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Understanding the Mellowing Effect of Bottle Aging on the Sensory and Aesthetic Perceptions of Varietal Dry White Wines

Giovanni Marasà, Joana Ferreira, Mariana Mota and Manuel Malfeito-Ferreira *

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Abstract: The aging ability of dry white wines has been increasingly recognised. The present work aimed to understand which sensory factors drive their quality evaluation by experienced tasters. Individuals assessed several aesthetic, synthetic, and analytic flavour attributes, using dark tasting glasses. Wines originated from the grape varieties Alvarinho, Arinto, Sauvignon Blanc and Pinot Bianco with a wide range of ages. Basic physical-chemical analysis, browning (Abs 420 nm), elemental composition and a partial volatile fraction were also determined. The overall quality scores were a function of complexity and balance and were negatively influenced by the perception of faultiness. The different ages could be associated with a continuous sensory space characterised by a decreasing perception of freshness and an increase in the mellowed flavours. All wines shared an austere in-mouth perception elicited by their acidity, saltiness, bitterness, smoothness and dryness. Nevertheless, quality scores were similar from the youngest to the oldest-tasted wines (17 years old). The exception was a Sauvignon Blanc wine from a recent vintage that was judged as faulty due to the perception of earthiness. Overall, the tasted wines displayed an unexpected aging ability as demonstrated by the difference between the predicted and real wine ages. To improve the recognition of old wines, the metaphor "mellowed by age" is proposed to describe flavours resulting from beneficial aging instead of "oxidised by age".

Keywords: fine wines; aging bouquet; vertical tasting; maturation; quality; complexity; sensory space; aroma; mouthfeel

1. Introduction

White wines are not commonly subjected to aging. However, recent changes in the trend of wine appreciation have witnessed a renewed interest in aged dry white wines [1]. During aging, the wine undergoes noticeable modifications in chemical composition and sensory characteristics [2]. Wine is expected to improve during this process but is also often associated with a negative development of the sensory characteristics [3]. Concerning dry white wines, it appears that the peak in quality may be reached after 2 to 7 years, depending primarily on the grape variety and on the initial chemical characteristics [3,4].

The physical-chemical changes comprise volatile and non-volatile molecules such as acids, alcohols, aldehydes, esters, terpenes, thiols, phenols, lactones, or tannins [5]. The different variation in the type and concentration of these molecules determines the quality of the ageing process [6], where oxygen reactivity plays a major role [2]. In addition, polyphenols, iron, copper and sulfur dioxide influence the initial phases of non-enzymatic wine oxidation [7]. In white wines, aging is also characterised by increased colour browning as measured by the absorbance at 420 nm [8]. The oxidative off-flavours are claimed to appear before the onset of the browning effect [9,10].

The sensory changes during aging are mostly described by a loss of fruitiness and the development of off-flavours like acacia blossom or naphthalene [3] and toasted, oak, honey or farmfeed [11,12]. The onset of oxidative aroma degradation may also begin with the perception of cooked-vegetable nuances [9], being acceptable to lemmatise and group these sensory descriptors in a category described as evolution/old/oxidation [6]. The factors underlying these changes have been studied mainly using accelerated aging to understand the role of oxygen, chemical composition, closure type, light exposure or temperature on the process [2,5,13]. The purpose of these works has been mostly directed to understand the spoilage by oxidation [8] and not to evidence the high quality of aged wines, probably fostered by the high proportion (48%) of oxidation-related problems in several contexts [14]. Moreover, reports on the effect of commercial bottle maturation, or "élevage", are usually limited to relatively short periods [15,16], which is understandable due to sample availability. Albeit unintentionally, the abundance of research on faulty aging might have contributed to the common belief that dry white wines do not age properly. Therefore, the study of high-quality whites with uncommon long aging periods would be interesting to contradict widespread prejudices and provide scientific support to the present rediscovery of this style [17]. Indeed, a recent report demonstrated the recognition, by experienced tasters, of the sensory space and fine wine quality of samples up to 47 years old from different varieties, regions or styles [1]. However, this study did not test wines with the same origin and diverse ages under an approach that is popularly known as "vertical tasting" [18]. Therefore, the main objective of this work was to characterise dry white wines of different ages regarding their sensory features and selected physical-chemical parameters. To achieve this purpose, commercial dry white wines with a wide range of bottle aging duration were selected among regions and varieties expected to have proper "élevage" characteristics.

2. Materials and Methods

2.1. Wine Samples

Different companies generously provided commercial wines (0.75 L bottles) covering a range of available vintages (Table 1). The older vintages, no longer available in the market, were stored at each winery's facilities. All wines were sealed with natural cork stoppers. The selection of wine varieties was based on their anticipated aging potential, considering their initial relatively low odour intensity and austere mouthfeel (Alvarinho, Arinto, Pinot Blanc). Sauvignon Blanc was included as a representative of the international commercial style, known for its exotic fruity and vegetal flavours. The regions of origin were chosen for their susceptibility to cooling influences, whether from the Atlantic (Lisboa), altitude (Alto Adige), or a combination of both (Monção/Melgaço). Moreover, wines were vinified typically as whites, without skin contact, and with fermenting temperature control in stainless steel tanks, according to supplier information.

Table 1. White wines used in the study.

Origin[a]	Brand	Grape variety	Vintages	Sample code
Vinhos Verdes DOC[b], Sub-region **Monção and Melgaço, Portugal**	Soalheiro	Alvarinho	2014, 2017, 2019, 2022	AL14, AL17, AL19, AL22
Lisboa DOC, Portugal	Quinta do Rol	Arinto	2006, 2010, 2011, 2015, 2017	AR06, AR10, AR11, AR15, AR17
Alto Adige DOC, Italy	Kellerei Terlan	Pinot Blanc	2006, 2010, 2012, 2015, 2020	PB06, PB10, PB12, PB15, PB20
Lisboa DOC, Portugal	Adega Mãe	Sauvignon Blanc	2013, 2016, 2019, 2021	SB13, SB16, SB19, SB21

[a] Local coordinates and altitude: Soalheiro 42.097446, -8.309966, 400 m; Quinta do Rol 39.217346, -9.252108, 80 m; Cantine Terlan 46.530816, 11.251858, 400-900 m; Adega Mãe 39.048905, -9.295658, 100 m. Information retrieved from winery websites assessed on 6th July 2024: www.soalheiro.com/natural-factors/; www.quintadorol.com/; www.cantina-terlano.com/en/terroir/; www. adegamae.pt/terroir/?lang=en. [b] Protected denomination of origin.

2.2. Tasting Panel

The tasting panel was composed of 23 persons, including 10 wine critics and oenologists aged between 30 and 70 years old, 13 students, aged between 24 and 30 years old, and comprising 7 females and 16 males. The students with extensive training were selected among those registered in the second year of the Vinifera Master of Oenology and Viticulture held in ISA faculty. Four tasting sessions were held at the Microbiology Laboratory of ISA. The first occurred on the 20th of April 2023, dedicated to students. The other sessions (27th April, 4th and 5th May 2023) were dedicated to the professional judges. Individuals were not paid and provided written consent to participate in the study.

2.3. Wine Evaluation

Each tasting session included 3 flights of 6 samples each. The wines were kept at 12-14 ºC and opened 30 minutes before the beginning of the tasting. The bottles were first checked for cork taint by 3 experienced members of the staff. The samples were coded with a 3-digit number and randomly distributed among the judges. Twenty-five mL were poured at 12-14°C into dark glasses (Sensus, Zwiesel Kristallglas AG, Zwiesel, Germany) and covered by glass Petri dishes. Mineral water and unsalted crackers were available as palate cleansers. The tasting sheet was adapted from Esteves et al. [1] being divided into 3 parts. In the first part, judges provided personal demographic information (professional career, years of experience in the wine sector, type of wine education). The second part consisted of the evaluation of 7 synthetic and aesthetic attributes (Evolutionary state, Quality, Complexity, Body, Linger, Faultiness and Balance), using a 10 cm unstructured linear scale anchored at the extremes. This part also included a sample description using the Check-All-That-Apply (CATA) methodology for aroma and in-mouth flavour perceptions. Following the CATA list evaluation, tasters were asked to predict the age of the wine, still using dark glasses to maintain objectivity. In the third part of the session, tasters focused on evaluating the colour of the wine and predicting its age. Eight samples (the youngest and oldest for each wine variety) were poured into ISO transparent glasses [19]. Tasters then indicated the wine color using an unstructured linear scale, guided by reference pictures of wine colors, and provided their age predictions. A detailed tasting sheet can be found in Supplementary Material Figure S1.

