0.05$). Capital letters refer to differences between wines; lowercase letters to saturation cycles

Table 4 The sensory characteristics of Sangiovese wines before (Zero time) and after the 3rd saturation cycle (sat3), expressed as the mean of intensity for astringency, balsamic odor, and aroma; and the mean of RATA for the astringency subqualities silk and velvet

Intensity[a]		Astringency	Balsamic odor	Balsamic aroma
Zero time (NO-OX)	CC-17	4.7 ± 0.1 b	2.5 ± 0.3 d	1.6 ± 0.0 d
	BM-17	4.7 ± 0.1 b	2.6 ± 0.3 d	1.7 ± 0.1 d
	CC-18	4.5 ± 0.2 bc	2.5 ± 0.2 d	1.8 ± 0.2 d
	BM-18	5.2 ± 0.0 a	2.7 ± 0.2 cd	1.9 ± 0.1 cd
sat3 (OX)	CC-17	4.4 ± 0.2 cd	3.2 ± 0.1 ab	2.6 ± 0.1 a
	BM-17	4.0 ± 0.1 e	3.2 ± 0.1 ab	2.2 ± 0.0 bc
	CC-18	3.7 ± 0.2 f	3.6 ± 0.0 a	2.4 ± 0.1 ab
	BM-18	4.2 ± 0.0 de	3.1 ± 0.1 bc	2.3 ± 0.1 b
Oxidation effect[b]				
	NO-OX	A	B	B
	OX	B	A	A
	p value	0.001	0.001	< 0.000

RATA[a]		Silk	Velvet
Zero time (NO-OX)	CC-17	5.1 ± 0.1 b	2.6 ± 0.1 cd
	BM-17	5.2 ± 0.4 b	3.5 ± 0.4 b
	CC-18	3.9 ± 0.1 c	2.3 ± 0.4 de
	BM-18	3.8 ± 0.2 c	1.9 ± 0.1 e
sat3 (OX)	CC-17	5.7 ± 0.2 ab	3.7 ± 0.3 b
	BM-17	5.5 ± 0.0 ab	5.3 ± 0.2 a
	CC-18	6.0 ± 0.3 a	3.3 ± 0.2 bc
	BM-18	6.0 ± 0.3 a	5.6 ± 0.3 a
Oxidation effect[b]			
	NO-OX	B	B
	OX	A	A
	p value	0.000	0.001

Different lowercase letters indicate a statistically significant difference of the sample for the intensity and the RATA of each sensory attribute, according to the Fisher LSD test ($p < 0.05$)

[a]The values represent the mean ± standard deviation over two replicates

[b]Different capital letters indicate a statistically significant difference between Zero time samples (NO-OX) and after sat3 (OX) according to the Fisher LSD test ($p < 0.05$)

oxidated wines (Table 4). The percentage variation of the silky feeling increases similarly for the wines of the same year (Fig. 5b). In particular, the increase is more pronounced in 2018 wines than 2017 wines, of about four and sevenfold for CC and BM, respectively. Moreover, the silky sensation negatively correlates with some monomeric anthocyanins, namely Mal-Ac (− 0.62) > Del-3 mg (− 0.60) > Pet-3 mg (− 0.55) > Mal-3 mg (− 0.54). This may mean that the degradation of these anthocyanins or their involvement in the formation of polymeric pigments could be responsible for increasing the wine silkiness during aging. The silk term has been associated with an anthocyanin-containing fraction rich in monomeric and small polymeric pigments [52]. However, the analysis of a wider range of red wines will be carried out in future works to support current data.

The velvety sensation increases around +30% in the CC wines (2017 and 2018) and BM-17, and +62% in the BM-18 (Fig. 5b); the latter represents the most involved in oxidation's smoothing effect. During aging, different chemical reactions can participate in changing the mouthfeel characteristics of red wine. Previous studies show that wine becomes soft and mellow for the decline of tannin mean degree of polymerization [40], velvet for the formation of the polymeric pigments [21], or satin for lower content of flavans and astringent tannins, together with a higher formation of polymers [22] after aging. In the current study, Sangiovese wines, after the saturation cycles, show lower monomeric anthocyanins, flavans, total phenolics, BSA-tannins content, and higher polymeric pigment formation than not-oxidated wines. The BM-18 is the wine with the highest content in polymeric pigments and the velvet sensation. Pearson's correlations reveal that polymeric pigments are highly correlated with the velvet sensation (0.90; $p < 0.05$) and with acetaldehyde production (0.51; $p < 0.05$),

