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Abstract: Chardonnay wines have a long-standing reputation regarding their aging potential. However, in some cases, they face premature oxidation a few years after bottling. Scientific reports are, for now, multiparametric and unclear. Polyphenols seem to be an important factor involved in the oxidative stability of white wines, but their role has not yet been completely characterized. The present study aimed to investigate the link between polyphenol content and the emergence of oxidative odors of bottle-aged Chardonnay wines. In order to obtain samples with noticeable differences in polyphenol content, as well as in sensory oxidative notes, wines from two different vintages were used. For each vintage, three levels of must clarification and two wine closures were implemented. Polyphenol content was analyzed chemically, and the oxidative character was assessed sensorially by a trained panel using a specific intensity scale. The results showed significant effects for closure type and turbidity. However, these effects were strongly affected by vintage. Concerning the polyphenol content, a clear difference was also found between vintages, closures and turbidity levels. Significant linear regression models for REDOX scores pointed out Flavon-3-ols as the main negative predictor, and grape reaction product (GRP) as the main positive predictor. The enological implications are discussed.

Keywords: flavan-3-ols; reduction; oxidation; wine aging; oxidative stability; clarification

1. Introduction

Chardonnay wines have a long-standing reputation regarding their aging potential. During bottle aging, wine is exposed to relatively low quantities of oxygen, which are nevertheless sufficient to influence its sensory characteristics [1]. In particular, oxygen modulates the extent of different reactions involving volatile and nonvolatile components, resulting in the formation/degradation of a number of powerful aroma compounds, with major consequences for the process of aroma evolution during bottle aging [2]. In addition, other chemical reactions taking place during bottle aging do not involve oxygen, meaning that, even in an environment completely devoid of oxygen, a certain form of aging will occur [3]. Wine aroma changes dramatically during bottle aging, through a complex array of chemical reactions that are only partly understood. In most cases, oxygen and polyphenols contribute to the evolution of these key aroma compounds [4] where iron and copper act as oxidation catalyzers [5]. To date, the majority of studies dealing with premature oxidation

have focused on the characterization of potent oxidation markers of defective aroma (off-flavors). According to these studies, changes in wine aroma properties linked to oxidation were related to the formation of off-flavors, mainly aldehyde compounds, such as phenylacetaldehyde, methional, *trans*-2-nonenal, o-aminoacetophenone and a lactone, the 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone (sotolon) [6,7]. The aldehydes, methional and phenylacetaldehyde, are among the oxidation-related aroma compounds that have drawn the most attention, due to their supposedly higher aroma impact and possible contribution to the aroma of red and white wines [8]. For these aldehydes, formation from the amino acids methionine and phenylalanine, respectively, via Strecker reaction involving the presence of a dicarbonyl compound has been proposed [9]. Different o-diphenols have been shown to form different quantities of aldehydes, with caffeic acid giving higher methional and phenylacetaldehyde compared to catechin and epicatechin [10], but this was observed at pH much higher than that of wine. More recently, it has been shown that in wine-like conditions, methionine and phenylalanine were not capable of reacting with a model quinone [11]. Finally, in a recent study, the polyfunctional thiol 3-ethylsulfanyl acetate was identified for the first time as a key contributor to off-flavors in sauvignon blanc wines [12]. This compound was observed at higher levels in aged wines and in wines obtained from juices exposed to air, but the effect of post-bottling oxygen exposure on its concentration remains to be investigated. Overall, although many aroma compounds relevant to wine oxidation have been identified, most studies have been aimed at understanding wine oxidative spoilage, and have often been carried out under conditions of extreme oxygen exposure. Conversely, oxidative processes taking place during bottle aging under normal conditions are rather "mild", and the sensory impact of such levels of oxidation to the aroma quality of wines remains to be established.

Many chemical approaches have been developed in the literature to understand white wine oxidation. However, there are fewer studies dealing with white wine oxidation from a sensory point of view. Typically, the researchers use a tasting panel to generate relevant attributes, which usually cover fresh fruit dimensions (depending on the grape variety at hand), oxidative aroma notes and a number of other descriptors unrelated with oxidation (such as spiciness, oakyness and other wine faults). Several authors have used descriptive analysis to characterize the type of closure and the effect of ascorbic acid addition on the oxidative or reductive aromas of white wine samples with several years of storage [13–15]. In Gooden et al. [13], fresh fruit attributes were overall fruit, pineapple, citrus/lime and tropical while the oxidation related attributes were oxidized and developed. Interestingly, the authors aimed to differentiate two different nuances of oxidation, developed being probably a mild and not necessarily negative version of oxidized. As expected, the authors showed a clear opposition between all fresh fruit dimensions, and oxidized and developed. Ulterior studies [14–16] slightly modified the lexicon by including some oxidation notes such as aldehyde, glue-like or wet wool, and some reduction notes, such as struck flint/rubber, gunflint, rotten egg, cabbage or stagnant water. In the case of rosé wine, Guaita and coworkers [17] suggested, despite the lack of significance of the results, that the closures with higher Oxygen Transfer Rate (OTR) values induced a stronger intensity of acacia flower and rose aromas in the wines than more protective closures. In the same vein, Rodrigues and coworkers [18] used the attribute honey/wax as an oxidation marker. A different strategy was used by Brajkovich and coworkers [19], since they basically used sauvignon blanc varietal descriptors such as passion fruit, cat urine, grassy and capsicum, and showed that the most oxidized samples were those showing lower intensities for thiol-related attributes.

In that context, the goal of the present study is to investigate the oxidative note of bottle-aged Chardonnay wines from a polyphenolic content perspective. To the best of our knowledge, no sensory studies have so far examined the link between the occurrence of oxidative aromas and the polyphenol content in white wines, in particular hydroxycinnamic acids and flavanols. This is all the more interesting because previous studies reported contradictory results; some have considered that the antioxidant activity of white wines is related to its polyphenolic content [20–22], whereas others have shown a direct correlation between the browning rate and the concentration of epicatechin [23]. In order to ensure wide differences in the polyphenol concentrations and sensory characteristics

between the samples, two different vintages with three levels of must turbidity and two different types of closure were used. The two kinds of stoppers chosen for this study (synthetic coextruded stoppers and screw cap) presented extreme OTR values to ensure oxidation differences as previously described [3]. Synthetic coextruded stoppers are able to diffuse 10 to 100 times more oxygen that screw caps, and could possibly reveal oxidative deviations earlier in wine compared to more reductive environments when screw caps are used [3,24].