2.4. Chemical Analysis

2.4.1. Standard Wine Analysis

Wines were analysed by FTIR spectroscopy (BACCHUS 3 Multispec, TDI, Barcelona, Spain) for alcoholic strength, total reducing sugars, pH, volatile acidity, total acidity, malic acid, lactic acid, following the OIV/OENO Resolution 390/2010 [20]. Free and total SO2 concentrations were determined using colorimetric titration by the Ripper method, following the OIV/OENO Resolution 377/2009 [21]. Samples were obtained by pooling wine from two bottles (100 mL) and each sample was analysed in duplicate ($n = 2$) for all chemical analyses.

2.4.2. Polyphenolic and Colour Determinations

Samples were centrifuged at 5000 rpm (10 minutes) and pipetted into 1 cm-path quartz cuvette. Absorbance (Abs) was determined at 280 nm (Abs 280), 320 nm (Abs 320) and 420 nm (Abs 420) by spectrophotometry (Agilent Cary 60 UV-Vis, Santa Clara, USA) for each sample. The Total Polyphenol Index (TPI) was determined by Abs 280 multiplied by the factor 20 corresponding to the sample dilution, according to Ribéreau-Gayon et al. (2006). Total hydroxycinnamates (TH) were quantified as caffeic acid equivalents by the formula TH = [(Abs 320-1.4)/0.9]*10, according to Sommers & Ziemelis (1985). Browning was given by the value of the absorbance measured at 420 nm.

The colorimetric analysis to obtain the CIELab coordinates (C*, H*, L*, a*, b*) was determined in the spectrophotometer Agilent Cary 100 UV-Vis (Santa Clara, USA) with illuminant D65 and observer placed at 10°. Samples were pre-treated by centrifugation at 5000 rpm for 10 minutes.

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Transmittances were acquired from 300 to 800 nm every 5 nm, in a glass cuvette with an optical thickness of 1 cm, using distilled water as a reference. The samples were analysed in duplicate (n = 2).

2.4.3. Elemental Analysis

The elemental composition was determined by an iCAP™ 7000 Plus Series (ICP-OES) inductively coupled plasma optical emission spectrometer (Thermo Fisher Scientific, Bremen, Germany), using Argon as gas, an Echelle optical design and a Charge Injection Device (CID) solid state-detector, as described by Harutyunian et al. [25]. Briefly, the instrumental conditions were 45 rpm for the analysis pump rate, 100 rpm for the flush pump rate, 0.7 L/min for the nebuliser gas, 12 L/min for the coolant gas, 0.5 L/min for the auxiliary gas and the plasma view was set in dual mode. To minimize possible matrix effects, suitable dilutions were made using 2% (v/v) nitric acid (HNO3).

2.4.4. Volatile Molecules Analysis

The volatiles were extracted following the method of Barata et al. [26]. Briefly, wine pH was adjusted to 8 with concentrated NaOH (Merck) and HCl (Merck) and mixed with 4 mL of ether–hexane with a magnetic bar for 5 minutes. The organic phase was obtained by 3 extractions using 2 mL of ether-hexane and refrigerated until used. Gas chromatography-mass spectrometry (CG-MS) data were collected using a Thermo Scientific TRACE 1300 gas chromatograph coupled to a Thermo ISQ mass selective detector. A DB-1 (30 m × 0.25 mm) fused silica capillary polar column with a 0.25 µm film thickness (Agilent, Santa Clara, California, USA) was used with helium as carrier gas (flow rate 1.0 mL/min). The injector temperature was 240 °C. Samples (0.5 µl) were injected using the split-less mode with a purge-closed time of 0.5 min. The oven temperature program started at 40 °C (not held) then increased at a rate of 7 °C/min to 220 °C, and held at this temperature for 10 min. The ion source temperature was kept at 180 °C, the transfer line was at 240 °C, and the mass spectra were obtained in the 50 to 500 m/z range, at an electron energy of 70 eV, with a delay of 3 min. Molecules were identified as TMS derivatives by comparing their (a) mass spectra with a GC-MS spectral library (Wiley Registry©/NIST) and (b) fragmentation profiles with published data [27,28]. The peak areas were determined and expressed as normalized relative percentages. The calculated composition was semi-quantitative/qualitative since no standards for each chemical family were co-injected or were their response factors determined. Each aliquot was injected in triplicate (n = 3).

2.5. Statistical Analysis

The synthetic scores did not follow a normal distribution and were analysed by the non-parametric Kruskal-Wallis test. Pair-wise comparisons were performed using the Tukey post-hoc test. For both tests, the α significance level was set as 0.05, with test p-values < 0.05 indicating significant differences.

Correlations among chemical and sensory parameters were calculated using the Spearman coefficient while Pearson coefficients were used to correlate chemical determinations. Correspondence analysis (CA) was performed based on the contingency tables of the descriptors quoted more than 10% of the time in at least one wine. Two cluster analyses were run, after data standardisation, using the Euclidean distance measure and the Ward.D clustering method, to obtain descriptor clusters (descriptors as rows and wines as columns) or wine clusters (wines as rows and descriptors as columns). The sensory and chemical data were submitted to Hierarchical Cluster Analysis (HCA) and Principal Component Analysis (PCA). Multifactorial Analysis (MFA) was used to describe the relations between selected chemical and sensory variables. Score comparison was obtained using the R-studio software and the various factorial analyses were run with the free statistical software Jamovi (version 2.3.28, www.jamovi.org, assessed on 1st June 2024).

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3. Results

3.1. Quality Prediction as a Function of Synthetic and Aesthetic Attributes

The scores of the synthetic and aesthetic attributes provided by the tasters are listed in Table 2. The dependent variables Quality, Balance, Evolution and Faulty showed significant differences among some of the wines, while Body, Complexity and Linger were similar among all samples. The variability of the scores was rather high as depicted by the box plots of Quality in Figure 1, by previous report using professional assessors [1]. Quality evaluation differed only in the Sauvignon Blanc wines, where sample SB19 was scored equal to SB16 and less than samples SB13 and SB21. Arinto did not show a difference in Quality along the vintages, while for Alvarinho and Pinot Blanc, there was a tendency to have lower scores with aging (p>0.05 for all wines of these varieties), as visualized in Figure 1.

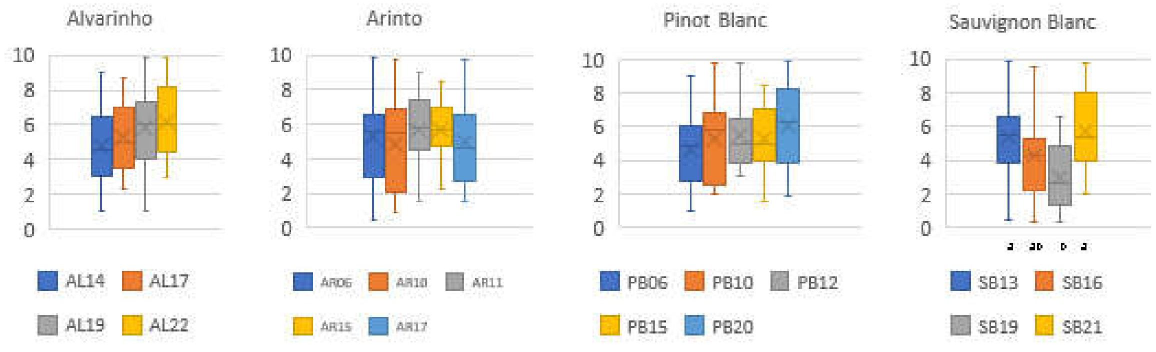

Figure 1. Box-plots depicting Quality score distributions among the tasted wines (different scores are indicated by different letters when p > 0.05).

Table 2. Mean scores of synthetic descriptors of white wines[a].

