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ORIGINAL RESEARCH ARTICLE

Sensory dimensions derived from competitive and creative perceptual interactions between fruity ethyl esters and woody odorants in wine-like models

Arancha de-la-Fuente-Blanco[1], María-Pilar Sáenz-Navajas[2], Jordi Ballester[3], Ernesto Franco-Luesma[3], Dominique Valentin[3] and Vicente Ferreira[1*]

[1] Laboratorio de Análisis del Aroma y Enología (LAAE), Departamento de Química Analítica, Universidad de Zaragoza, Instituto Agroalimentario de Aragón (IA2) (UNIZAR-CITA), C/ Pedro Cerbuna 12, 50009 Zaragoza, Spain
[2] Instituto de Ciencias de la Vid y del Vino (Universidad de La Rioja-Consejo Superior de Investigaciones Científicas-Gobierno de La Rioja). Department of Enology, Logroño, La Rioja, Spain
[3] Centre des Sciences du Goût et de l'Alimentation, INRA, Université Bourgogne Franche-Comté, Dijon, France

*correspondence:
vferre@unizar.es

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ABSTRACT

The present study analyses the sensory effects associated with the interactions between different woody aroma compositions and a simple fruity ester vector in red wines. The semi-synthetic wine models contained a fixed aroma composition, the dearomatised non-volatile fraction of red wine and 21 different combinations of ethyl 2-methylbutyrate (fruity vector) plus 1 out of 3 woody aroma compositions (woody vectors) at 3 possible levels of concentration each. Woody vectors imitated a highly toasted American oak (HAO—elevated levels of whiskylactones and furaneol), a highly toasted French oak (HFO—low levels of whiskylactone and vanillin levels, high levels of eugenol and guaiacol) and a medium toasted French oak (MFO—low levels of whiskylactones, eugenol, guaiacol and furaneol and high levels of vanilla). Models were sensorily assessed by a sorting task and by descriptive analysis. The increase in woody notes causes a concomitant decrease in fruity notes by a competitive perceptual interaction. HAO models are richest in coconut and woody notes and poorest in fruity notes, while HFO models keep strawberry and apple notes. At certain specific fruity-woody vector ratios, particularly in the MFO model, blackcurrant notes emerge, which can be considered a creative perceptual interaction.

KEYWORDS: oak aroma, ethyl 2-methylbutyrate, sensory notes, new odours, odour competition, black fruit, red fruit

INTRODUCTION

Recent research suggests that the perceived complexity of the wine is a multifaceted parameter related to both liking and perceived quality. It was observed that more complex wines are described with a more consistent vocabulary (Wang and Spence, 2018). Moreover, complexity is also related to the number of different perceptions it can elicit, both during a prolonged mouthful (Meillon *et al*., 2010) or over the lifetime of wine in the bottle (Parr *et al*., 2011; Spence and Wang, 2018). Those perceptions result from complex perceptual interactions between the many chemical components with odour present in the sample (Ferreira *et al*., 2021a). Perceptual interactions are a group of peripheral and central biochemical and neurophysiological events that modulate, filter, compress and integrate the primary input signals produced by the odorants reaching the olfactory receptors. These interactions bridge the odorant space of the product and the human perceptual spaces (Thomas-Danguin *et al*., 2016).

To understand them and be limited to odour × odour interactions, it has to be stressed that olfaction, like the other senses, relies on the encoding of meaningful odour objects (Thomas-Danguin *et al*., 2014). This implies that the process of identifying in a complex mixture of odorants new odours qualitatively untraceable to the individual constituents is a powerful and natural process (Lindqvist *et al*., 2012).

Perceptual interactions between odours have been recently classified into four different categories: competitive, cooperative, destructive and creative (Ferreira *et al*., 2021b). They are ranked in increasing order of the complexity of the perceptual interaction and in decreasing order of occurrence. Competitive interactions are the natural outcome of mixing non-blending dissimilar odours displaying normal poor additivity so that they can be considered the normal outcome of analytical processing. On the other side, creative interactions require a high degree of configurational or synthetic processing, which is known to depend on specific associations of odorants in strict concentration ratios (Romagny *et al*., 2018).

One of the characteristics of high-quality and complex wines is that they do not smell anything specific but that their aroma reminds or evokes more or less vaguely the odours of different products. This means that the signals from more than 30–40 odorants present at concentrations above the threshold, plus eventually some others at subthreshold levels, undergo integration processes in which only some of the odour features of key odorants remain, and many odour features from odorants present at specific ratios blend into new odours (Ferreira *et al*., 2021b; Romagny *et al*., 2018). One of the most expected and valuable outcomes in red wine is the development of different fruit-related notes, with a large preference towards fresh red and black fruits (Picard *et al*., 2015). The understanding of those fruity perceptions has been subjected to much research. Undoubtedly, the backbone of such perceptions is the 14 fruity esters present in wines. These compounds have been shown to integrate qualitatively so that in complex mixtures, they can be well represented just by one component (de-la-Fuente-Blanco *et al*., 2019). However, these compounds per se are not enough to introduce in the mixture of major alcohols, carbonyls and acids formed by alcoholic fermentation, or wine aroma buffer, the desirable fruity notes. Some other components seem to be essential for that, such as β-damascenone at certain ratios (Pineau *et al*., 2007; San-Juan *et al*., 2011), furaneol (Ferreira *et al*., 2016), little amounts of DMS (Escudero *et al*., 2007; Lytra *et al*., 2014; Picard *et al*., 2015; Segurel *et al*., 2004) or little amounts of 4-methyl-4-mercaptopentan-2-one (Escudero *et al*., 2004), 3-mercaptohexanol (Picard *et al*., 2015) or of the three varietal polyfunctional mercaptans (Rigou *et al*., 2014).

In the creation of new odours in wine, oak wood has a particularly important technological relevance since it can introduce into the wine at least three different categories of powerful odorants at quite different ratios depending on its origin and thermal processing (Cadahia *et al*., 2003; Cerdan *et al*., 2002; Chatonnet and Dubourdieu, 1998): vanilla-like odorants (vanillin, acetovanillone, methyl vanillate and ethyl vanillate), the coconut-peachy aroma of whiskylactones (E- and Z-whiskylactones), and the clove-like and smoke-like notes of volatile phenols (eugenol, guaiacol, isoeugenol). Consumers and experts have repeatedly identified fruity and woody aromas as markers of quality and preference in red wines (Hopfer and Heymann, 2014; Saenz-Navajas *et al*., 2013).

Because of these reasons, the interactions between fruity and woody notes have been the subject of some research. In general, those studies have shown that certain woody odorants (whiskylactones or eugenol) tend to dominate over fruity notes (isoamyl acetate or ethyl butyrate) (Atanasova *et al*., 2005) and that woody odorants usually induce a decrease in the perception of fruitiness (Atanasova *et al*., 2004), this effect being more evident with increasing toasting levels (Cameleyre *et al*., 2020). Both fruity and woody odours mask the alcohol odour, which can suppress the synergistic effects of fruity by woody in aqueous solutions (Le Berre *et al*., 2007). These previous studies, however, use simple aroma models, not considering all the odorants forming part of the wine aroma buffer nor the complexity and variability within the different types of wood extractables. Because of this, the present paper has as a major objective to study the perceptual odour interactions, at different concentration levels, between the ethyl ester fruity vector and three types of woody vectors, paying special attention to identifying the different qualitative sensory dimensions elicited out of these interactions. Specifically, this work tested the hypothesis that the combination of fruity and woody vectors can generate other sensory interactions, different from the masking effect of the woody vector over the fruity intensity, in a complex context similar to red wine.