2. Materials and Methods

2.1. Wines

Chardonnay dry white wines from Burgundy were elaborated following the same winemaking process during vintages 2009 and 2010. Chardonnay grapes were hand-harvested and pressed with a pneumatic press. Must was protected with 4 $g \cdot hL^{-1}$ of SO_2. Cold racking at 12 °C from 12 to 24 h enabled to reach the three levels of must turbidity: 300 NTU (Low), 600 NTU (Medium) and 800 NTU (High). Alcoholic and malolactic fermentations were conducted in oak barrels followed by oak aging for 6 months. Dry white wines were then filtered, and SO_2 was adjusted to 40 $mg \cdot L^{-1}$ prior to bottling. Wine bottles were stored in a cellar with a constant temperature (12 °C) until required for chemical analysis (April 2015). Table 1 summarizes the characteristics of the samples used in this study.

Table 1. Characteristics of the white wines used in this study and their codes.

Sample Code	Closure	Turbidity (NTU)
S-L-2009	synthetic coextruded stopper	LOW
S-M-2009	synthetic coextruded stopper	MEDIUM
S-H-2009	synthetic coextruded stopper	HIGH
C-L-2009	screw cap	LOW
C-M-2009	screw cap	MEDIUM
C-H-2009	screw cap	HIGH
S-L-2010	synthetic coextruded stopper	LOW
S-M-2010	synthetic coextruded stopper	MEDIUM
S-H-2010	synthetic coextruded stopper	HIGH
C-L-2010	screw cap	LOW
C-M-2010	screw cap	MEDIUM
C-H-2010	screw cap	HIGH

2.2. Sensory Analysis

Sensory sessions took place between October and December 2015. After a training period, the selected assessors performed a monadic assessment of the reductive and oxidative aromas of the samples and a sensory description on frequency of citation [25]. Afterwards, a subset of the samples was analyzed again, not in monadic presentation, but in simultaneous presentations in order to induce a comparative assessment and therefore detect more subtle differences between samples.

2.2.1. Panel: Training and Selection

Twenty-six candidates were recruited from among the students at the enology school of Dijon (France). As part of their enology training, they all attended a wine-tasting course (36 h), where they learned the main olfactory notes of wine. Apart from this general training, the panelists took part in 6 training sessions and 4 selection sessions organized as part of the present study. The general goal of the training was to familiarize the assessors to the common oxidation and reduction aroma notes, and to quantitatively calibrate their measurements. With this purpose, natural and spiked wines with a range of reduction or oxidation intensities were selected for the training. Also, several "clean" wines (with no obvious oxidation or reduction notes) were used as controls during the training. Sessions were conducted twice a week, in groups of 10–13 candidates and lasted about 40 min. The specific goals and the content of each session are summarized in Table 2. A specific structured scale (called here the REDOX odor scale) was created to assess the global oxidative or reductive odor of the samples. This 11-point scale went from −5 (strong reduction) to +5 (strong oxidation), with 0 (neither reduced nor oxidized) being in the middle of the scale. Expected REDOX odor levels of the training samples were roughly determined by consensus by three experienced tasters as a guideline only. Systematic feedback was given at the end of each session or at the beginning of the subsequent session.

Table 2. Summary of the training and selection sessions prior to sensory description.

Session Number	Goals and Protocols	Materials
Session 1	Familiarization with phenylacetaldehyde and methional in flasks and in water. Furaneol (oaky note) was used as a distractor. Assessors' descriptions were discussed and compared to the descriptors from the literature.	Phenylacetaldehyde 2,2 µg/L Methional 5 mg/L Furfural, one drop on a cotton ball in a flask.
Session 2	Familiarization with sotolon, 2-aminoacetophenone and methional in flasks and in water. *Trans*-2-nonanal (cardboard note) was used as a distractor. Assessors' descriptions were discussed and compared to the descriptors from the literature.	Sotolon 90 µg/L 2-aminoacetophenone 10 µg/L Methional 5 mg/L *Trans*-2-nonanal, one drop on a cotton ball in a flask.
Session 3	Familiarization with reduction notes: H2S, Ethanthiol, DMS. Recognition of 2-aminoacetophenone and phenylacetaldehyde. Assessors' descriptions were discussed and compared to the descriptors from the literature. Familiarization with the redox scale on the previous solutions	H2S 40 µg/L Ethanthiol 200 µg/L DMS 55 µg/L 2-aminoacetophenone 10 µg/L Phenylacetaldehyde 14 µg/L
Session 4	Discrimination between oxidation and reduction notes in spiked wines and recognition of the different molecules and their descriptors. Familiarization with the redox scale on the previous spiked wines	Base wine (Muscadet, 2014) Base wine + phenylacetaldehyde 14 µg/L Base wine + methional 5 mg/L Base wine + Ethanthiol 200 µg/L Base wine + sotolon 120 µg/L Base wine + 2-aminoacétophenone 10 µg/L
Session 5	Test assessors' discrimination abilities on oxidized samples (spiked wines) by means of a triangle test. Odor characterization of 3 samples using the redox scale and the odor descriptors list.	Base wine (Muscadet, 2014) Used in the triangle test: Base wine + phenylacetaldehyde 2.2 µg/L + sotolon 90 µg/L and Base wine + 2-aminoacétophenone 15 µg/L + sotolon 90 µg/L
Session 6	Test assessors' discrimination abilities between an oxidized sample (spiked wines) and the base wine. Odor characterization of 2 samples using the redox scale and the odor descriptors list.	Base wine (Muscadet, 2014) Base wine + sotolon 90 µg/L