Sample	Quality	Balance	Evolution	Faulty	Body	Complexity	Linger
AL14	4.79 ab	5.30 ab	5.92 abcd	3.73 abc	4.63	4.94	5.71
AL17	5.26 ab	5.87 a	5.34 bcd	2.41 bc	4.44	5.09	5.09
AL19	5.82 a	6.12 a	5.34 bcd	2.73 abc	5.10	5.33	5.33
AL22	6.16 a	5.80 a	4.85 cd	1.50 c	5.32	5.46	5.86
AR06	5.27 ab	4.06 ab	6.93 ab	3.87 abc	4.83	6.12	5.57
AR10	4.80 ab	4.50 ab	7.08 ab	4.76 ab	4.97	5.45	5.31
AR11	5.74 a	5.61 a	5.76 abcd	2.47 bc	5.69	5.87	5.70
AR15	5.70 a	5.43 a	5.26 bcd	2.74 abc	4.96	5.53	5.55
AR17	4.90 ab	4.73 ab	6.44 abcd	3.99 abc	4.63	5.30	6.03
PB06	4.61 ab	4.17 ab	7.43 a	3.82 abc	5.32	4.81	5.55
PB10	5.27 ab	5.67 a	5.43 bcd	2.13 bc	4.72	4.38	4.20
PB12	5.55 a	5.53 a	4.88 cd	2.13 bc	5.47	5.20	5.81
PB15	5.28 ab	5.02 ab	5.19 bcd	2.18 bc	5.18	4.99	5.30
PB20	6.01 a	5.26 ab	4.63 d	1.92 bc	5.67	5.64	6.42
SB13	5.40 a	5.72 a	5.50 bcd	3.55 abc	5.63	5.50	6.17
SB16	4.26 ab	4.32 ab	6.34 abcd	3.53 abc	4.03	4.47	3.85
SB19	2.99 b	2.82 b	6.54 abc	6.03 a	4.00	4.63	5.14
SB21	5.66 a	5.56 a	5.33 bcd	2.91 abc	4.55	5.24	5.54
p- value *Kruskal Wallis*	0.002	0.001	3.56e-09	0.001	0.2425	0.213	0.100
p-value *Tukey- Kramer*	0.00065***	0.00066***	3.3e-09***	2.8e-05***	-	-	-

[a] Different letters in the same column indicate significant differences ($p < 0.05$).

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The measurements of Quality could be predicted as a function of the other synthetic and aesthetic attributes. The goodness of the prediction was assessed by the coefficient of correlation (r2). The following equations were obtained:

$$Quality = 0.36*Complexity+0.35*Balance-0.04*Body+0.08*Linger-0.18*Faulty-0.04*Evolution,\ r^2 = 0.592$$

$$Quality = 0.37*Complexity+0.34*Balance-0.04*Evolution-0.19*Faulty,\ r^2 = 0.588$$

$$Quality = 0.37*Complexity+0.35*Balance-0.19*Faulty,\ r^2 = 0.588$$

This analysis showed that quality could be fairly predicted just by the three variables Complexity, Balance and Faulty. The value $r^2 = 0.588$ indicates that the independent variables in the model explain about 58.8% of the variance of the dependent variable. Thus, the remaining 41.2% of the variance remains unexplained by the model, which could be due to other factors not included in the model or to random noise. These 3 variables have an aesthetic significance consistent with the definition of fine wines [29] and should be used more frequently to assess the "excellence" of wine quality [30].

3.2. The Effect of Age on the Synthetic and Aesthetic Attributes

The effect of wine age was first assessed by determining Spearman correlation coefficients among the median value of each synthetic attribute. The results listed in Table 3 showed that age was not well correlated with the scores given to these variables. Only a moderate positive correlation was found with Body (r = 0.490). This means that tasters recognised the positive attributes (Quality, Balance, Body, Complexity and Linger) independently from age. Table 3 also shows positive significant correlations among attributes with aesthetic significance like Quality and Balance or Complexity. Quality was negatively correlated with Faultiness while Balance was negatively correlated with Evolution and Faulty.

Table 3. Spearman correlation coefficients (r) among the median scores of synthetic or aesthetic attributes and age of the wines.

	Age	Quality	Balance	Evolution	Faulty	Body	Complexity	Linger
Age	—							
Quality	-0.100	—						
Balance	-0.367	0.522 *	—					
Evolution	0.418	-0.410	-0.490 *	—				
Faultiness	0.179	-0.614 **	-0.498 *	0.666 **	—			
Body	0.490 *	0.316	0.084	0.171	0.123	—		
Complexity	-0.137	0.623 **	0.188	0.049	-0.175	0.236	—	
Linger	-0.070	0.199	0.269	0.145	0.233	0.575 *	0.223	—

* $p < .05$, ** $p < .01$.

The former relations among the variables may be better understood using PCA. Figure 2 illustrates how the different attributes were distributed in the space. The variance was well explained (72.4%) by the first two components, with 49% to component 1 and 23.4% to component 2. Quality and Balance were opposed to Evolution and Faultiness while Complexity, Body and Linger were in the positive quadrant related to Quality. Interestingly, Age, as an illustrative variable, was closer to Evolution and Faultiness, indicating a tendency of the panel to prefer younger wines. This tendency was not observed by Esteves et al. [1], which might be explained by top-down inferences characteristic of expert tasters [29]. Indeed, when judges were informed that were assessing old whites, they penalised wines with younger sensory features [1]. On the contrary, in the present experiments, tasters were informed that were rating wines with a wide range of ages and so provided responses according to their quality perception irrespective of wine age. Whether individual liking,

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or preference, drove this quality judgement, is a question that remains to be clarified as discussed by Malfeito-Ferreira [31].

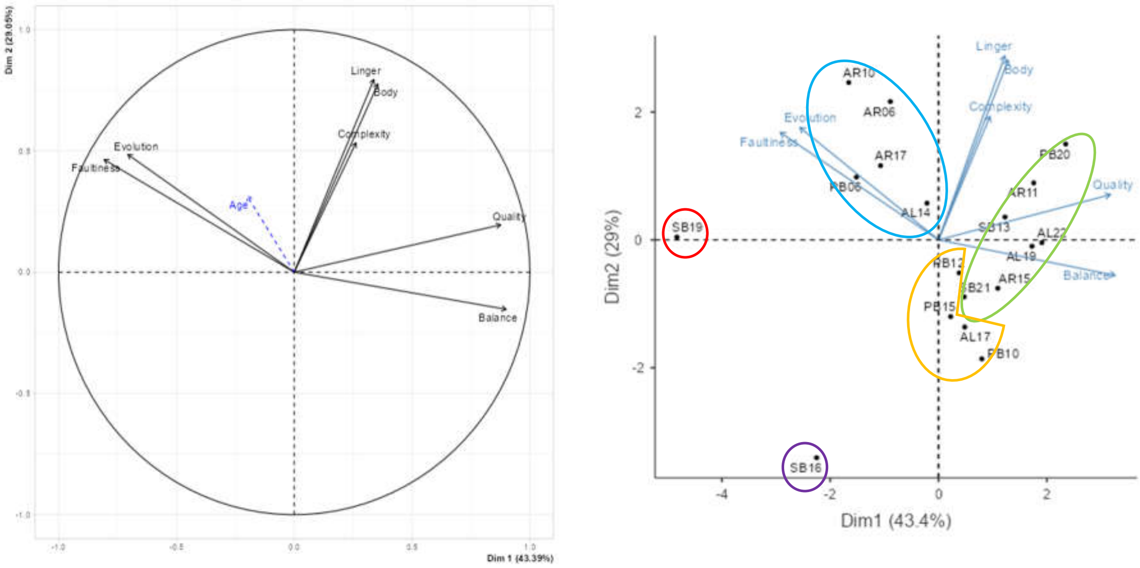

Figure 2. Principal component analysis of active (black) and supplementary (blue) variables (left panel) and biplot with active variables and wines (right panel). Wines are grouped according to Cluster Analysis.

A cluster analysis was conducted using all synthetic parameters as factors, allowing the grouping of wines based on the overall perception of their synthetic and aesthetic attributes (see Figure 2 for cluster definition). Wines SB19 and SB16 were distinct from the others due to negative perceptions related to faultiness and evolution, resulting in lower quality ratings for SB19. However, these negative perceptions were less pronounced in SB16, which received quality scores comparable to the rest of the wines (refer to Table 2). The remaining wines were clustered together regardless of their age, region, or grape variety. This indicates that tasters identified their synthetic and aesthetic properties across a diverse range of bottle aging durations and conditions. In particular, Sauvignon Blanc from 2013 showed that a variety typically reputed for its highly aromatic young character [32] may also age well, albeit more susceptible to vintage effects than other varieties, as illustrated by SB16 and SB19. Probably, minute concentrations of acetaldehyde play a role in the process by enhancing its fruity character with aging [33].