MATERIALS AND METHODS

1. Compounds and standards

Solvents: LiChrosolv quality ethanol was purchased from Merck (Darmstadt, Germany), and mineral water was purchased from a local supermarket.

Standards: Chemical standards were supplied by Sigma-Aldrich (Madrid, Spain) and Firmenich (Geneva, Switzerland) and were of the highest purity available.

2. Preparation of wine models

2.1. Purification of ethyl esters

Standards of ethyl esters were isolated and purified using a liquid–liquid extraction of the commercial standard with a 5 % bicarbonate solution according to the procedure described elsewhere (de-la-Fuente-Blanco *et al*., 2019).

2.2. Preparation of wine models

Twenty-one wine models were generated by mixing a set of common components of red wines, both volatile and non-volatile. The non-volatile fraction was obtained by lyophilisation of red wine (total polyphenol index measured as absorbance at 280 nm = 59.5 au, total acidity = 3.6 g L^{-1} expressed in sulphuric acid, lactic acid = 1.9 g L^{-1}, malic acid = 0.3 g L^{-1} and reducing sugars = 5.8 g L^{-1}). The concentration of volatile compounds in the base wine B is given in Table 1 and corresponds to the average concentrations found in 96 commercial samples (San Juan *et al*., 2012). Attending to previous results (de-la-Fuente-Blanco *et al*., 2019), the fruity aroma vector was composed of a single ester (ethyl 2-methylbutyrate) which was spiked to this base wine at 434.7 (doubling the concentration in B) and 1292 mg L^{-1} (multiplying by four the concentration in B) for levels E1 and E2, respectively. Three different woody vectors, named HAO, HFO and MFO, were also generated. These vectors were composed of the same seven components (Z/E-whiskylactone, vanillin, ethyl vanillate, acetovanillone, guaiacol, furaneol and eugenol) at different concentrations, as detailed in Table 1, imitating different origins (French or American oaks, with low or high whiskylactone levels) and levels of toasting (varying the levels of vanillin, furaneol and eugenol/guaiacol, which are components that develop with toasting). Highly toasted American oak (HAO) is characterised by elevated levels of whiskylactones and furaneol. Medium-toasted French oak (MFO) is represented by lower concentrations of whiskylactones, eugenol, guaiacol and furaneol and increased levels of vanilla. Highly toasted French oak (HFO) is mimicked by low whiskylactone and vanillin levels and increased levels of eugenol and guaiacol (Morata, 2018). The following 21 models with final ethanol contents of 12 % (v/v) and at a pH = 3.5 were then generated: B, BMFO1, BMFO2, BHFO1, BHFO2, BHAO1, BHAO2, E1, E1MFO1, E1MFO2, E1HFO1, E1HFO2, E1HAO1, E1HAO2, E2, E2MFO1, E2MFO2, E2HFO1, E2HFO2, E2HAO1 and E2HAO2. The first letter determines the level of esters (B, E1 or E2), the next three, the woody vector (HAO, HFO or MFO) when added, and the last number corresponds to the level of the woody vector (1–2) (see supporting information Table A.1 of Appendix A).

3. Sensory analysis

Two sensory tasks were carried out to describe the samples: free sorting task followed by descriptive analysis. Eighteen millilitres of the sample (20 ± 1 °C) were presented in dark ISO-approved wineglasses labelled with a three-digit code and covered with a Petri dish according to a random and different order for each judge. All wines were presented at room temperature (20 ± 2 ºC) and evaluated in individual booths. All responses were collected in paper ballots. In both cases, participants were neither informed about the objective of the study nor paid for their participation.

3.1. Free sorting task

Participants: This task was carried out by twenty untrained panellists (12 men and 8 women, aged between 20 and 30 years, with an average age of 23 years), including students of the oenology master of the University of Burgundy (Dijon, France) and regular wine consumers.

Procedure: A total of twenty-one red wine models were evaluated regarding orthonasal aroma. Panellists were first required to smell each sample once in the proposed order. Afterwards, they were allowed to smell samples as many times as they wanted and in any order, and they had to group them according to their aromatic similarity. They were free to generate as many groups as they wanted and to include in each group as many wines as they wished. Once they had built the groups, they were instructed to describe the groups with a maximum of three attributes. There was no time limit for both tasks. All samples were presented simultaneously, according to a random arrangement different for each panellist.

3.2. Descriptive analysis

This task was carried out in two steps: 1) panel training and selection of panellists; 2) evaluation of the samples of the study.

Panel training and selection of the panellists: Seventeen panellists (10 men and 7 women, aged between 20 and 29, with an average age of 23 years), including students of the oenology master of the University of Burgundy (Dijon, France) and with experience in the evaluation of wine aroma, attended seven 60-minute descriptive training sessions over a period of three weeks. They worked in subgroups of 7–9 people. In sessions 1 and 2, different reference standards representative of 10 aroma descriptors (“fruit”, “strawberry”, “blackcurrant”, “green apple”, “dried fruit”, “wood”, “coconut”, “clove”, “caramel” and “vanilla”) were presented and discussed with the panellists. These descriptors were the most cited in the characterisation of the wine groups formed in the sorting task. The standards were commercially available odorants, syrups or fresh products prepared at the beginning of each session as described elsewhere (Saenz-Navajas *et al*., 2011). Panellists learned to identify them correctly. In session 3, different samples were prepared

TABLE 1. Aromatic composition of red wine models (µg L^{-1}).

	Compound	Concentration	
Mixture of compounds forming the common base aroma (B)	Isoamyl alcohol	180,000	
	β-phenylethanol	30,000	
	Acetic acid	150,000	
	Ethyl acetate	50,000	
	Hexanoic acid	2000	
	3-methylbutyric acid	300	
	2,3-butanodione	400	
	Isoamyl acetate	1000	
	Ethyl vanillate	250	
	Vanillin	70	
	γ-nonalactone	20	
	Guaiacol	10	
	β-damascenone	4.0	
	β-ionone	0.30	
	4-Hydroxi-2,5-dimethyl-3(2H)-furanone (furaneol)	30	
	Ethyl cinnamate	0.43	
	Linalool	7.0	
	Ethyl 2-methylbutyrate	432	
	Geraniol	0.13	
		Level 1	Level 2
Ester vector (E)	Ethyl 2-methylbutyrate	434.7	1292
Highly toasted American oak (HAO)	Whiskylactone	333	667
	Vanillin	167	333
	Ethyl vanillate	833	1667
	Acetovanillone	333	667
	Eugenol	333	667
	Guaiacol	33	67
	4-Hydroxi-2,5-dimethyl-3(2*H*)-furanone (furaneol)	33	67
Highly toasted French oak (HFO)	Whiskylactone	111	222
	Vanillin	167	333
	Ethyl vanillate	833	1667
	Acetovanillone	333	667
	Eugenol	333	667
	Guaiacol	33	67
	4-Hydroxi-2,5-dimethyl-3(2*H*)-furanone (furaneol)	17	33
Medium-toasted French oak (MFO)	Whiskylactone	111	222
	Vanillin	333	667
	Ethyl vanillate	833	1667
	Acetovanillone	333	667
	Eugenol	185	370
	Guaiacol	19	37
	4-Hydroxi-2,5-dimethyl-3(2*H*)-furanone (furaneol)	9	19

by spiking commercial wines with different concentrations of these standards to train them in ranking by intensity. In sessions 4–6, they were trained to use an 8-point intensity scale (with 0 = "absence" and 7 = "very intense") and familiarised with the sensory space of the model wines used in the study. In the last session, the ability of panellists to rate the different descriptors was evaluated. For that purpose, nine wines, corresponding to three triplicated wines, were presented. Samples were labelled with a three-digit random code and covered by a Petri dish. Participants were asked to score the ten aroma attributes on structured 8-point scales.