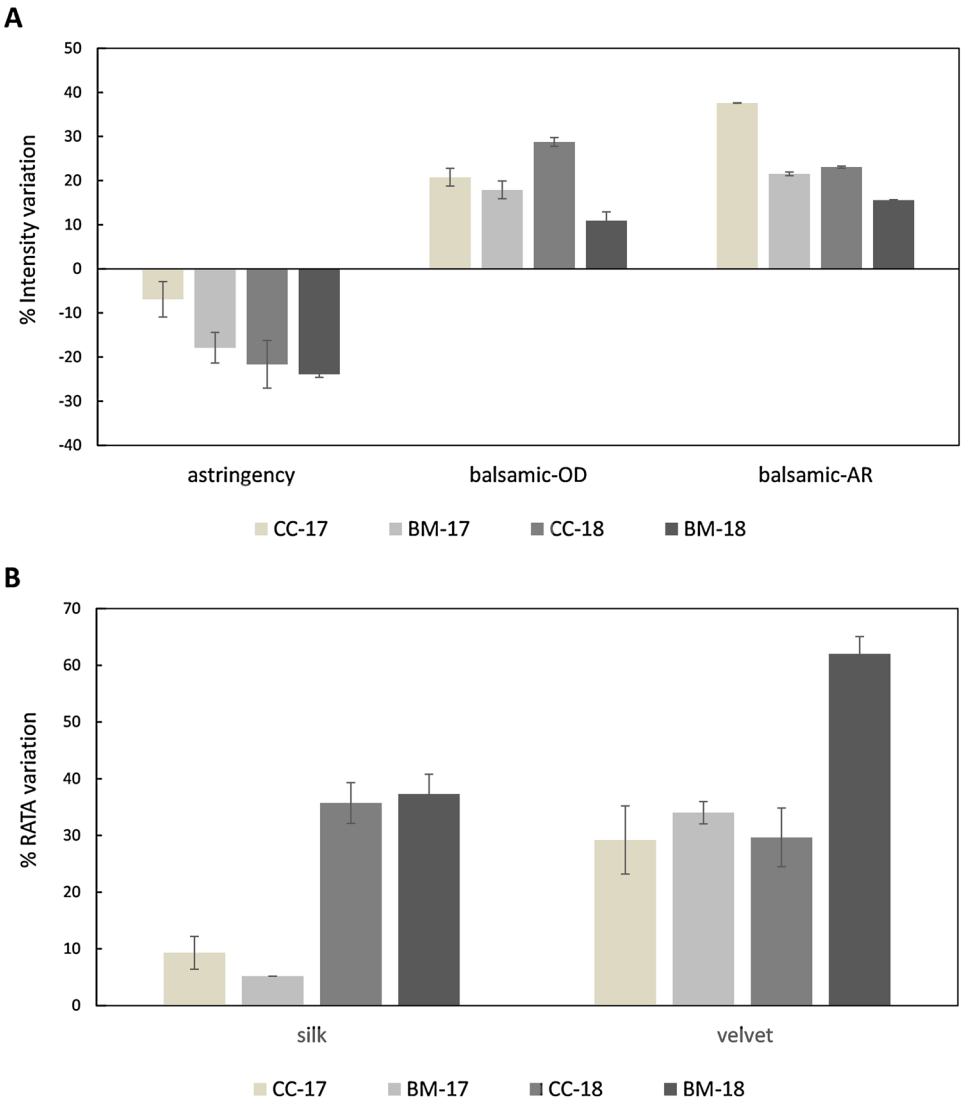

Fig. 5 The percentage variation (between the zero time and sat3) of the sensory characteristics influenced by oxidation of 2017 and 2018 Sangiovese wines (CC-17; BM-17; CC-18; BM-18). **a** The intensity variation of astringency, balsamic odor (-OD), and aroma (-AR). **b** The RATA variation of silk and velvet (astringency subqualities)

in accordance with previous studies [21, 22]. A significant correlation (0.51; $p < 0.05$) was also found between the decrease in malvidin 3-(6^{II}-acetyl)-monoglucoside and the velvet subquality. It seems that the formation of other anthocyanin-derived structures involving acetaldehyde could be linked to a more velvety astringency [15]. In recent work, the increase of the velvet sensation is observed in Tuscan Sangiovese wines after two years of aging [16].