3.2. Aroma Taste and Mouthfeel Perception

The CATA methodology enabled to characterise of the wines according to their aroma and in-mouth flavour descriptors. The respective Contingency tables are shown in Supplementary material (Tables S1 and S2). The descriptors cited more than 10% were subjected to a Correspondence Analysis (wines as rows, descriptors as columns) yielding a Pearson Chi-square value of 535 (df = 442, p = 0.002) (Figure 3). The significance of the analysis was due to the aroma variables since the flavour ones did not provide significant sample discrimination ($p>0.05$) (results not shown), aligned with the data reported by Esteves et al. [1]. The Quality underscored sample SB19 was placed close to Earthy and Sourness, indicating the likely causes of its faultiness as detected during the synthetic evaluation (see Table 2). Indeed, other faults were not consistently detected since the free option to cite off-flavours was only mentioned twice for reduction in SB19 (results not shown).

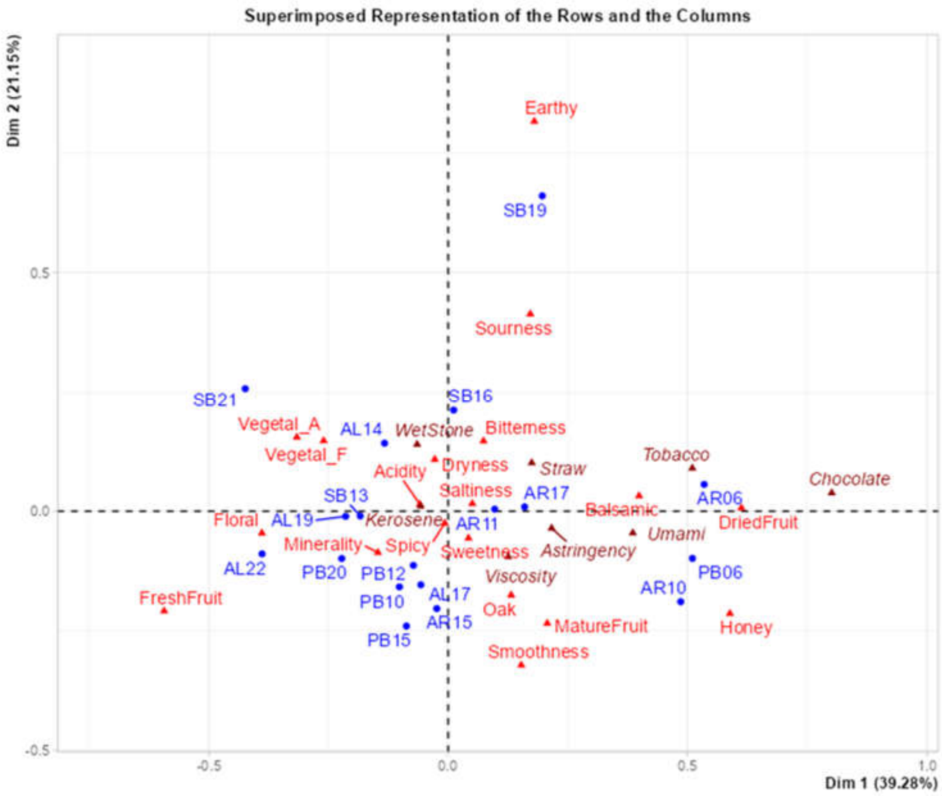

Figure 3. Correspondence analysis of aroma and flavour descriptors cited more than 10%, at least in one wine, as active variables (in red) and those less cited as supplementary variables (in brown).

The Cluster analysis combined the wines according to their sensory descriptors, when wines were computed as rows and descriptors as columns (Figure 4a). The wine SB19 was removed from this analysis since it was dominated by faultiness, blurring the observation of the effect of proper aging.

Another Cluster analysis was carried out, using descriptors as rows and wines as columns, showing 5 different clusters (Figure 4b). One cluster comprised flavours that could be understood under the concept of Freshness (Vegetal_A, Vegetal_F, Spicy, Sweetness, Floral, Fresh Fruit). Another cluster was dominated by in-mouth perceptions related to an Austere flavour (Minerality, Saltiness, Bitterness, Dryness, Smoothness, Mature Fruit). The cluster comprising Balsamic, Oak, Honey and Dried Fruit, Sourness and Earthy, could be described as the Mellowed group. Interestingly, Sourness and Earthy were clustered together within the Mellowed group while Acidity was clearly separated from all other clusters. In the free response alternative, oxidative aroma was only cited once, regarding AR06, and oxidative flavours were only used for the older wines (twice for AR06 and once for PB06), meaning that tasters did not significantly perceive these wines as oxidised.

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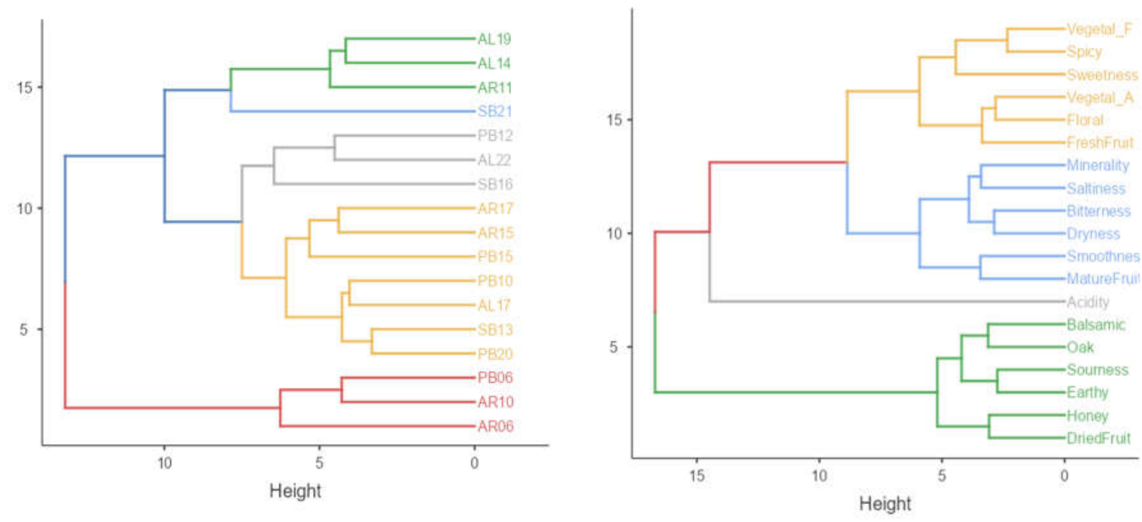

(**a**) (**b**)

Figure 4. Cluster analysis of wines grouped according to (a) their CATA characterisation and (b) their sensory descriptors according to wines.

The frequency of citations of the descriptors gathered according to cluster analysis enabled us to observe the evolution of the sensory cluster families, calculated for each of the wine clusters discriminated in Figure 4 (Supplementary Table S3). The evolution is depicted in Figure 5 using the frequency of citation as a percentage. Wine SB19 was not included since it was regarded as defective. Thus, the sensory space of proper aging may be described by a decrease in freshness, accompanied by an increase in the mellowed descriptors and austere character. The perception of acidity tended to decrease which may be understood as a result of the increasing mellowed perception since chemical acidity did not change (section 3.4.1). This flavour continuum corresponds fairly well with the results described by Esteves et al. [1], while it adds the perception of faultiness to the conceptual space. Moreover, the vegetal descriptors, either in aroma (Vegetal_A) or in flavour (Vegetal_F), more frequently quoted in the present work might be explained by the influence of Sauvignon Blanc wines [34]. Interestingly, the earthiness in SB19 might be due to higher levels of methoxypyrazines [33] but also to volatile thiols, since earthy aromas dominated de-aromatized wines spiked only with these molecules [34].

Regarding tastes, acidity and sourness are considered synonyms, but acidity was more frequently quoted (Supplementary Table S3) while sourness appeared closely related to earthy. This might be explained by the alternative meaning of sourness, as related to an unfavourable taste [35], and thus linked to the negative earthy perception.