Evaluation of samples: Attending to individual performance in panel training and selection steps, fourteen out of seventeen panellists were selected for the evaluation of samples. Twenty-one red wine models were described in two sessions held on two different days. In each session, panellists were presented with 10–11 wine samples with an imposed 5-min break every five samples. All participants evaluated the 21 wine samples in a sequential monadic manner. They were instructed to score the intensity of the ten attributes on a structured 8-point scale as previously described. The wine models were prepared one day before the sensory session, stored at 10 °C and served 15 min before the session.

4. Data analysis

4.1. Free sorting task

Multidimensional analysis: The number of times each wine was classified in the same group was counted and compiled in a frequency table in which panellists were placed in columns and samples in rows. Data were analysed using a multi-block generalisation of MDS called DISTATIS (Lahne *et al*., 2018) using R (version 3.5.1). This analysis provides a spatial representation of samples. To explore the grouping of the samples in this space, a Hierarchical Agglomerative Cluster Analysis (HCA) was calculated with the coordinates of the samples onto all the DISTATIS dimensions using XLSTAT (Addinsoft, version 2015).

The descriptors employed in the second task to characterise the groups were collected, and their citation frequency was counted. Most cited attributes were selected for the descriptive task.

4.2. Descriptive analysis

Selection of panellists: With data derived from the 7th training session, for each attribute, a three-way ANOVA with judges/panellists (J), samples (S) and replicates (R) as fixed factors and first-order interactions was calculated. When J × S effects were observed for a given attribute, a Principal Component Analysis (PCA) was calculated to identify the accordance among judges. For these attributes, the scores of panellists, which presented a minimum correlation coefficient with the first PC of 0.4, were arbitrarily considered consistent, and thus, these panellists were selected for the evaluation of the samples.

Evaluation of panel performance: With the 14 selected panellists and with data derived from the 7th training session, for each attribute, a three-way ANOVA was performed considering samples (S), judges (J) and repetitions (R) as fixed factors and all first-order interactions.

Characterisation of the wines of the study: the scores obtained for each attribute were subjected to a two-way ANOVA (panellist as a random factor and wines as a fixed factor). A PCA was calculated with mean scores (n = 14) of the significant aroma attributes (alpha risk = 5 %). Further, HCA with the Ward method was calculated on all dimensions derived from PCA. The attributes best defining the resulting clusters were identified by calculating an ANOVA with clusters as fixed factors (alpha risk = 5 %). For significant attributes, the attribute with the highest score/s for each cluster was chosen as the attribute/s best defining that cluster.

Subsequently, to assess the effect of the level of esters, the level of wood and their interaction on the sensory attributes, three three-way ANOVAs (with panellists as a random factor and the levels of ester vector and wood vector as fixed factors) were calculated for each woody vector (HAO, HFO and MFO).

The degree of similarity between the sensory spaces derived from the sorting task and descriptive analysis was assessed by calculating RV coefficients (Robert and Escoufier, 1976). All statistical analyses were carried out using XLSTAT (Addinsoft, version 2015) and R (version 3.5.1).

5. Representation of the odour intensity of the mixture vs the fraction of woody vector

The measured intensities of fruity and woody notes in the wine models were represented versus the fraction of woody vector added to the mixture. In these graphs, the ordinate represents the sum of intensities of the wood or fruit attributes, and the abscissa corresponds to a relative compositional parameter emulating the odour fraction, typically represented by τ_B, where $\tau_B = I_B/(I_A + I_B)$ (Cain *et al*., 1995).

6. PLS-models

To obtain preliminary theoretical models between analyte concentrations and sensory attributes found to discriminate between samples, a partial least squares regression analysis (PLS1) (Unscrambler 9.7 CAMO A/S Trondheim, Norway) was carried out. With this purpose, a model was built using X variables (quantitative data), showing the best individual correlation with the Y variable (sensory descriptor). These models were built with quantitative data for woody and fruity compounds (ethyl 2-methylbutyrate, Z/E-whiskylactone, vanillin, ethyl vanillate, acetovanillone, guaiacol, furaneol and eugenol), average intensity of sensory descriptors and samples. An attempt was made to achieve the simplest model (fewer variables) with a greater predictive capacity, measuring this by cross-validation. The quality parameters studied to evaluate the prediction ability of the models were the root mean square error for the prediction (RMSEP) and the percentage of variance explained by the model (% EV).

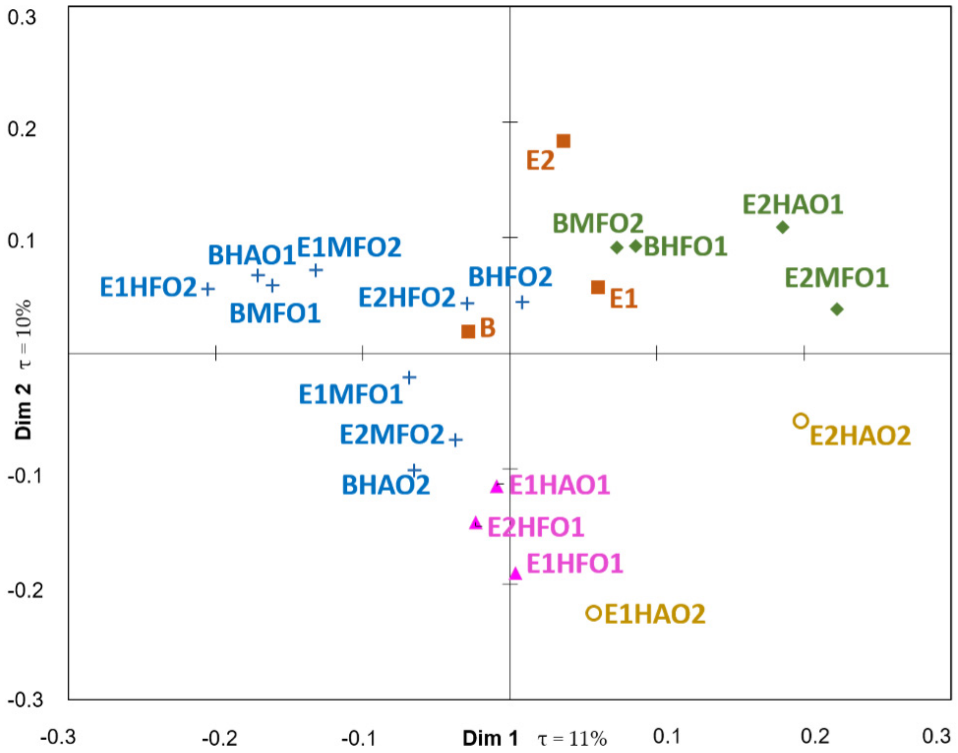

FIGURE 1. Scatter plot obtained on the first two dimensions derived from DISTATIS for the 21 wines submitted to the free sorting task. Colours indicate the group to which each sample belongs according to cluster analysis.

RESULTS AND DISCUSSION

1. Free sorting task

The sensory panel assessed the 21 samples containing three different levels of the ethyl ester vector (B, E1 and E2) and three levels (0, 1 and 2) of three different compositions of woody odorants (HAO, HFO and MFO) as described in Table 1. Results from the free sorting task are presented in Figure 1.