Regarding the other sensory characteristics, the wines at time zero do not differ in the intensity of balsamic odor and aroma (Table 4). Still, after oxidation, a significant increase of these attributes (between 11 and 37%) is perceived in all Sangiovese wines (Fig. 5a). The balsamic odor category contains resin, eucalyptus incense, resinous, and turpentine notes. The compounds that potentially contribute to these scents have been shown to accumulate during Valpolicella wines' aging. These odorous aging markers are found to be the 1,8-cineole, 1,4-cineol, and p-cymene, and their accumulation also depends on the grape variety [53]. In Sangiovese wines, the balsamic notes increase seems to be more related to wine type than vintage (Table 4). The CC wines show a higher increase in the balsamic notes than BM wines, and between the two consecutive years, the 2018 wine has a more balsamic odor, while a more balsamic aroma is perceived in the 2017 wine. The Sangiovese CC wine will be bottled as Chianti Classico DOCG, which, following a previous study, is characterized by balsamic odor and velvety tannins after 2 years of aging [16]. Therefore, the sensory evolution of the Sangiovese wines after the aging through moderate oxygen exposure results in the typical sensory characteristics found in the commercial wines after 2 years of bottle aging. Finally, the saturation test has been shown to provide useful information on the evolution of Sangiovese wines. Further studies will be carried out on the sensory changes related to aroma and astringency associated with natural and induced aging.

Conclusions

Consecutive oxygen saturation cycles, providing around 18 mg O_2 /L, represent a useful tool to simulate wine aging under moderate oxygen exposure. Results give available information on the aging of Sangiovese wines such as CC and BM, the most important wines in the Tuscany region and Italy. After each oxidation step, the monomeric anthocyanins decrease, the polymeric pigments are formed, and acetaldehyde is produced. Polymeric pigments, which assure color stability during the time, are mainly formed in BM than CC, independently from the vintage. Color intensity increases as the yellow tint. While the low molecular weight proanthocyanidins follow a similar decrease, polymerized tannins decrease three times more in CC than BM. However, after the last saturation cycle, the tannin content is similar between all wines. Oxidation also induces changes in the sensory characteristics of Sangiovese wines. The intensity of overall astringency decreases much more in 2018 wines, where a higher monomeric anthocyanins decline was also detected. The wines became silky and velvety after oxidation. The silk sensation increases more in 2018 wines and correlates with Mal-Ac > Del-3 mg > Pet-3 mg > Mal-3 mg decrease. The velvet subquality depends on the wine composition: it correlates with Mal-Ac decrease, polymeric pigments formation, and acetaldehyde production.

Aged CC and BM wines are significantly characterized by balsamic notes, which are revealed by the effect of oxidation. Results highlight that a saturation test can help enologists managing oxygen exposure during aging, thus avoiding detrimental variation in phenolics, pigments, and aroma, while improving mouthfeel. Further studies will be carried out to understand better the structural changes in the phenolic profile and the appearance of balsamic notes of wines, as well as the mouthfeel improvement by oxidation.

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Author contributions AR: Conceptualization, investigation, formal analysis, methodology, writing original draft. LP: Formal analysis, methodology. SS: Review and editing. EB: Review and editing. VF: Resources, supervision. LM: Resources, supervision. AG: Conceptualization, supervision, review and editing.

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Data availability Data are available upon request; instead, wines are not more available.

Declarations

Conflict of interest The authors declare no conflict of interest.

Compliance with ethics requirements All procedures performed in studies involving human participants were in accordance with the ethical standards of "IFST Guidelines for Ethical and Professional Practices for the Sensory Analysis of Foods" and with the 1964 Helsinki declaration and its later amendments or comparable ethical standards.

Consent to participate Panelists gave their informed consent, and their privacy rights have always been observed.

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