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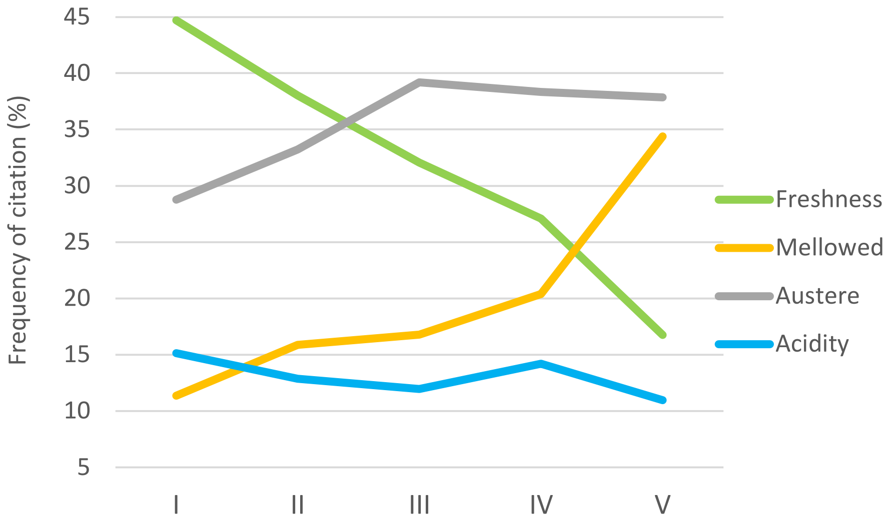

Figure 5. Frequency of citation (%) of flavour families according to the wine clusters (I, SB21; II, AL22, SB16, PB12; III, AL17, AR17, AR15, PB20, PB15, PB10, SB13; IV, AL19, AL14, AR11; V, AR10, AR06, PB06).

3.3. Age Prediction

After sensory description, assessors were asked to predict the age of each wine poured in dark glasses. Therefore, age prediction was only the dependent on the individual memory of the aroma and flavour perceptions and not biased by the observation of colour. Figure 6 shows the age prediction as a function of the freshness and mellowed perceptions (SB19 was not used). The decay in freshness was accompanied by the rise in the citation of mellowed flavours, demonstrating that it was the balance between both perceptions that determined the evolutionary stage of the wines. The intersection between both regression lines (close to 7 years) might be understood as the predicted age where the wines change from the dominant fresh flavour to the mellowed ageing character. Coincidently, Monforte et al. [36] established a priori this value as the limit to separate young (2-7 years) from old dry whites (> 8 years).

The results of the average predicted ages are listed in Table 4, showing a clear trend to rate the wines with ages lower than the actual age, except for the younger wines (AL22, AL19, AR17, PB20, SB19, SB21), as primarily observed by Esteves et al. [1]. The case of SB19, where the predicted age almost doubled the real age, may be explained by faultiness detected in this wine. Nevertheless, the Pearson correlation between the actual age and the predicted age was good ($r = 0.727$, $p < 0.001$, without SB19). The higher differences in predictions were observed in the older wines of each variety, showing that tasters were not familiar with their sensory features.

Table 4. Average age prediction according to the visual condition and respective colour score.

Sample	Real age	Predicted age (dark glass)	Predicted age (transparent glass)	Colour score (cm)
AL14	9	5.6 ± 4.6	5.4 ± 3.8	5.2 ± 1.9
AL17	6	4.3 ± 3.2	-	-
AL19	4	4.7 ± 3.8	-	-
AL22	1	3.5 ± 2.9	1.8 ± 1.1	1.3 ± 1.5
AR06	17	10.1 ± 7.3	10.0 ± 4.5	7.6 ± 1.5
AR10	13	9.8 ± 6.2	-	-
AR11	12	5.4 ± 2.5	-	-
AR15	8	5.8 ± 4.8	-	-
AR17	6	7.2 ± 3.0	4.7 ± 2.5	4.3 ± 1.4

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PB06	17	9.0 ± 4.9	6.4 ± 4.9	5.7 ± 1.8
PB10	13	5.0 ± 3.8	-	-
PB12	11	3.7 ± 2.1	-	-
PB15	8	4.1 ± 2.9	-	-
PB20	3	4.1 ± 2.6	3.2 ± 3.2	1.8 ± 0.9
SB13	10	5.7 ± 4.4	3.4 ± 1.6	3.3 ± 1.0
SB16	7	6.0 ± 3.4	-	-
SB19	4	7.8 ± 7.2	-	-
SB21	2	4.2 ± 3.7	2.1 ± 1.3	1.8 ± 0.8

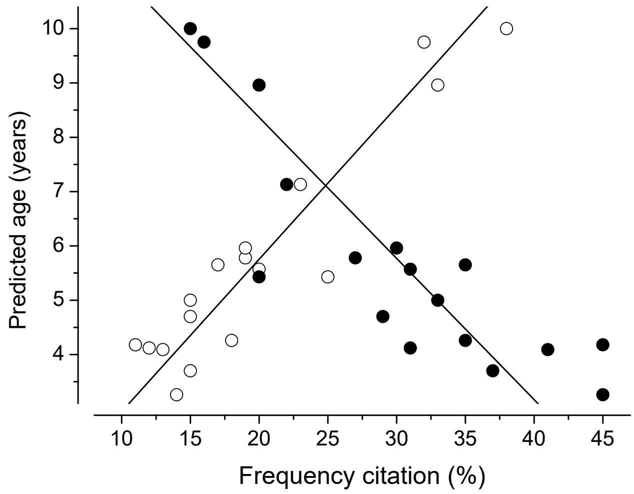

Figure 6. Frequency of citation of the flavour clusters Freshness (●) and Mellowed (○) for all white wines except SB19. Regression lines: Freshness, slope -3.85 ± 0.59, r = - 0.860, p < 0.0001; mellowed, slope 3.60 ± 0.34, r = 0.939, p < 0.0001.

At the end of tasting trials, individuals were also asked to predict the age of the youngest and oldest sample for each grape variety using transparent glasses. In parallel, a scale anchored by figures of glasses with the different colours (Supplementary Figure S1) was used to provide a colour score. The average results are presented in Table 4. The predicted mean age in transparent glasses tended to be lower than the real age in 5 out of the 8 wines, which demonstrates the bias induced by colour on sensory description. Probably, tasters would have described the wines with a younger character had they been tasted in transparent glasses.

The relation between predicted wine age and colour score shows that the panel correctly understood the variations in colour and provided sensory descriptions consistent with the colour, despite the high individual variability in the responses (Figure 7). Indeed, the correlation between real and predicted age scores was very good (r = 0.966, slope 1.15 ± 0.13, p < 0.0001). Then, the difference between both ages may be used as an empirical measure of the quality of the aging process. In relative terms, the higher the difference, the better wines had aged since they kept the sensory profile of a younger wine, as initially proposed by Esteves et al. [1].

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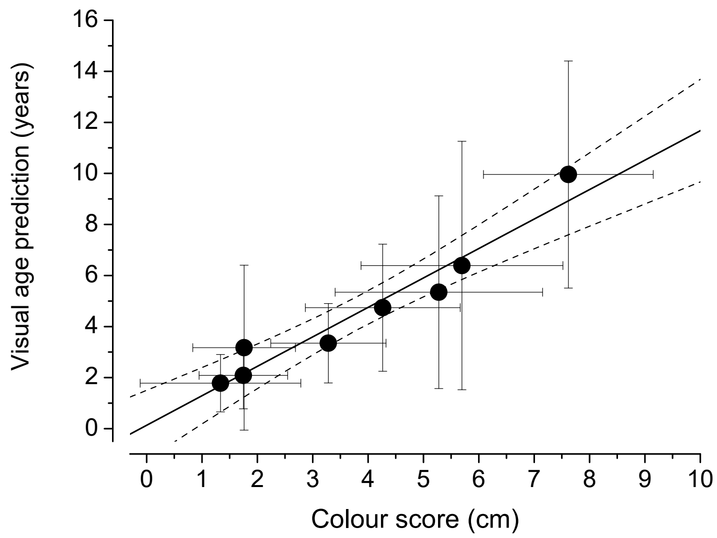

Figure 7. Relation between the average predicted age and the average colour score of the oldest and youngest wine of each grape variety (-, linear fit; ---, 95% confidence limits; vertical and horizontal bars, standard deviations).

3.4. Chemical Analysis

To assess the chemical differences according to wine age, Pearson correlation coefficients were first calculated between age and various physical-chemical compositions. When warranted, a PCA followed by hierarchical clustering was employed to identify similarities among the different wines. This method was not designed to elucidate the effect of age on chemical compositions, as factors like initial wine compositions, bottling variables (e.g., cork quality, sulfur dioxide additions), and storage conditions (e.g., temperature) were not standardized. Therefore, the results do not represent the evolution of a specific wine over time but can be viewed as an illustrative example of how each varietal wine evolves under its respective winery's conditions. Despite these limitations, this "vertical tasting" provided samples that reveal an overall chemical behaviour resulting from aging, offering valuable insights for future research.