Table 2. *Cont.*

Session Number	Goals and Protocols	Materials
Session 7	Quantitative calibration of the anchors of the scale. Redox rating practice with the redox scale on three wines previously checked by two wine experts as representative of three oxidation levels.	Used for calibration: Reduction anchor (very reduced): H_2S 40 µg/L Oxidation anchor (very oxidized): St Aubin 1998 in a dark ISO glass Used for practice with the redox scale: Saint Aubin 1998 (very oxidized) Marsannay 2007 (moderately oxidized) Viré Clessé 2014 (neither reduced nor oxidized)
Session 8	Redox rating of 4 wines previously checked by two wine experts as representative of three oxidation levels.	Bourgogne Aligoté 2014 (neither reduced nor oxidized) Chardonnay blend with 7% of St Aubin 1998 (slightly oxidized) Petit Chablis 2009 (moderately oxidized) Chablis 1er Cru 2012 (slightly reduced)
Session 9	Redox rating of 6 wines previously checked by two wine experts as representative of three oxidation levels. The last three samples are tasted in session 10 as well in order to check candidates' repeatability.	Chassagne Montrachet, 2002 (moderately oxidized) Chenin Blanc 2013 (slightly reduced) Mâcon Clessé 1994 (very oxidized) Viré Clessé 2014 + Petit Chablis 2009 (moderately oxidized) Chardonnay Pays d'Oc 2014 (clean or just slightly oxidized) Chardonnay Pays d'Oc 2014+ 20% Saint Aubin 1er Cru 1998 (moderately oxidized) Chardonnay California 2010 (moderately oxidized)
Session 10	Redox rating of 6 wines previously checked by two wine experts as representative of three oxidation levels. The last three samples are tasted in session 10 as well, in order to check candidates' repeatability.	Chablis 1er Cru 2012 Aligoté Marsannay 2015 (neither reduced nor oxidized) Viré-Clessé 2014 + 20% Ugni Blanc 1969 (moderately oxidized) Viognier 2013 + 50% Chardonnay 2013 (clean or just slightly oxidized) Chardonnay Pays d'Oc 2014 (clean or just slightly oxidized) Chardonnay Pays d'Oc 2014+ 20% SaintAubin 1er Cru 1998 (moderately oxidized) Chardonnay California 2010 (moderately oxidized)

The selection of the panelists was based on the results of sessions 7 to 10. Three main criteria were considered in order to rank the candidates according to their sensory performance: recognition of oxidation and reduction notes and proximity with the expected REDOX odor levels, repeatability, and consensus with the rest of the panel. Panelists were ranked according to each of these three criteria, and then an average rank was computed for each candidate.

At the end of the selection process, 14 assessors were selected to form the final panel (three females and eleven males, average age 24.4; SD = 1.4). Among them, eleven (three females and eight males, average age 24.3; SD = 1.3) participated in the monadic sensory sessions. All fourteen assessors participated in the comparative REDOX odor assessment.

2.2.2. Monadic Sensory Description

Sensory analyses took place in a sensory room equipped with individual booths. Two bottles of each sample were assessed in standardized black glasses coded by 3-digit numbers. Each sample contained 20 mL of wine at ambient temperature and was covered with a plastic Petri dish. First, participants were asked to rate the intensity of oxidation-reduction of the samples orthonasally (i.e., by smell only) and afterwards globally (nose and palate) using the REDOX odor scale. Finally, panelists were asked to describe the odor of the samples using a list of odor attributes (Figure 1) from which assessors should select all the attributes they considered appropriate to describe each sample. The sensory terms aimed to cover varietal chardonnay aromas, oxidation and reduction notes and other wine faults (in order to control for false positives). The list was based on previous research on white wine oxidation [13–16], and the terms were listed in alphabetical order. If panelists needed an attribute that was missing from the list, two slots (other 1 and other 2) were allowed for their own descriptors. During each session, the twelve samples (six wines replicated) were randomly split into three series of four samples. The tasting order within the series was specific to each participant and followed a William's Latin square. In order to reduce sensory fatigue, panelists were asked to take a short break between series. Monadic sensory description took two sessions, one for 2009 samples and another, the day after, for the 2010 samples.

Bitter almond		Herbaceous		Rotten egg/cabbage	
Bruised apple		Honey		Spicy	
Butter		Leather/Stable		Stagnant water	
Caramel/vanila		Mineral		Toasted	
Citrus		nail polish remover		Tropical fruits	
Cooked vegetables		Woody		Wallnut/curry	
Corked		Prune		Wax/mothball	
Dust/carboard		Quince		White fruits	
Floral		Rancid butter		Yellow fruits	
Forest floor		Rancid honey		Other 1	
Hay		Rancio/madere		Other2	

Figure 1. Ballot used for the general odor description (translated from French).

2.2.3. Comparative REDOX Odor Assessment between Turbidity Levels with Synthetic Coextruded Stopper

This second descriptive analysis was carried out in order to focus specifically on the effect of turbidity. According to the results of the monadic description (see Section 3.1), the samples closed with screw caps barely showed oxidative notes. In particular, for the 2010 samples, the differences in redox score between closure types were really important which were assumed to mask subtle

differences between turbidity levels (see Section 3.1). Therefore, only the samples with synthetic coextruded stoppers were chosen for the comparative REDOX odor assessment. Moreover, inspired by Godden and coworkers [13], a full comparative approach was carried out in order to accede to more subtle differences.

Only one bottle of each treatment was used in this session. Comparative REDOX odor assessment took only one session, during which 6 samples were assessed. The three 2009 samples were simultaneously presented to the assessors (instead of monadically), in order to facilitate the comparison between samples. Panelists were allowed to freely retaste and compare the samples before scoring them. Then, after a short break, the three 2010 samples were presented for assessment, also simultaneously. As in the monadic descriptive analysis, participants were asked to rate the REDOX odor character of the samples orthonasally and, afterwards, globally, using the REDOX odor scale. In order to increase the sensitivity of the REDOX odor scale, a continuous version of the scale was presented to the panelists (instead the discrete scale used in the monadic profile). A subsequent general description based on frequencies of citation was not carried out in this case. Within each series, samples were presented according to a specific order for each participant following a William's Latin square.