According to Figure 1, the panel separated the samples into five different clusters. Remarkably, only two out of the five classes contained compositionally homogeneous samples. One of these clusters is formed by the three model wines with no woody vector (B, E1 and E2, in orange). Interestingly, this group projects quite close to the centre of the plot (as well as on dimensions 3 and 4, data not shown), which suggests that the absence of woody components, even if it is a grouping factor, does not make the samples to be particularly different. Or the other way around, that the addition of the different woody vectors introduces a wide range of heterogeneous differences. The second homogeneous group is formed by two of the samples containing the HAO woody vector (rich in whiskylactones and furaneol, in yellow) at high concentration (E1HAO2 and E2HAO2) represented in the right-down part of the plot, suggesting that they have a quite distinctive sensory profile. No further compositionally homogeneous groups are observed. The plot also reveals that, hierarchically, the most relevant compositional element seems to be the ethyl ester vector. This can be seen in the fact that all samples with high levels of this vector (marked with E2) present scores in the first component higher than –0.04. Additionally, the three samples with maxima scores in the first component (> 0.1) have all maxima levels of the ethyl ester vector, while all samples with the lowest level of this fruity vector (except BHAO2) have positive scores in the second component.

Considering that the sorting task is a descriptive technique that highlights the salient differential dimensions among the sample set rather than providing a detailed description of the wines, a descriptive analysis, including a training step, was further carried out.

2. Descriptive analysis

2.1. Panel performance

Fourteen trained panellists were selected. The performance of this selected panel was confirmed by three-way ANOVAs involving samples (S), judges (J) and repetitions (R) as fixed factors and including their first-order interactions (see supporting information Table A.2 of Appendix A). The sample effect (S) was significant for all attributes except for "clove", which confirms the discrimination ability of the panel. No replication effect (R, S × R, J × R) was observed, which confirms the repeatability of the panel. The interaction sample by the judge (S × J) was significant only for the attributes “dried fruit” and “wood”. In addition, the PCA calculated with both attributes showed a fair agreement between judges since all of them were represented on the positive side of PC1 in both cases (arbitrary correlation coefficients ≥ 0.4, in all cases). These results confirm that these significant S × J interactions were mainly due to disagreement in the use of the scale and confirmed the consistency of the panel in the evaluation of the attributes.

2.2. Global sensory characterisation (two-way ANOVA)

The scores of the 14 selected panellists were firstly submitted to two-way ANOVA (panellists as random factor and samples as fixed factor) on the 21 studied wines to have a more precise insight into the diversity of the sensory effects caused by the type of woody vector. The panel found significant differences between samples in four out of the ten attributes (Table A.3 in supporting information of Appendix A). These were “blackcurrant” ($F = 1.812$, $P < 0.05$), “dried fruit” ($F = 2.062$, $P < 0.01$), “wood” ($F = 2.688$, $P < 0.001$) and “coconut” ($F = 2.068$, $P < 0.001$). Figure 2 shows the PCA calculated with these four attributes. The first three dimensions explain ca. 90 % of the original variance. The projections of the 21 wine samples and the factorial loadings of the four significant attributes on the first two or on the 1st and 3rd dimensions are given in Figure 2a,b, respectively. The first PC shows a clear opposition between the fruity descriptors “blackcurrant” and “dried fruit” and the woody descriptors “coconut” and “wood”. Such opposition indicates that samples are either fruity or oaky, regardless of whether the fruit is typical of young wines (blackcurrant) or aged wines (dried fruit). This opposition between fruity and woody aromas has already been reported in previous works (Tavares *et al.*, 2017). The second dimension explains 29 % of the original variance and discriminates between the fruity descriptors, with “blackcurrant” on the positive part of the PC2 and “dried fruit” on the negative part of this axe. The third dimension explains 16 % of the original variance and separates the two wood-related attributes. The generic term “wood” is on the negative part of this PC3, and the specific term “coconut” is on the positive one.

The PCA is complemented with the cluster analysis carried out with all PCA dimensions, whose results are summarised in the dendrogram shown in Figure 3 and with the average (among samples belonging to each cluster) aroma scores of the four significant attributes of the four clusters (Figure 4).

Results indicate that samples can be classified into four differentiated groups. The two most compact clusters are cluster 1, containing the unoaked samples E2 and B (in purple in Figure 2, mainly characterised as “dried fruit”), and cluster 3, containing the samples E2MFO1 and BMFO1 (in red in Figure 2, described as “blackcurrant”). Cluster 2 includes the three samples with the HAO2 vector (BHAO2, E1HAO2 and E2HAO2, in orange in Figure 2, and mainly described with the “coconut” descriptor), and cluster 4 contains the rest of the wines (in green in Figure 2, globally described with “wood” character).

It should be noted that there are some similarities between the representations of descriptive analysis in Figure 2 with that obtained in the sorting task given in Figure 1. In fact, the RV coefficient calculated on the sensory spaces derived from both sensory tasks is significant ($RV = 0.326$; $P < 0.05$). In both cases, samples without wood (B, E1 and E2) are clustered together or closely positioned in the plots, and in both cases, the saliency of the HAO2 vector is recognised. However, the sorting task was unable to identify the existence of Cluster 3 (blackcurrant) or Cluster 1 (dried fruit), even though this last one was the most different, attending to Figures 2 and 3. While these differences result

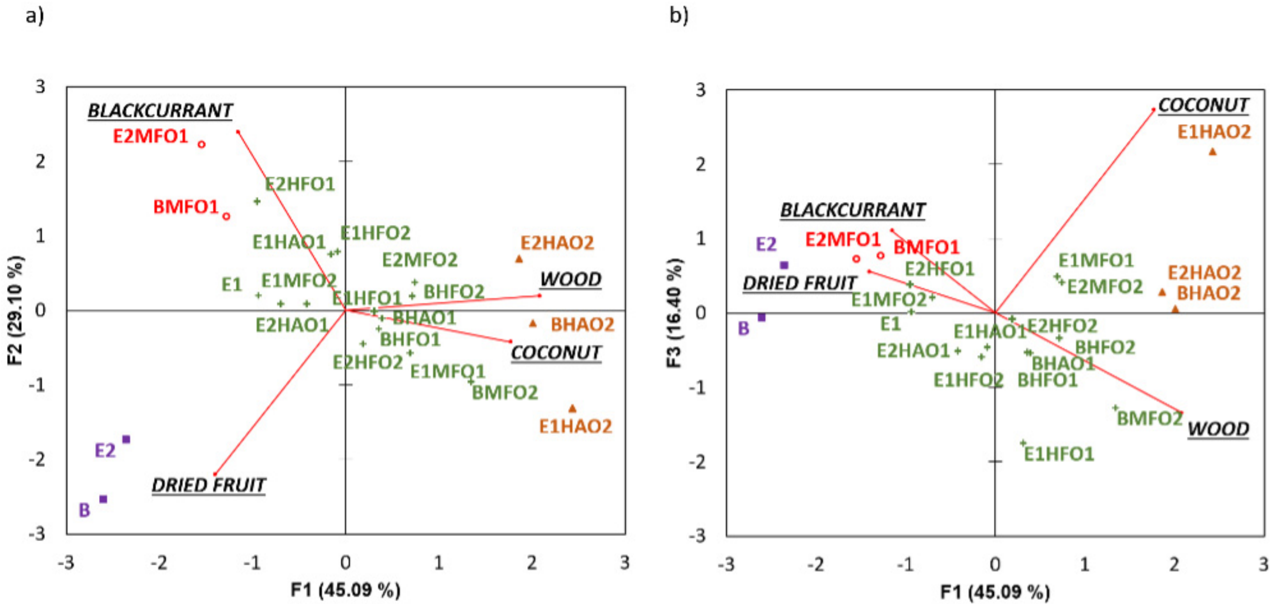

FIGURE 2. Projection of the 21 wine models and significant attributes derived from conventional descriptive analysis on a) PC1–PC2; b) PC1–PC3. Wines belonging to different clusters are marked with different colours and icons.