3.4.1. Standard Analysis

The results of the standard wine analyses are shown in Supplementary Table S4. The range of values are within the expected range of values for dry white wines. Ethanol varied from 11.9 to 14.5 % (v/v) while total acidity ranged from 4.5 to 6.6 g/L (as tartaric acid). Volatile acidity varied from 0.24 to 0.38 g/L, well below the legal limit (1.2 g/L as acetic acid, www.oiv.int/standards/international-code-of-oenological-practices/annexes/maximum-acceptable-limits, assessed on 6th July 2024). Total sulphur dioxide was also well below the legal limit (220 mg/l). The residual sugar found in some samples was over 4 g/L. According to European Union standards, wines with residual sugar over 4 g/l and total acidity (as tartaric acid) less than 2 g/L of the residual sugar are regarded as medium-dry [37]. These medium-dry wines might represent a technical option to counteract wine total acidity [38] and meet the consumer demand for smoother wines [39], as typically observed in German Riesling wines [16]. Interestingly, in Pinot Bianco, the lower sugar concentration in the three most recent vintages might be explained by the changes in wine appreciation, where drier and leaner styles are becoming more valorised [40].

The effect of age on the standard chemical parameters would not be expected since bottled commercial wines are supposed to be stable in this regard. Indeed, significant but low correlations were only observed for volatile acidity volatile acidity (r = -0.494) and lactic acid (r = 0.592) (Supplementary Table S5) without any likely influence on sensory parameters, given their relatively low concentrations.

3.4.2. Polyphenolic and Colour Analysis

The polyphenolic determinations are shown in Supplementary Table S6 while the Pearson correlations coefficients are shown in Supplementary Table S7. The best-correlated parameter with wine age was the absorbance at 420 nm, which is an indicator of colour browning [12]. The TPI, HA and the CIELab parameters displayed lower correlations demonstrating that do not perform as well as the Abs420 as a measure of colour evolution according to age. The positive relation between Abs 420 and b* (yellowness), or negative concerning L* (clarity), was relatively low, probably because aging was not forced by high temperature as experimented by Mafata et al. [12]. Filipe-Ribeiro et al. [41] also used the value of b* to model the oxidation induced by forced aging. The positive significant relation between Abs 420 and TPI, was lower than that found by Ricci et al. [42] using accelerated aging by temperature. In addition, to estimate white wine shelf life, Ferreira et al. [43] calculated an index of resistance to oxidation that was better related to sensory degradation than Abs 420, under forced aging. Overall, these results suggest that forced aging may not be an accurate way to predict proper wine aging under real conditions.

Figure 8 illustrates the browning effect according to age that achieved a relatively high correlation ($r = 0.816$, slope 0.016 ± 0.003, $p < 0.0001$) since it was obtained for wines with different origins, grape varieties, chemical compositions and storage conditions. Monforte et al. [36] also found a high correlation ($r = 0.73$, $p < 0.01$) between age and Abs 420, in parallel with acetaldehyde formation. Interestingly, in the early report of Simpson [4], the increase in Abs 420 up to 0.210 in the older wine (a Riesling with 10 years from Eden Valley, South Australia) was described as an evolution in colour from straw to deep yellow but not associated to browning.

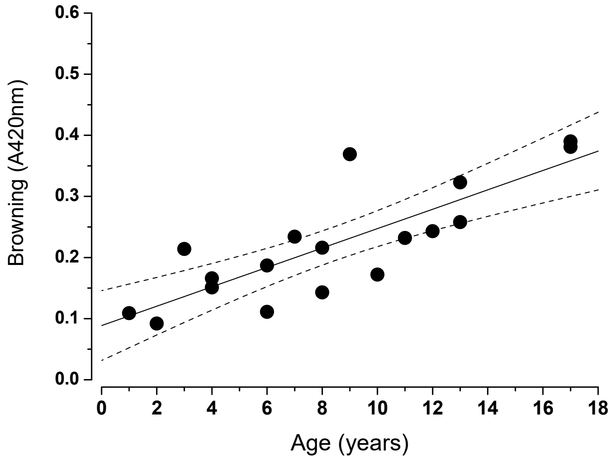

Figure 8. The effect of age on wine browning measured as the absorbance at 420 nm (-, linear fit; --, 95% confidence limit).

In addition, the changes in flavour clusters relative to browning and predicted age are illustrated in Figure 9. The two younger wines (SB21, AL22) were the fresher and, accordingly, the less deeply coloured. However, higher Abs 420 was observed with relative low mellowed flavours and low predicted age (wine AL14). Therefore, sensory changes do not always anticipate the browning effect, as described by Escudero et al. [9] and Marrufo-Curtido et al. [10]. The explanation may be related to: (a) the chemical changes that precede browning and may elicit younger perceptions due to the masking effect of certain volatiles; (b) the utilisation of taste and mouthfeel perceptions that may enhance the sensation of freshness. Overall, the tasting sequence, where in-mouth perceptions preceded aroma description, might have minimised top-down effects induced by “oxidised” aromas. Simultaneously, the use of dark glasses prevented the likely occurrence of biases induced by colour [44,45].

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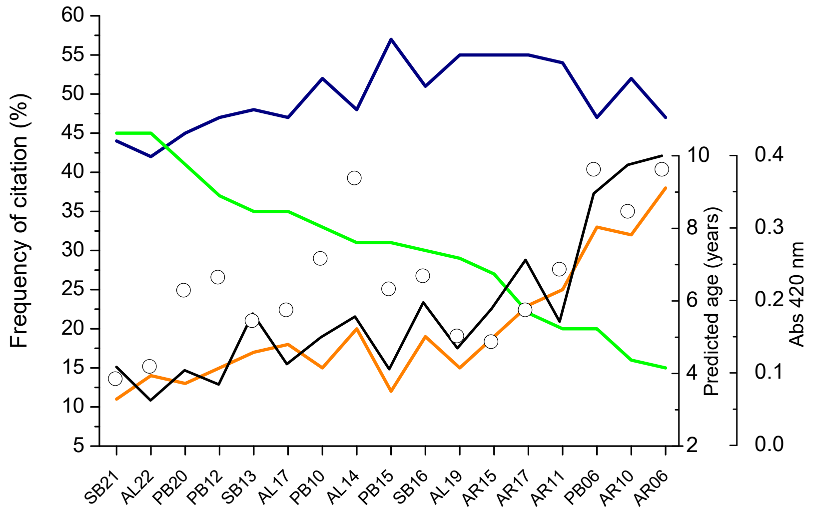

Figure 9. Flavour and colour changes in wines tasted in dark glasses (flavour families: Freshness, green; Mellowed, brown; Austere/Acidity, blue; predicted age, black; Abs420 nm, open circles). Wines were aligned according to decreasing values of Freshness perception.

3.4.3. Elemental Analysis by ICP-OES

The concentration of both the macro-elements (Na, K, Ca, Mg, P and S) and the micro-elements (Fe, Cu, Zn, Mn, B, Pb, Cr, Ni and Cd) are shown in Supplementary Table S8. The Pearson correlation coefficients between elements and age are shown in Table 5. Significant correlations were only observed in Alvarinho (K, Ca, Fe and Zn) and Pinot Bianco (B) wines. Other correlations were also either positive or negative according to the grape variety. In the case of K and Zn, positive correlations were found for all varietal wines, with different magnitudes, justifying their use in the Multifactorial Analysis (section 3.5). Cu and Mn showed negative and positive correlations, respectively, but with rather low r values (Table 5).

Table 5. Pearson correlation coefficients among the age and the elemental composition.

Element	Alvarinho	Arinto	Pinot Bianco	Sauvignon Blanc
Na	0.810	0.828	0.874	-0.139
K	0.978*	0.498	0.573	0.193
Ca	0.988*	0.388	-0.665	0.182
Mg	0.914	0.552	-0.508	-0.883
P	0.941	-0.185	-0.275	0.245
S	0.759	0.482	-0.348	-0.089
Fe	0.966*	0.440	-0.196	-0.219
Cu	-0.255	-0.380	-0.073	-0.403
Zn	0.960*	0.721	0.400	0.712
Mn	0.230	0.369	0.311	0.161
B	0.901	0.808	0.961**	-0.666
Pb	0.253	0.032	-0.145	0.098
Cr	0.928	-0.393	0.214	-0.753
Ni	-0.466	0.754	0.800	0.686
Cd	-0.601	-0.324	0.092	-0.740

* $p < .05$, ** $p < .01$.

Agazzi et al. [46] reported that the Ca and K concentrations decreased while Na and Mg concentrations increased after 5 years of aging in Malbec wines from Argentina. The depletion of K and Ca was explained by the precipitation as K bitartrate and Ca tartrate, respectively, but this effect

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should have not occurred in the bottles used in the present work since no visible crystals were present.