2.3. Chemical Analysis

2.3.1. Reagents

Gallic acid, protocatechuic acid, hydroxybenzoic acid, tyrosol, hydroxytyrosol, salicylic acid, coumaric acid, ferulic acid, caffeic acid, caftaric acid, catechin, epicatehin, chlorogenic acid and gentisic acid were purchased from Sigma Aldrich. High-grade quality methanol and formic acid were used for chromatographic elutions.

2.3.2. Wine Polyphenols Analyses

Total polyphenols Index (TPI) measurements. Each wine sample was characterized by measuring the absorbance at 280 nm with a spectrophotometer (UV-1800, Shimadzu, Kyoto, Japan), after dilution, representing the global content of wine polyphenols expressed as TPI index.

UPLC-DAD/FLD phenolic compounds analysis. An Acquity UPLC H-Class (Waters, Milford, MA, USA) with a quaternary pump and an autosampler were coupled to a fluorimetric and a diode array detector. Under optimized conditions, the column oven was thermostated at 35 °C, and the sample system at 12 °C. The acquisitions, the solvent delivery and the detection were performed by Empower 2. About 2 mL of wine was filtered through a 0.45 µm PTFE filter (Restek, Lisses, France), of which 2 µL was injected into a reversed-phase BEH C18 (150 mm × 2.1 mm, 1.7 µm) Waters column. A binary solvent was run at a flow rate of 0.25 mL/min employing (A) H_2O:methanol (95:5) *v*/*v* formic acid (0.1%) and (B) methanol (100%). The optimized elution system consisted of a stepwise gradient as follows: from 3 to 5% B (0–4 min), 5 to 8% B (4–10 min), 8% B (10–12 min), 8 to 10% B (12–14 min), 10 to 15% B (14–17 min), 15 to 30.1% B (17–19 min), 30.1 to 38% B (19–21 min), 38 to 41% B (21–24 min), 41 to 50% B (24–30 min), 50 to 100% B (30–31 min), 100% B (31– 31.5 min), 100 to 3% B (31.5–32.5 min), 3% B (32.5–35 min). The diode array detector was set at 320 nm for *trans*-caftaric acid, gentisic acid, *trans*-caffeic acid, *trans*-coutaric acid, 2-S-glutathionylcaftaric acid (GRP), *trans*-ferulic acid, at 305 nm for salicylic acid, *trans*-coumaric acid, at 280 nm for gallic acid and at 260 nm for protocatechuic acid and hydroxybenzoïc acid. The fluorescence detector was set at λex = 270 nm and λem = 322 nm for hydroxytyrosol, tyrosol, catechin, epicatechin, proanthocyanidin B1, proanthocyanidin B2. Polyphenols were identified using a combination of commercial standards and the UV-visible spectra associated to chromatographic peaks in comparison with published procedures, as described previously [26,27]. *Trans*-Coutaric acid and GRP were quantified via their respective absorbance at 320 nm and expressed in *trans*-caftaric acid equivalents. Unknown concentrations were determined from the regression equation, and the results were converted into milligrams per liter. The sum of each individual polyphenol concentration was calculated and defined as the

total polyphenol for five specific chemical families: phenolic acids, cinnamic acids, flavan-3-ols, Grape reaction product (GRP) and tyrosol.

2.4. Statistical Analysis

Differences in REDOX scores were tested statistically using analysis of variance (ANOVA) with $\alpha = 5\%$. Monadic data were subjected to three-way ANOVAs with wine closures (synthetic cork or screw cap) and turbidity (LOW, MEDIUM and HIGH) as within-subject factors. Subjects (panelists) were considered a random factor. Turbidity × closure, panelists × closure, and panelists × turbidity interactions were also tested. Main treatment effects (closure and turbidity) were tested using their respective interaction with panelists as the appropriate error term.

When a significant main effect was found, pairwise comparisons were carried out using Newman-Keuls' test ($\alpha = 5\%$). In the particular case of the comparative REDOX odor assessment, since all the samples had the same closure system, only the turbidity was set as a within-subject factor.

Concerning the general description, the frequency of citation of each term was determined for each wine. Only the descriptors cited at least 3 times for a given wine were considered in subsequent analyses; the other descriptors were discarded. The resulting contingency table containing the frequency of citation of each term for each wine was submitted to Correspondence Analysis (CA). In order to identify wine clusters, wine coordinates on the two first factors (F1 and F2) on both CAs were submitted to a Hierarchical Cluster Analysis (HCA) with the Ward criterion. Orthonasal and global REDOX average scores were projected as supplementary variables in each CA.

The effect of vintage, closure and turbidity on wine polyphenolic content was analyzed statistically by a three-factor ANOVA with ($\alpha = 5\%$) for each phenolic family. All second-order interactions were also included in the ANOVA model.

Relationships between polyphenol concentrations and REDOX odor scores were explored by means of multiple linear regressions (stepwise method), separately for 2009 and 2010. The added concentration of each family of polyphenols was used as predictors and the REDOX average intensity as the dependent variable.

All statistical analyses were carried out using XLStat 2017 (Addinsoft, Paris, France).

3. Results

3.1. Sensory Characterization by Monadic Profile

3.1.1. REDOX Odor Assessment

The results of the ANOVA performed on the orthonasal and global REDOX odor scores for 2009 and 2010 samples are presented in Table 3. The factor panelists showed a significant effect in both conditions and for both vintages. This is a common phenomenon in sensory evaluation, and indicates that the assessors used different parts of the scale.

Type of closure did not show significant effects for 2009 samples. Concerning the 2010 samples, significant panelists×closure interactions were found for both for orthonasal and global assessments, that some of the panelists did not achieve perfect concept alignment. The MS of panelists × closure interactions was used as an error term to test the closure effect, which was still significant for both assessment conditions (Table 3).

Turbidity level was significant for both conditions of the 2009 samples, but not for the 2010 ones. However, the interaction turbidity×closure was also significant, which means that it is not possible to generalize the turbidity effect to all the closure types. A closer look at this interaction (Figure 2a,b) shows that, for screw caps, medium and high turbidity levels tend to show lower REDOX odor values. However, this pattern does not fit the results for synthetic corks, for which the lowest turbidity also shows the lowest REDOX odor score (Figure 2a,b). Further research needs to be carried out to better understand the relationship between turbidity and oxidation.