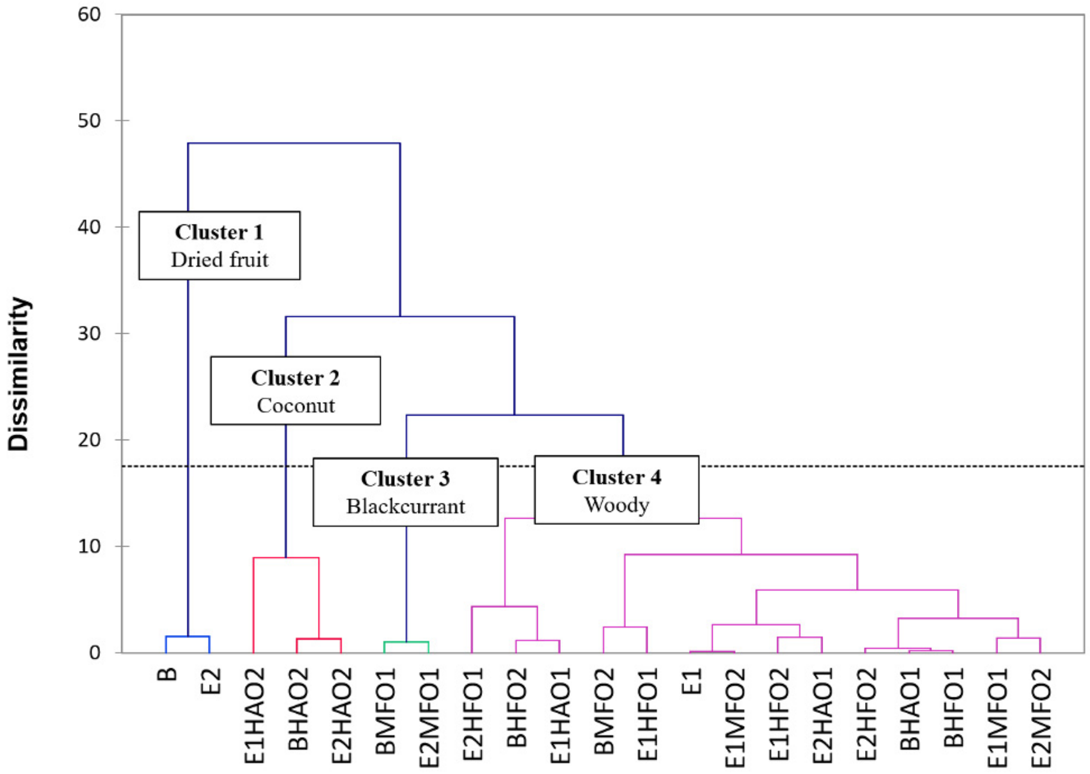

FIGURE 3. Dendrogram showing the four wine clusters derived from hierarchical cluster analysis calculated on all dimensions of the PCA performed with the 21 studied samples and using significant attributes. Attributes describing each cluster are those with significantly higher scores in the given cluster.

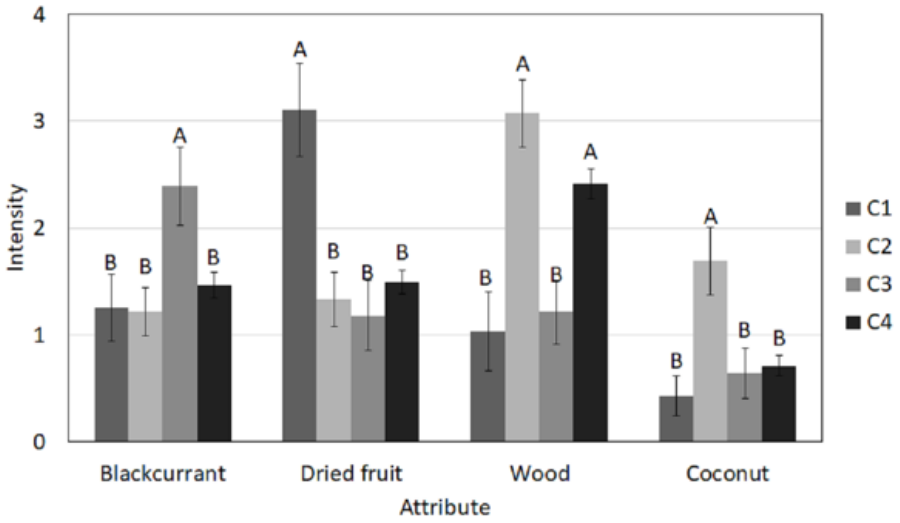

FIGURE 4. Bar chart with the average intensities (of the 14 panellists and the wines belonging to each cluster) of significant attributes for each of the four clusters (C1–C4). Error bars are mean standard errors. Different letters mean significant differences (P < 0.05).

from the difference in mental processes associated with the two different tasks, they may also suggest that "dried fruit" and, particularly, "blackcurrant" is far less salient than the mixture of coconut and woody.

The opposition between woody and fruity descriptors shown in Figure 2a is sometimes interpreted as a suppression of the fruity character by the woody vector (Atanasova *et al*., 2005a; Atanasova *et al*., 2005b; Atanasova *et al*., 2004). However, as discussed in the literature (Ferreira *et al*., 2021b), this is not a question of suppression but the expected result of mixing two dissimilar and non-blending odours following the most common "partial addition" behaviour (Cameleyre *et al*., 2020). Suppression takes place when the intensity of one of

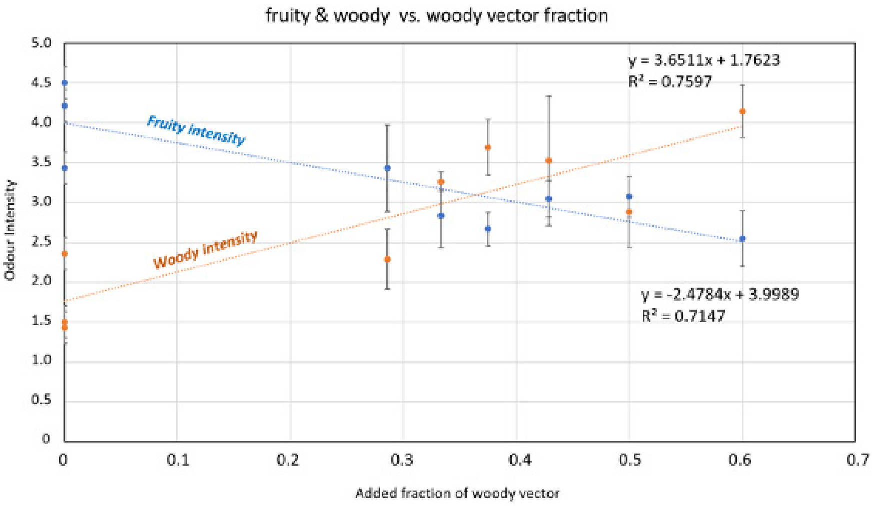

FIGURE 5. Representation of the intensities of fruity notes ("blackcurrant" + "dried fruits") or woody notes ("woody" + "coconut") versus the approximated fraction of woody vector added to the mixture.

the attributes decreases without any noticeable increase in the intensity of the other, and it is linked to the existence of "compromise" in the mixture of the two odours (in compromise, the perceived intensity of the mixture is smaller than the intensity of its most intense component). However, this is not the case, as seen in the representation in Figure 5. The figure represents the measured intensities of fruity and woody notes in the model wines versus the fraction of woody vector added to the mixture. The abscissa is a relative compositional parameter emulating the odour fraction, typically represented by t_b, which is the preferred parameter to study the odour properties of binary mixtures (Cain *et al.*, 1995).