Gambetta et al. [47] associated higher quality of Chardonnay wines with higher levels of Cu and Zn in juices. Ferreira et al. [48], using red wines subjected to accelerated aging, found positive correlations between Zn and aldehyde formation while the oxidative degradation of S-reduced molecules (methanetiol) was negatively related with Cu ($r = -0.59$, $p < 0.05$), Fe ($r = - 0.67$, $p < 0.01$) and Mn ($r = -0.67$, $p < 0.01$).

Furthermore, SB19 sample showed the highest concentration of S, which could be related to the earthy off-flavours detected by the judges, since volatile thiols might have been involved [34]. Na levels were higher in the wines closer to the ocean (Arinto and Sauvignon Blanc, Lisbon DOC), but this observation needs further investigation since soil composition and rootstock are other modulators of Na concentration [49].

Elemental analysis has typically been used to differentiate wines based on their origin rather than their vintage [50]. The HCA using as cut-off the value corresponding to 5 clusters, showed that Pinot Bianco and Sauvignon Blanc could be separated from the other wines independently from wine age (Figure 10). Two clusters included only 2 wines from Arinto and other 2 from Alvarinho. On the contrary, another cluster comprised Arinto and Alvarinho, irrespective of their age, indicating the absence of a vintage effect, in accordance to Duley et al. [50].

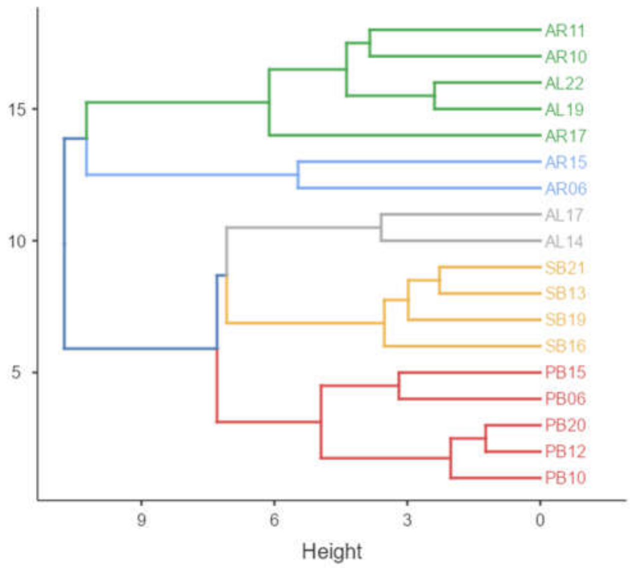

Figure 10. Hierarchical clustering dendogram of the wines according to their elemental constitution.

3.4.4. Analysis of Volatile Molecules

The results of the GC-MS analysis are shown in Supplementary Table S9, where a total of 88 molecules were detected among the various wines and vintages analysed. To understand the effect of age on the volatile composition, the Pearson correlation coefficients were determined. Only those molecules showing a significant correlation ($p < 0.05$) in at least one variety were kept (Supplementary Table S10). Most volatiles showed a different trend at least in one grape variety. Significant trends (positive or negative) for all varieties were observed only for isoamyl acetate, diethyl succinate, ethyl linoleate, stearic acid and 2-propenal, 3-(2,6,6-trimethyl-1-cyclohexen-1-yl).

The aldehyde related to propenal has been seldom mentioned in wines. Nicolli et al. [51] found it Merlot wines, using polar and medium polar columns, probably contributed to their herbaceous aroma. Isoamyl acetate and diethyl succinate showed negative and positive, respectively, age trends according to the seminal work of Simpson [4]. Makhotkina et al. [52] also showed similar trends with isoamyl acetate and diethyl succinate, stimulated by temperature in Sauvignon Blanc. In addition, isoamyl acetate showed negative correlations with the age of several commercial dry white wines

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[36]. Indeed, ethyl esters of fatty acids and acetate esters tend to hydrolyse, whereas the ethyl esters of branched acids may be formed during storage in the bottle [53] and references cited therein). Ethyl linoleate has been found in elevated levels in rot-affected grapes [54] but its evolution over time has not been reported. Similarly, stearic acid, a constituent of Sauvignon Blanc juices [52], was not detected in the older vintages.

Other molecules proposed to be markers of dry white wine aging [36,55,56] were not determined in this work or showed different behaviours according to the variety. Vitispirane increased during refrigerated [4] or forced aging [57] but our results failed to show this behaviour in Pinot Bianco. Interestingly, the expected decrease of linalool with aging [4] was also not observed in Pinot Bianco ($r = 0.951$, $p < 0.05$). These results illustrate the complexity of the age effect on volatile composition, further enhanced by the complexity of sensory perception since flavour deterioration by some volatiles might be masked by the increase in others [55].

3.5. Multifactorial Analysis (MFA)

The utilisation of MFA analysis enables us to observe which might be the contribution of each chemical and sensory dimension to the quality evaluation of the tasted wines. MFA analyses the outputs in several sets of variables, seeking the common structures present in all or some of these sets [58]. To reduce the noise of the outputs, several assumptions were taken given their previously described relation to wine age. The sensory parameters were reduced to the 4 flavour families and 4 synthetic attributes (quality, complexity, balance, faulty). The standard chemical parameters were not used while Abs420 was the single determination retained from the polyphenolic analysis. The elemental analysis provided the K and Zn concentrations while only 4 volatile molecules were retained. Stearic acid, although with positive correlations for all wines, was not kept since 9 out of 18 determinations were below the detection limit. Regarding wines, only those with equal Quality scores were kept so SB19 was removed. Therefore, the results of MFA might provide clues about the relation among the variables underlying proper aging.

The first MFA was run using age perception as an a priori set of variables. Three groups were obtained from clusters I and II (young), II and IV (mature) and V (mellowed) presented in Figure 5. The localisation of the sets of variables on the biplot is presented in Supplementary Figure S2 and the contribution of each quantitative variable to the 2 first dimensions is shown in Supplementary Figure S3. The biplot illustrating the localisation of the quantitative variables is shown in Figure 11a. This plot clearly illustrates the opposite localisation of the fresher character of younger wines (left quadrants) against the mellowed character of older wines (right quadrants), under an explained variance of 58.5% for dimensions 1 and 2. Thus, the younger profiles tended to be more valorised, as described by Franco-Luesma et al. [6] based on the orthonasal aroma of white wines spiked with oxidation-related volatiles. The mellowed perception, despite being associated with faultiness, did not elicit a significant drop in the quality scores. The localisation of austere in the middle of the plot means that this flavour, dominated by in-mouth perceptions, did not contribute to the separation between ages and might be the cue to understand the propensity of a wine to age without losing quality. The chemical factors were distributed in the plot according to the previously described correlations, showing the higher associations of isoamyl acetate with freshness and diethyl succinate, A420 and Zn with the age character.