Table 4 shows the results of the post-hoc mean comparison for Closure type and Turbidity level. Wines closed with synthetic cork showed significantly higher sensory oxidation than wines closed with screw caps, but only for 2010 samples.

Table 3. Summary of the significance level of the ANOVAS performed on the orthonasal and global REDOX odor scores for the 2009 and 2010 samples. Probabilities in bold are significant at $\alpha = 5\%$.

Assessment Condition	Sources of Variation	2009 Vintage		2010 Vintage	
		F	p	F	p
OTHONASAL PERCEPTION	PANELISTS	3.748	0.0003	3.633	0.0005
	CLOSURE	0.697	0.171	10.108	0.010
	TURBIDITY	3.412	0.038	0.540	0.591
	PANELISTS ×CLOSURE	3.122	0.002	3.289	0.001
	PANELISTS ×TURBIDITY	0.620	0.887	1.016	0.453
	TURBIDITY ×CLOSURE	5.880	0.004	2.081	0.131
GLOBAL PERCEPTION	PANELISTS	3.760	0.0003	3.812	0.0003
	CLOSURE	3.205	0.104	8.109	0.0173
	TURBIDITY	7.044	0.005	1.574	0.2318
	PANELISTS ×CLOSURE	1.827	0.068	2.870	0.0039
	PANELISTS ×TURBIDITY	0.683	0.832	0.994	0.4779
	TURBIDITY ×CLOSURE	3.182	0.046	0.287	0.7510

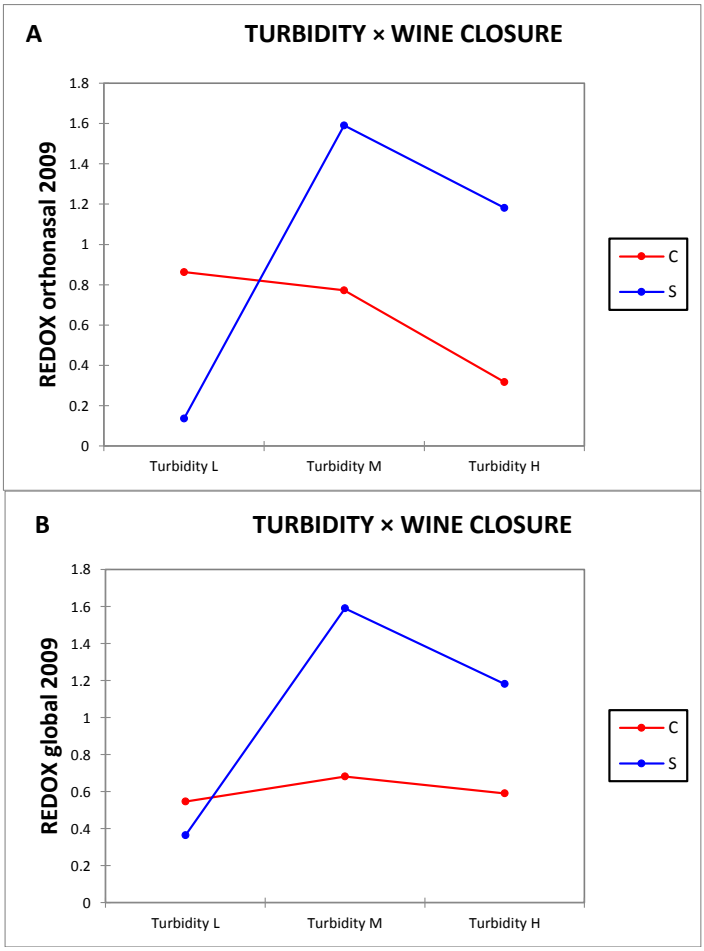

Figure 2. Turbidity*wine closure interaction plots for 2009 data (**A**) orthonasal evaluation and (**B**): global evaluation). S: synthetic coextruded stoppers; C: screw cap stoppers. L: low; M; medium; H: high.

Table 4. Results of the post-hoc Newman-Keuls' tests (α = 0.05) on the REDOX odor intensities comparing the two closure types and the three turbidity levels for each vintage and each evaluation condition. S: synthetic coextruded stoppers; C: screw cap stoppers. L: low; M: medium; H: high.

Factor	Level	2009 Orthonasal	2009 Global	2010 Orthonasal	2010 Global
CLOSURE	S	0.97 a	1.045 a	1.62 a	1.35 a
	C	0.65 a	0.61 a	0.38 b	0.53 b
TURBIDITY	M	1.182 a	1.14 a	1.16 a	1.045 a
	H	0.75 ab	0.89 ab	0.93 a	1.045 a
	L	0.5 b	0.45 b	0.91 a	0.73 a

Samples associated with the same letter were not significantly different.

Concerning the effect of turbidity and regardless the assessment condition, the REDOX odor score for the lowest level was significantly lower than the intermediate level, but wasn't different from the highest level. However, the significant closure*turbidity interactions found for 2009 samples prevent us from generalizing the significant differences found for turbidity.

3.1.2. General Description

Correspondence Analyses were carried out on the matrix containing the frequencies of citation of the most cited attributes for each vintage separately. Moreover, the average orthonasal and global REDOX odor scores were projected as supplementary variables, which were significantly correlated for both 2009 ($r = 0.88$, $p < 0.05$) and 2010 ($r = 0.86$, $p < 0.05$) vintages.

Concerning 2009 samples the CA followed by a HCA did not yield any meaningful or interpretable clusters (data not given). The four emerging clusters were not based on type of closure or turbidity level. Neither the samples nor the attributes seemed organized across a reduction–oxidation gradient (data not given).

Figure 3 shows the first and second dimensions of the CA on the frequencies of citation of the descriptors for the 2010 samples. Dimensions 1 and 2 accounted for 38.33% and 18.54% of the inertia, respectively.