As the odour intensities in isolation of the fruity vector and of the woody vectors used to prepare the models are not known, these have been approximated by the values 1, 2 and 2.5 for the low, medium and high levels of the fruity vector, and by 0, 1 and 1.5 for the low, medium and high levels of the woody vectors. The plot reveals that the intensity of the fruity notes significantly decreases with the increase of the fraction of the woody vector ($P < 0.01$) and that such a decrease is concomitant with the increase of the woody notes ($P < 0.01$). A similar result is obtained if the representation includes the summation of all the fruity descriptors ("fruit", "strawberry", "blackcurrant", "apple" and "dried fruit"), whose decrease with the fraction of woody vector is significant at $P < 0.05$, and the summation of all the woody descriptors ("wood", "vanilla", "caramel" and "coconut"), whose increase is significant at $P < 0.01$. This type of odour interaction has been recently named competitive (Ferreira *et al.*, 2021b) since it seems to result from a phenomenon of divided attention between two different non-blending odour objects. The increase in the intensity of one of the objects brings about a decrease in the perceived intensity of the other.

The existence of a partial addition behaviour between fruity and woody odorants is indirectly demonstrated in the slight but significant increases in the total intensity of the mixtures with the increase of the mass of woody odorants or in the slight and non-significant increases with the increase of the mass of fruity odorants, as was demonstrated by Cameleyre *et al.* (2020). The total intensity was estimated as the summation of the intensities of all perceived descriptors. The average increase in intensity linked to the increase in the level of woody vectors (from 0 to 1 or from 1 to 2) is 0.77, a 5.5 % in average, significant at $P < 0.05$, while the average increase in intensity linked to the increase in the level of the fruity vector (from B to 1 or from 1 to 2) is 0.58, a 4.0 % in average, non-significant with $P = 0.072$. Both data are consistent with a partial addition pattern.

2.3. Specific effect of the type of wood (three-way ANOVA)

To analyse the specificities of the interactions between fruity and woody odorants linked to the type of woody vector, the sample set was divided into three subsets, one by type of wood (Table A.1 in supporting information of Appendix A). In the three sets, unwooded samples were included as 0 levels, and a three-way ANOVA was carried out with panellists as random factor and levels of ester and woody vectors as fixed factors. Table 2 reveals interesting differences between the woody vectors. In fact, common effects are limited to the terms "woody" and "dried fruit". The former increases, and the latter decreases with the level of woody vector added, as already mentioned.

Leaving aside these two common effects, the table reveals the existence of quite exclusive effects. The highly toasted American oak vector (HAO, the richest in whiskylactones and furaneol) significantly affects "coconut" and "strawberry" notes ($P < 0.001$ and $P < 0.05$, respectively). The highly toasted French oak vector (HFO, containing average concentrations

TABLE 2. Three-way ANOVA results (participants as random and ester and woody levels as fixed factors) were calculated to assess the existence of significant effects. A three-factor ANOVA has been calculated for each type of wood (HAO, HFO or MFO). Significant effects are marked in bold (P < 0.05).

Type of wood	**HAO**						**HFO**						**MFO**					
Interaction [a]	**E**		**HAO**		**E × HAO**		**E**		**HFO**		**E × HFO**		**E**		**MFO**		**E × MFO**	
Significant effect [b]	**F**	**P**	**F**	**P**	**F**	**P**	**F**	**P**	**F**	**P**	**F**	**P**	**F**	**P**	**F**	**P**	**F**	**P**
Attribute																		
Fruity	1.08	ns	0.79	ns	0.74	ns	1.65	ns	0.32	ns	0.41	ns	2.30	ns	1.63	ns	0.10	ns
Strawberry	1.75	ns	3.20	*	1.36	ns	3.54	*	0.25	ns	0.70	ns	2.14	ns	0.08	ns	0.93	ns
Blackcurrant	0.57	ns	0.47	ns	0.70	ns	0.90	ns	0.28	ns	2.04	ns	1.59	ns	3.02	ns	3.83	**
Apple	1.02	ns	0.40	ns	1.54	ns	2.52	ns	0.84	ns	0.85	ns	4.97	**	1.11	ns	0.99	ns
Dried fruit	0.94	ns	8.20	***	2.12	ns	2.60	ns	6.96	**	0.90	ns	0.68	ns	8.39	***	1.71	ns
Wood	1.61	ns	9.52	***	0.75	ns	1.34	ns	11.39	***	1.01	ns	0.96	ns	6.62	***	1.51	ns
Clove	0.06	ns	2.54	ns	1.05	ns	0.23	ns	1.83	ns	0.99	ns	2.14	ns	0.42	ns	1.13	ns
Vanilla	0.32	ns	0.89	ns	1.07	ns	1.23	ns	0.87	ns	0.25	ns	0.33	ns	1.03	ns	0.76	ns
Caramel	1.13	ns	1.86	ns	0.59	ns	0.35	ns	0.24	ns	0.93	ns	0.11	ns	1.36	ns	0.78	ns
Coconut	1.07	ns	10.49	***	0.92	ns	0.85	ns	0.90	ns	0.68	ns	0.64	ns	1.59	ns	0.86	ns

[a] E: ester vector; HAO: vector representative of a type of wood rich in whiskylactone and furaneol; HFO: type of wood containing average concentrations of odorants; MFO: type of wood which is the richest in vanillin and the poorest in furaneol, guaiacol and eugenol. [b] Significant effects are marked in bold (P < 0.05). ns: not significant, *, ** and *** indicate the level of statistical significance with P < 0.05, 0.01 and 0.001, respectively.

of odorants) does not significantly affect any other sensory note, but in this context, the fruity vector significantly affects “strawberry” notes (P < 0.05). The most complex effects are observed in the medium-toasted French oak vector (MFO), which is the richest in vanillin and the poorest in furaneol, guaiacol and eugenol. Its addition does not significantly affect any other sensory note, but in its presence, the fruity vector affects “apple” notes (P < 0.01) and a quite interesting significant interaction between both vectors emerges on the “blackcurrant” note (P < 0.01).

Regarding the specific effect of the highly toasted American oak vector (HAO) on “coconut”, it can be seen in the plot shown in Figure 6a that the highest levels of the vector produce a significant increase in the intensity of this attribute (the reference data is collected in Table A.4 in the supporting information of Appendix A). While the effect was expected at these levels of whiskylactone (Boidron *et al*., 1998), it should be remarked that such an increase is particularly intense in the models containing the fruity vector at intermediate levels (E1HAO2, grey bars). This is directly connected to the effects on “strawberry” notes, as can be seen in Figure 6b. “Strawberry” reaches maximum intensity in unwooded wines containing the fruity vector at the intermediate level (E1, blue bars). The presence of the HAO woody vector makes this sensory note decrease so that the minima levels for this note are registered in the base wine not containing any vector (B, blue bars) and in the three models containing the HAO vector at the highest level (BHAO2, E1HAO2, E2HAO2, grey bars). This competitive effect is particularly interesting and may be related to the fact that strawberry flavours often have γ-lactones (γ-nona and γ-decalactone) and furaneol as normal constituents (Ubeda *et al*., 2012). As aforementioned, this competitive effect was formerly regarded as a suppression effect (Atanasova *et al*., 2005; Atanasova *et al*., 2004).