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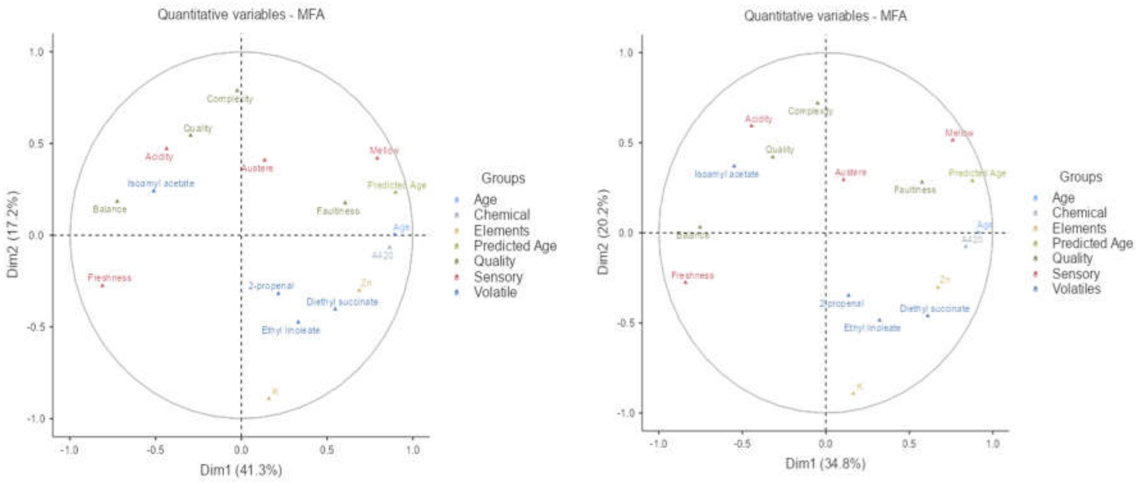
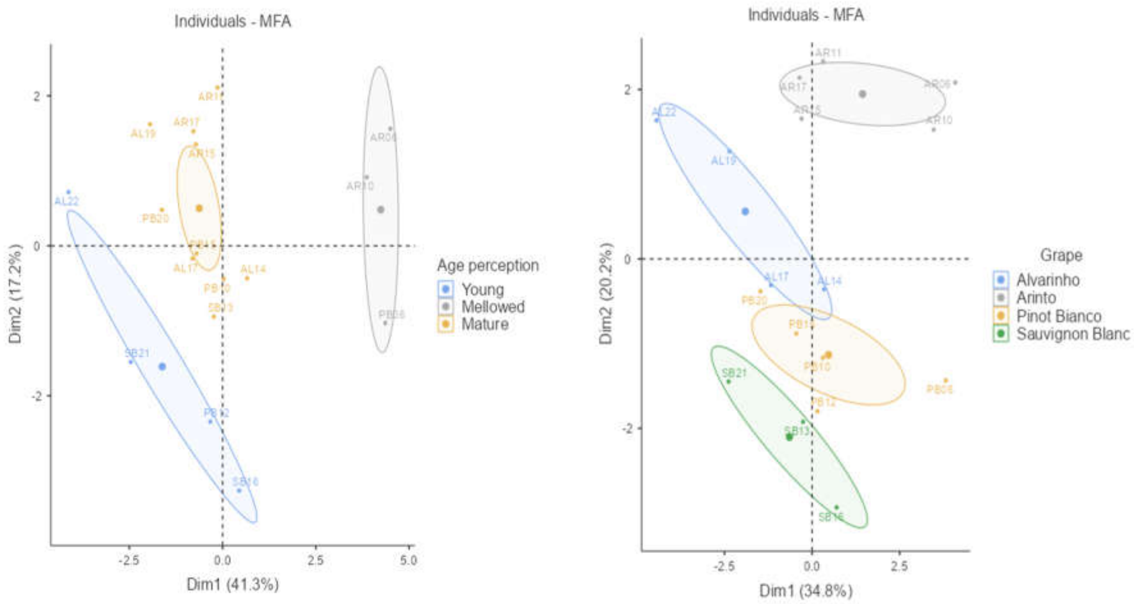


(**a**) (**b**)

Figure 11. Biplot of Multiple Factorial Analysis illustrative of quantitative variables associated to proper aging using wines grouped by age perception (a) and grape variety (b) (2-propenal stands for 2-propenal, 3-(2,6,6-trimethyl-1-cyclohexen-1-yl).

MFA may also provide a comparative characterisation of the wines adding the grape variety as one a priori set of variables instead of age perception. Figure 11b shows that the localisation of the quantitative variables is similar in both cases, although the explained variance by both dimensions decreased slightly to 55%. The localisation of the sets of variables on the biplot is presented in Supplementary Figure S4 while the contribution of each quantitative variable was mostly similar to the previous one (results not shown).

The main differences between the MFA's computed with age perception or grape variety as a priori set of variables are illustrated in Figure 12. The outputs provided wine clusters according to this set of variables. The age perception clusters gathered wines from different varieties (Figure 12a) while grape variety clusters gathered wines with different ages (Figure 12b). Alvarinho and Pinot Bianco were placed closer in the biplot while Arinto and Sauvignon Blanc were placed in opposite locations. MFA explained a slightly higher percentage of variance when wines were grouped by age perception instead of grape variety.



(**a**) (**b**)

Figure 12. Biplot of Multiple Factorial Analysis illustrative of wines grouped by age perception (a) and grape variety (b) using confidence ellipses.

4. Limitations and Future Prospects

This report represents an extension of the work initiated by Esteves et al. [1] and was designed considering the limitations pointed out. Nevertheless, other issues may also be questioned. The studies of dry white wine aging are usually directed to oxidation problems and forced tests are used to analyse wines under standardised conditions. The option is understandable from the scientific point of view since influencing variables must be individualised. However, this requirement limits the utilisation of wines from a wide range of vintages where the initial and storage conditions can hardly be standardised. Therefore, the primary limitation of this work could not be overcome by choosing equal aging conditions for all commercial wines. Nevertheless, an overall sensory evolution with age could be described by a continuous sensory space, according to previous results [1]. Other varieties and regions should be tested in future to validate the present results. In addition, the boundaries of the sensory space would be better defined if very old "flat" wines were used, mainly concerning the effect on the in-mouth austere perception that seems to be the key to understanding wine aging propensity.

This work showed that tasters did not recognise oxidation as an aging marker, although they recognised the evolution and faultiness of the wines. It may be hypothesised that the absence of oxidation from the CATA list would preclude its detection, although it could have been chosen as a free citation, as described by Franco-Luesma et al. [6]. In opposition, it may also be posited that, if oxidation was present in the CATA list, it would have blurred the quality evaluation. Indeed, even when properly aged samples are used (up to 37 years old), the studies usually mention the "oxidized character" to describe the age effect of dry white wines [36]. This feature may have the unintentional drawback of associating proper ageing with unwanted or premature oxidation. In certain cases, the absence of fresh fruity ("not fruity") may be understood as an indication of oxidation and consequent hedonic negative perceptions [6], but this inference is not sufficient as a quality assessment [30]. Interestingly, the early report of Simpson [4] described "bottle age" aroma as being more intense in the older wines with minor differences in grape bouquets, oxidised aroma and quality assessments. Probably, the subsequent lines of research grounded on forced aging favoured the oxidation character as a more appropriate descriptor, despite its negative connotation. Therefore, in studies aimed at studying the evolution of fine dry white wines, the utilisation of the metaphor "mellowed by age" appears to be more suitable than "oxidised by age".

5. Conclusions

This work explored the changes during bottle ageing of white wines from a sensory and chemical point of view. The results contributed to the understanding of how white wines develop, how they are perceived and how they change over time. The chemical parameter better correlated with aging was the absorbance at 420 nm. The untargeted analyses of volatile compounds illustrated the complexity of their contribution to the aging sensory character of white wines.

The professional panel members tended to associate the increase in bottle ageing with the lowering of quality perception and the increase in the perception of off-flavours. Nevertheless, tasters agreed on the evolution of the wines and did not underscore quality unless they perceived strong faultiness. Furthermore, the difference between perceived and real age may be understood as a measure of the aging propensity. If wines are judged younger, they aged properly for more years than anticipated.

The results here presented illustrated the evolution of the sensory space during aging, from decreasing perception of freshness to an increase in the mellowed perception, keeping constant an austere character. This continuum represents an evolving sensory space, a description which may be used in educational programs to widen the range of perceptions of fine wine quality among professionals and consumers.

Supplementary Materials: The following supporting information can be downloaded at the website of this paper posted on Preprints.org. Figure S1. Tasting sheet (miniaturised from size A4); Table S1. Frequency of citation of aroma descriptors of all tasted wines (descriptors used in correspondence analysis are written in bold); Table S2. Frequency of citation of flavour descriptors of all tasted wines (descriptors used in correspondence analysis are written in bold); Table S3. Frequency of citation of wines clustered according to their sensory descriptors; Table S4. Standard wine physical-chemical analysis; Table S5. Pearson coefficients of correlation among standard physical-chemical analysis; Table S6. Polyphenolic and CIEelab determinations¸ Table S7. Pearson coefficients of correlation among spectrophotometric and CIElab colour determinations; Table S8. Elemental composition of the analysed wines (mg/L); Table S9. Concentration of volatile molecules of the analysed wines (mg/L) (value 0 corresponds to non-detected molecules); Table S10. Pearson correlation coefficients between wine age and volatile molecules analysed by GC-MS using a polar column; Figure S2. Projection plan of the variables in the Multiple Factorial Analysis, when age perception was used as one of the sets of variables. Figure S3. Contribution of quantitative variables to dimensions 1 (a) and 2 (b). The expected average contribution is indicated by a dashed red line. Figure S4. Projection plan of the set of variables in the Multiple Factorial Analysis, when grape was used as one of the sets of variables.

Author Contributions: Conceptualization, M.F. and G.M.; methodology, M.F., G.M. and J.F.; software, M.F. and M.M.; validation, M.F.; investigation, G.M.; writing—original draft preparation, G.M., writing—review and editing, M.F.; supervision, M.F. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The data presented in this study are available on request from the corresponding author due to the absence of a data repository for this specific dataset.

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Conflicts of Interest: The authors declare no conflicts of interest.

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