According to cluster analysis, samples were sorted into two main groups. A first group, located in the negative values of the first dimension, was composed by samples with synthetic corks, and was described with oxidative terms such as "rancid honey", "cooked vegetables", "walnut/curry" and "bruised apple". Wines with screw caps were located at positive values of the first dimension and were split into two sub groups. HCA yielded three groups, clearly segmenting the samples according to their type of closure, but not according to their turbidity level. The samples C1 bottle1 and C3 bottle1 were without any taint, and were characterized by the terms "woody", "white fruits", "yellow fruits" and "honey". The other group (C1 bottle2, C3 bottle2, C2 bottle1 and C2 bottle2) was characterized by some reduction terms as "dust/cardboard" or "rotten egg/cabbage" as well as "toasted", "citrus" and "butter". The separation between the two bottles of the samples C1 and C3 suggest a bottle effect or maybe also a lack of repeatability of the panel for these two samples.

Contrary to the CA of the 2009 samples (data not given), the CA of the 2010 samples showed a better discrimination between samples and a better consistency between REDOX odor scores and general attributes.

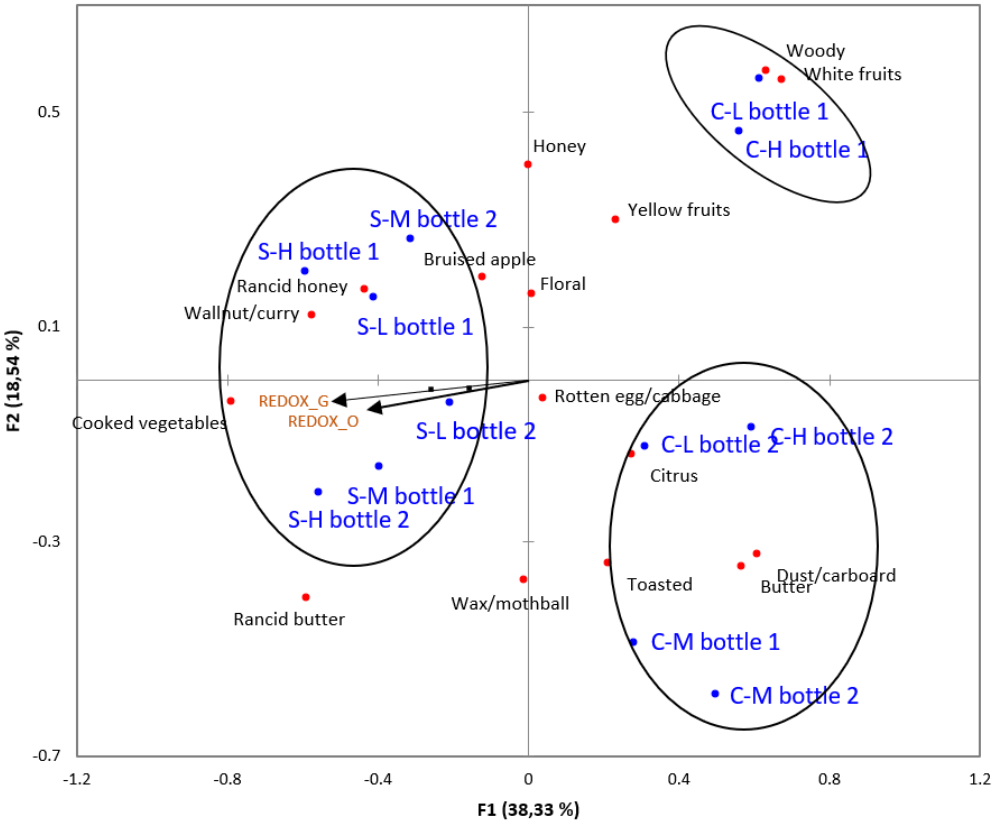

Figure 3. Correspondence Analysis of the general description of the two bottles of each 2010 sample. Ellipses indicate the clusters obtained by HCA using Ward's criterion. O: orthonasal; G: global.

3.2. Comparative Sensory Description of Synthetic Coextruded Stopper Samples

The strong oxidation differences observed between closures on the results of the monadic sensory description are likely to hide the subtle differences between turbidity levels (because of a contrast sensory effect). In order to better explore subtle differences due to turbidity levels, a comparative sensory assessment was carried out only on the three turbidity levels for the synthetic coextruded stopper samples.

The mean values for orthonasal and global REDOX odor scores are presented in Table 5. The results showed that there was a turbidity effect for 2010 samples in orthonasal ($F = 5.34$, $p = 0.011$) and in global ($F = 10.04$, $p = 0.001$) conditions, but no significant effect was found for 2009 samples ($F = 0.845$, $p = 0.44$ for orthonasal and $F = 0.942$, $p = 0.40$ for global conditions).

Table 5. Average scores for orthonasal and global REDOX odor assessment of the three turbidity levels for 2009 and 2010 vintages followed by Newman-Keuls' tests (α = 5%).

Sample Code	2009 Vintage		2010 Vintage	
	REDOX Orthonasal	REDOX Global	REDOX Orthonasal	REDOX Global
S-H	1.46 a	1.01 a	2.69 a	2.57 a
S-M	1.52 a	1.02 a	2.04 ab	2.17 a
S-L	1.93 a	1.48 a	1.47 b	0.93 b

Samples associated with the same letter were not significantly different.

Table 6. Results of the ANOVAS performed on the concentration of polyphenols for 2009 and 2010 samples. Probabilities in bold are significant at α = 5%. *F*: Fisher F-ratio; *Pr* > *F*: probability associated to the *F*-ratio.