The intensity of the “strawberry” note in the model wines containing the highly toasted French oak vector (HFO) can be seen in Figure 6c. It can be appreciated that in this case, the presence of the woody vector does not make “strawberry” decrease, so models containing high or intermediate levels of the woody vector (red and grey bars) and intermediate or high levels of the fruity vectors (E1 or E2), retain strong levels of this descriptor, which makes the effect of increasing the ester vector significant. In the medium-toasted French oak vector (MFO, the richest in vanillin and the poorest in furaneol, guaiacol and eugenol), shown in Figure 6d, there is a chance to retain high woody levels with “strawberry” notes in models containing both vectors at maxima levels (E2MFO2).

Finally, the effects on “apple” and “blackcurrant” notes observed in models containing the medium-toasted French oak vector (MFO) can be seen in Figures 6e,f, respectively. When added at intermediate or high levels, this woody vector can retain the “apple” notes associated with high levels of fruity esters (MFO1 or MFO2). The “blackcurrant” descriptor is clearly perceived exclusively in the models containing intermediate levels of the woody vector and low

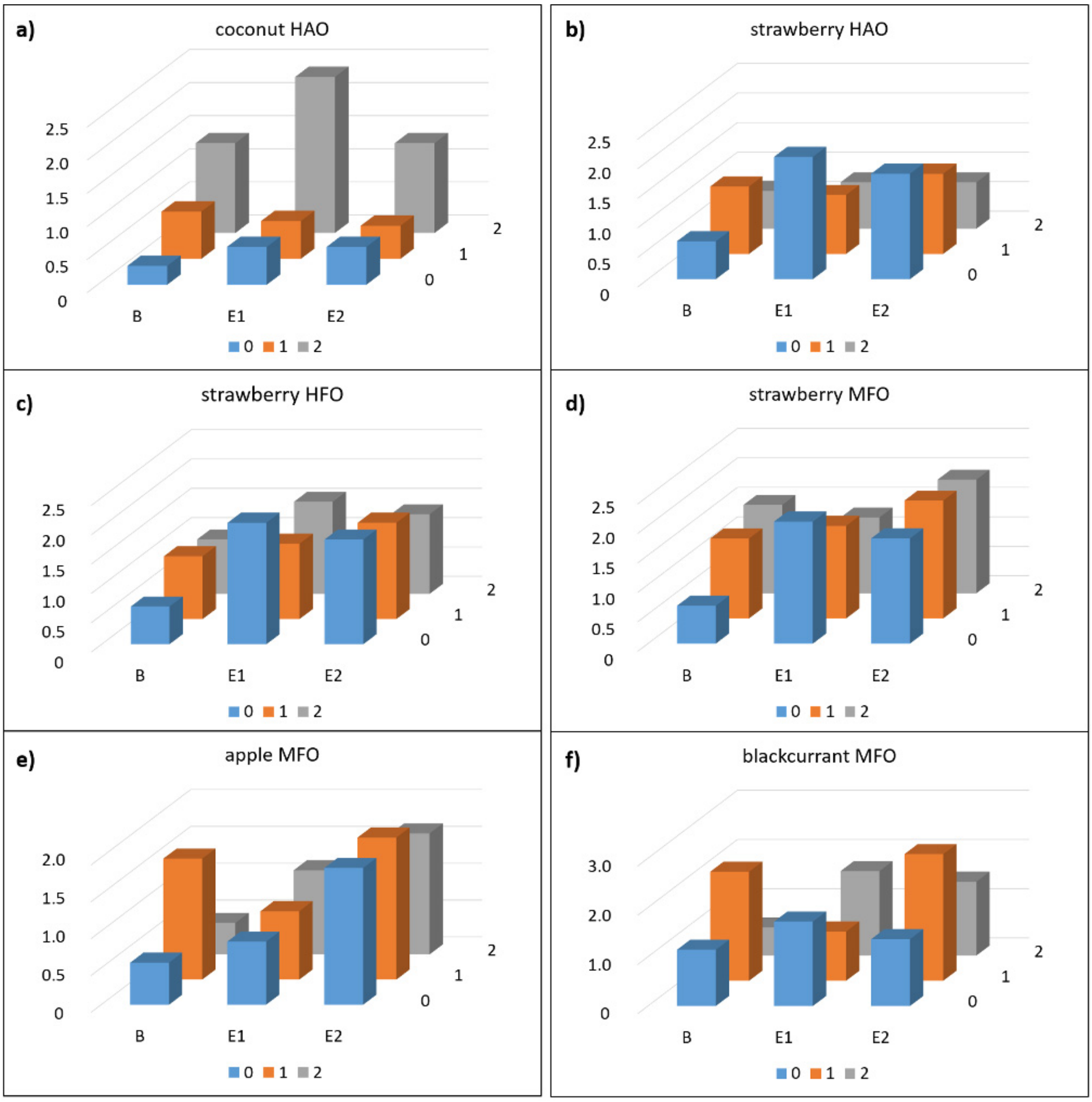

FIGURE 6. Representation of the intensities of a) "coconut" in HAO woody vector; b) "strawberry" in HAO woody vector; c) "strawberry" in HFO woody vector; d) "strawberry" in MFO woody vector; e) "apple" in MFO woody vector; f) "blackcurrant" in MFO woody vector, all of them at different concentration levels (0, 1 and 2) of the cited woody vector.

or high levels of fruity esters (BMFO1, E2MFO1, red bars). In the case of the HFO model, the note is also perceived at intermediate levels of the woody vector and high levels of the fruity vector.

A pertinent question is why the competitive effects between woody odorants on "strawberry" are clearly observed in the HAO model, while they are very weak or inexistent in HFO and MFO models, respectively. The answer to this question is found in the plots comparing the profiles of the models (Figure 7). It can be seen that the intensity of the two woody descriptors ("coconut" and "woody") is much higher in models containing the HAO vector (Figure 7b). This explains the strong decrease in intensities of fruity descriptors noted with the addition of the HAO vector (Figure 7a,b). By contrast, HFO models (Figure 7c) have high intensity of the "woody" descriptor but do not score in "coconut", while MFO models (Figure 7d) have the smallest intensity of woody descriptors. This explains that these models keep the fruity notes originally present in the unwooded samples, "strawberry" and "apple" (Figure 7a,d).