Source of Variation	Phenolic Acids		Cinnamic Acids		Flavan-3-ols		GRP		Tyrosol		Total Polyphenols		TPI	
	F	***Pr > F***	***F***	***Pr > F***	***F***	***Pr > F***	***F***	***Pr > F***	***F***	***Pr > F***	***F***	***Pr > F***	***F***	***Pr > F***
vintage	224.9	<0.0001	3711.5	<0.0001	161.89	<0.0001	1334.9	<0.0001	14.05	0.002	3471.11	<0.0001	796.39	<0.0001
closure	3.3	0.09	15.7	0.001	26.05	0.0001	1.63	0.22	1.10	0.31	20.21	0.001	0.36	0.56
turbidity	32.8	<0.0001	0.16	0.85	0.36	0.70	116.6	<0.0001	309.26	<0.0001	35.44	<0.0001	6.68	0.009
vintage*closure	6.5	0.023	0.10	0.76	3.11	0.01	17.94	0.001	14.07	0.002	0.26	0.62	0.59	0.46
vintage*turbidity	2.0	0.17	30.0	<0.0001	0.36	0.706	7.30	0.007	66.77	<0.0001	7.94	0.005	6.38	0.011
closure*turbidity	1.0	0.38	0.28	0.76	0.76	0.484	3.29	0.067	0.468	0.64	0.15	0.87	1.27	0.31

Table 7. Results of the Newman-Keuls' test (α = 5%) performed on the concentration of polyphenols for 2009 and 2010 samples.

Main Effects	Phenolic Acids	Cinnamic Acids	Flavan-3-ols	GRP	Tyrosol	Total Polyphenols	TPI
vintage	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)
2009	4.54 a	57.47 a	1.30 a	3.56 a	23.49 a	90.37 a	8.39 a
2010	3.93 b	43.09 b	0.29 b	2.88 b	23.156 b	73.36 b	7.29 b
closure	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)
C	4.27 a	50.75 a	1.00 a	3.21 a	23.28 a	82.5 a	7.85 a
S	4.20 a	49.82 b	0.60 b	3.24 a	23.37 a	81.2 b	7.83 a
turbidity	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)	Average ($mg{\cdot}L^{-1}$)
L	4.13 b	50.28 a	0.84 a	3.35 a	24.81 a	83.41 a	7.93 a
M	4.47 a	50.21 a	0.77 a	3.29 b	23.01 b	81.75 b	7.84 ab
H	4.11 b	50.37 a	0.78 a	3.03 c	22.15 c	80.44 c	7.75 b

Samples associated with the same letter were not significantly different.

Table 5 shows that for 2010 samples and for both conditions, S-H was perceived as significantly more oxidized than S-L but not than S-M. As for the monadic profile, orthonasal and global REDOX odor scores were significantly correlated ($r = 0.95$, $p < 0.05$).

As for the general description, the results suggest that 2010 samples presented wider oxidation differences than 2009.

3.3. Phenolic Composition

The results of the ANOVA carried out on the phenolic compositions from both 2009 and 2010 vintages are presented in Table 6. Concerning the main effects, vintage reached significance for each class of polyphenols (phenolic acids, cinnamic acids, flavan-3-ols, tyrosol and GRP) and for total polyphenols and TPI. Table 7 shows the post-hoc Newman-Keuls' mean comparison of the average concentrations of all polyphenolic families for each on the main ANOVA factors tested. Table 7 shows that 2009 samples presented significantly higher concentrations for all the families of polyphenols after several years of bottle aging.

Screw caps showed higher concentrations for cinnamic acid, total polyphenols and Flavo-3-ols. However, the latter showed a significant interaction with vintage. Cinnamic acids and total polyphenols showed significant interactions with vintage. On the other hand, phenolic acids and GRP, the major polyphenols in chardonnay wines, were fairly similar between wines under cork or screw cap.

Concerning the effect of turbidity, significance was reached for the concentrations of phenolic acids, GRP, tyrosol, total polyphenols and TPI, but only phenolic acids showed an effect independent of the vintage, with the medium level of turbidity being slightly more concentrated than high and low levels.

3.4. Relationships between Sensory Characteristics and Phenolic Composition

We tried to predict the REDOX scores for orthonasal and global conditions for every vintage from their phenolic concentrations by means of multiple linear regressions. Only variables showing a correlation with the corresponding REDOX scores higher than 0.3 were considered in the linear regressions. The results of the stepwise linear regressions for both vintages and both conditions are presented in Table 8.

Table 8. Results of the multiple regression analysis carried out to predict REDOX odor character by phenolic concentrations. R^2: determination coefficient of the regression; t = Student value; $Pr > |t|$: probability associated with the absolute Student value. – indicates that the variable was not selected for the model because of an $r < 0.3$; ns indicates that the contribution of the variable to the predictive model was not significant. ns*: not significant.

Variable	REDOX 2009 OrthonasalR^2 = 0.26; p = 0.086		REDOX 2009 GlobalR^2 = 0.61; p = 0.014		REDOX 2010 OrthonasalR^2 = 0.94; $p < 0.0001$		REDOX 2010 GlobalR^2 = 0.92; $p < 0.0001$	
	t	*Pr* > |*t*|	*t*	*Pr* > |*t*|	*t*	*Pr* > |*t*|	*t*	*Pr* > |*t*|
Phenol Acids	ns*	ns	ns	ns	–	–	–	–
Cinnamic acids	–	–	ns	ns	ns	ns	–	–
Flavan-3-ols	ns	ns	−3.12	0.012	−10.93	<0.0001	−8.06	<0.0001
GRP	–	–	2.26	0.05	4.12	0.003	6.72	<0.0001
Tyrosol	–	–	ns	ns	–	–	–	–
Total polyphenols	–	–	ns	ns	ns	ns	–	–
TPI	ns	ns	–	–	–	–	–	–

Table 8 shows that no significant predictive model could be obtained from the 2009 orthonasal data. However, the predictive linear model for 2009 global was significant, and the ones for 2010 were very significant. The three significant predictive models were:

$$\text{REDOX 2009-global} = -3.54 - 0.93 \times \text{Flavan-3-ols} + 1.56 \times \text{GRP}$$
$$\text{REDOX 2010-orthonasal} = -1.932 - 4.08 \times \text{Flavan-3-ols} + 1.43 \times \text{GRP}$$
$$\text{REDOX 2010-global} = -3.63 - 2.35 \times \text{Flavan-3-ols} + 1.82 \times \text{GRP}$$

All three predictive models had basically the same pattern, with Flavan-3-ols as negative predictors and GRP as positive predictors. These results suggest that the presence of Flavan-3-ol can predict lower sensory oxidations, whereas high GRP levels predict higher oxidative notes. These results are particularly consistent between vintages for global perception.

4. Discussion

We implemented a combination of sensory and physicochemical analytical strategies in order to better understand the relationship between the wine polyphenol composition and the occurrence of oxidative notes. First of all, it is interesting to note that orthonasal and global assessment conditions were significantly correlated. This result is in agreement with previous findings [13]. However, as seen in Section 3.4, global evaluation enabled better regression models than orthonasal evaluations. One possible reason is that the in-mouth conditions in terms of temperature and contact surface with air favored the release of aroma compounds that were subsequently better perceived retronasally.

According to previous literature, strong vintage and closure effects were expected. Concerning turbidity, no particular hypothesis was formulated. The effects of the studied factors on the chemical and sensory characteristics of the samples are discussed in the following sections.

4.1. Effect of Vintage, Closure and Turbidity on the Wines Polyphenolic Content

The results showed a clear effect of vintage on the concentrations of all of the phenolic classes. Previous studies have already pointed out that for each vintage, a unique polyphenolic composition is conferred upon the resulting wine [26,28]. Flavan-3-ols, catechin and epicatechin were greatly impacted by closure compared to phenolic acids and GRP, likely due to their higher susceptibility to participation in slow oxidative processes during bottle storage [4]. Recent results have consistently shown that an unprecedented diversity of compounds could actually be involved in the discrimination of wine composition according to the type of closure [28]. Finally, must clarification level showed a significant effect on several polyphenolic classes, albeit largely modulated by vintage, but the mechanisms involved are still unclear.

4.2. Vintage Effect on REDOX Perception

Since the two vintages tested were quite different in terms of polyphenolic composition, vintages were analyzed separately for the sensory data. Taken together, the results confirm the importance of the composition of raw material, which is mostly vintage-dependent. Indeed, 2009 samples seemed more resistant to oxidation than those from 2010, while the latter showed wider sensory differences.

4.3. Closure Effect on REDOX Perception

The monadic evaluation clearly showed significant differences between closure types for 2010 samples, but not for 2009. It has been previously shown in the literature that wines closed with synthetic cork are more prone to oxidation than wines closed with screw caps [14,16]. The choice of the stoppers was made in order to increase the probability of having REDOX odor differences between samples. Concerning the general description of the 2009 samples, no segmentation by closure was shown. The 2010 samples showed a clear segmentation by closure type, with the samples closed with screw cap described as fruitier, more oaky and even showing a slight reductive aroma. On the other hand, the samples closed with synthetic cork showed clear signs of oxidation (“rancid honey”, “cooked vegetables”, “walnut/curry” and “bruised apple”). These results are consistent with previous research [13,14].

4.4. Turbidity Effect on REDOX Perception

The monadic evaluation of 2009 samples showed significant differences in turbidity for both conditions; unfortunately, significant turbidity*wine closure interactions prevent us from interpreting the main effects.

However, concerning the 2010 samples, the panel failed to show a significant turbidity effect, probably because the important differences between types of closures masked more subtle differences between turbidity levels.

A modification in the sensory protocol was implemented in a subsequent sensory session to confirm the existence of significant REDOX odor differences between turbidity levels for both vintages. As a result, a comparative assessment of the synthetic closures showed significant differences only for 2010 samples. Taken together, these results suggest for the first time that must turbidity could have an impact on the oxidative stability of white wines. To date, there are virtually no references concerning the relationship between must clarification and the polyphenol composition of the resulting wine, so the mechanism of this effect is still unclear, and further research is needed. One possible explanation is that, as seen in Section 4.1, must clarification somehow changes the polyphenolic composition of the wine, which could impact its sensory REDOX characteristics.

4.5. Effect of the Phenolic Composition on REDOX Perception

Multiple linear regressions showed significant predictive models of REDOX perception scores for 2010 data (both conditions) and for global perception of 2009 data. For all three models, our results suggest that the presence of Flavan-3-ol could predict lower sensory oxidations, whereas high GRP levels predicts higher oxidative notes. Such results provide an unprecedented model of the oxidation sensory note of dry chardonnay wine based on the concentration of two major polyphenol-related compounds, which is highly consistent with the literature. Flavan-3-ols and GRP have indeed already been described as being involved in oxidation reactions in wine, but never subjected to a sensory analysis. GRP is a well-known polyphenolic compound that appears preferentially during prefermentative steps, where non-protected musts lead to higher concentration of GRP due to the nucleophilic attack of the quinone of caftaric acid with the reduced form of glutathione [29]. Additionally, some studies have also shown that GRP continues to accumulate in wine during bottle aging under oxidative environments [30,31] without knowing exactly its origin. GRP is thus considered a final marker of an historical event of oxidation in must and/or wine. Similarly, the oxidation and browning of white musts have been shown to be largely correlated to the content of hydroxycinnamic acids and favored by the presence of Flavan-3-ols [32,33]. Additionally, Flavan-3-ols are slowly oxidized in wine in presence of oxygen and metal catalysts as clearly described by Danilewicz [5]. Therefore, our results shed new and interesting light on the impact that prefermentative management of musts can have on the later REDOX sensory note of bottle-aged dry chardonnay wines. In particular, they suggest that actions that reduce the production of GRP and maintain high levels of flavanols, such as must protection before alcoholic fermentation, could lead to lower oxidation notes.

5. Conclusions

The present study showed that the sensory oxidative character of bottle-aged chardonnay wines results from complex interactions between the different variables included in the experiment. It also showed the difficulty of generalizing the effects to different vintages. The polyphenolic composition was also quite vintage-dependent, and could partially explain the sensory differences between vintages. The main result of this study is that concentrations of Flavan-3-ols and GRP could both be significant predictors of wines oxidative sensory character, but with opposite effects.

Altogether, these results bring valuable new clues for questioning the impact of oenological practices—from prefermentative steps to bottling—on the subsequent development of oxidation, through the combined analysis of bottle aged wines.

Supplementary Materials: The following are available online at www.mdpi.com/2306-5710/4/1/19/s1. Table S1: raw data corresponding to the sensory sessions; Table S2: raw data corresponding to the polyphenols chemical analysis.

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