Regarding the apparition of the "blackcurrant" note, it seems to be the result of the integration of the "strawberry/apple" notes from the fruity ester with the specific aromas of the HFO (Figure 7c) and particularly MFO woody vectors (Figure 7d) at quite specific concentration ratios. This phenomenon can be classified within the "creative" interactions between odorants, which are the type of interactions leading to the formation of a new odour object or the complexion or perfection of an already present one (Ferreira *et al*., 2021b). It should be noted that "blackcurrant" notes have been

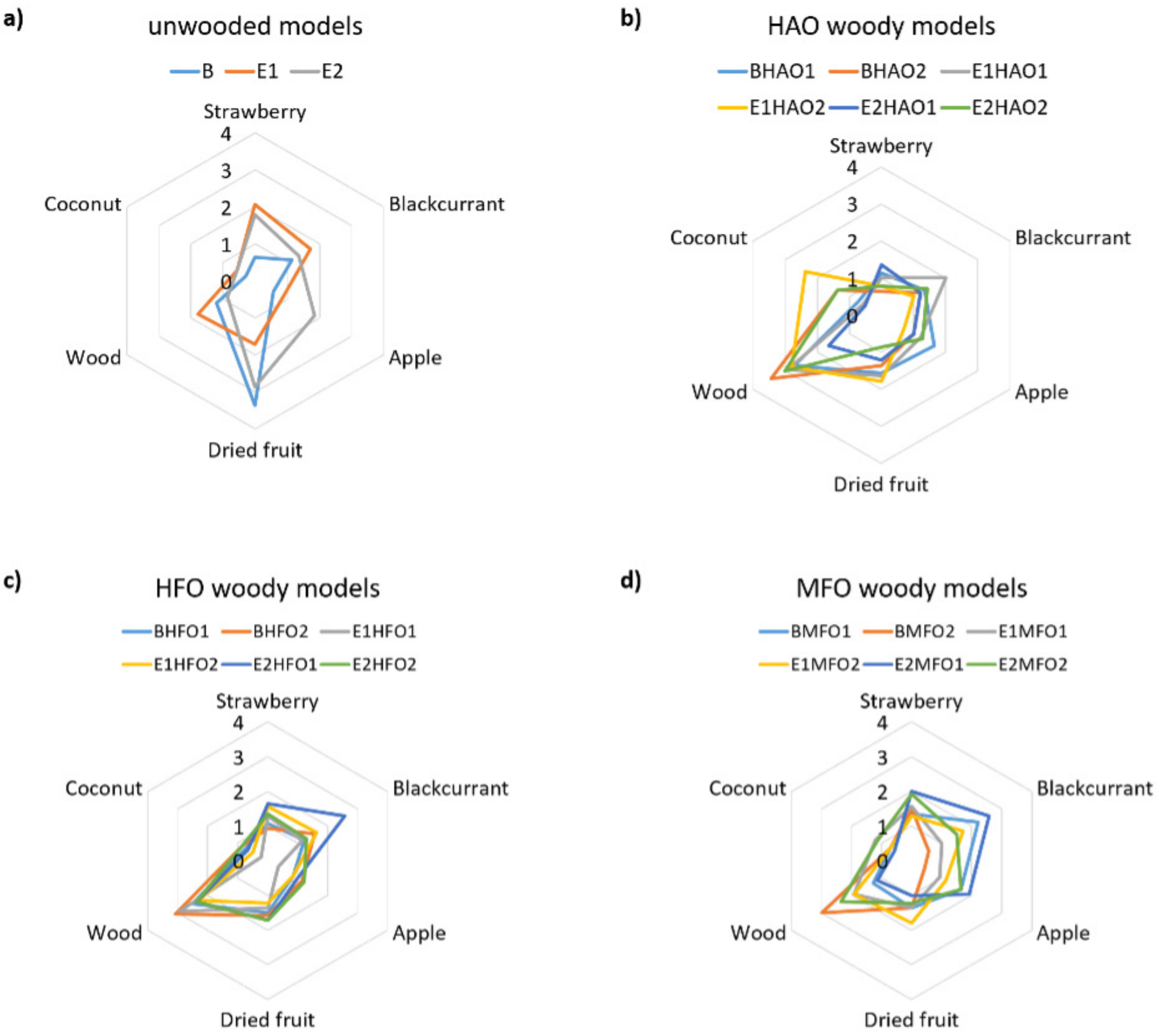

FIGURE 7. Spider plots with the sensory profiles of the different models: a) unwooded; b) woody model HAO; c) woody model HFO; and d) woody model MFO.

reported to emerge in different models or wines containing fruity esters and DMS (Lytra *et al*., 2014), fruity esters and 4-mercapto-4-methyl-2-pentanone (Rigou *et al*., 2014), or fruity esters and 3-mercaptohexyl acetate (Ferreira, authors' unpublished work based on previous observations in the laboratory). This suggests that "blackcurrant" notes are a current outcome of red wine aroma and can emerge from the creative interaction between fruity esters and different combinations of odorants.

2.4. PLS models

Although the experiment was designed to assess sensory variability and not to create models, PLS modelling has been used as an additional tool to explore the relationship between sensory descriptors and chemical composition. The best models are summarised in Table 3. A notable observation derived from the models (Table 3) is the role played by vanillin. Vanillin seems to have a positive contribution to "strawberry", a negative contribution to "woody", and has no role in the model explaining the combination of woody-related sensory notes, including "vanilla". A second observation derives from the model of "dried fruit", in which only furaneol has a weak positive coefficient, and all the other components have negative coefficients. This confirms that "dried fruit" is an odour related to the compounds forming the aromatic base of the models that decreases with the addition of whatever odorant. The negative contribution of furaneol and whiskylactone to the "strawberry" note is also notable, as well as the negative contribution of these volatile phenols to "coconut". The result for furaneol is surprising since it is an important impact compound of strawberry aroma (Rapp *et al*., 1980). As expected from the concept of competitive interactions, the combined models for fruity concepts, other than a blackcurrant, and for woody concepts are completely complementary. Finally, it is also remarkable that the impossibility of modelling "blackcurrant" or "apple" notes with linear models such as PLS, which is also expected from the nature of creative interaction.

TABLE 3. PLS models explain some of the sensory notes or a combination of them.

Attribute	Model
Strawberry*	$Y = 0.799 + 0.312 \cdot OAV_{E2M} + 0.179 \cdot OAV_{Van} - 0.120 \cdot OAV_{Wy} - 0.095 \cdot OAV_{Fur} - 0{,}014 \cdot (OAV_{Eug} + OAV_{Gua} + OAV_{EVa} + OAV_{Ava})$ Explained Variance: 64.8 %; RMSE: 0.265 (19 % scale); 2PCs *(sample E1MFO0 excluded)
Dried fruit	$Y = 3.183 + 0.059 \cdot \sqrt{Fur} - 0.145 \cdot \sqrt{E2M} - 0.136 \cdot \sqrt{Ava} - 0.129 \cdot \sqrt{Van} - 0{,}108 \cdot \sqrt{Eug} - 0.101 \cdot \sqrt{Wy} - 0.945 \cdot \sqrt{EVa} + 0.044 \cdot \sqrt{Gua}$ Explained Variance: 50.6 %; RMSE: 0.556 (21 % scale); 2PCs
∑ (strawberry + apple + fruity + dried fruit)	$Y = 6.221 + 0.666 \cdot OAV_{E2M} - 0.274 \cdot OAV_{Wy} - 0.270 \cdot \left(OAV_{Eug} + OAV_{Gua} + OAV_{EVa} + OAV_{Ava}\right) - 0.225 \cdot OAV_{Fur} - 0.083 \cdot OAV_{Van}$ Explained Variance: 72.1 %; RMSE: 0.700 (15.9 % scale); 2PCs
Coconut	$Y = 0.193 + 0.111 \cdot OAV_{Fur} + 0.109 \cdot OAV_{Wy} - 0.085 \cdot \left(OAV_{Eug} + OAV_{Gua} + OAV_{EVa} + OAV_{Ava}\right) - 0.065 \cdot OAV_{Van} - 0{,}009 \cdot OAV_{E2M}$ Explained Variance: 46.1 %; RMSE: 0.33 (15.0 % scale); 1PC
Woody	$Y = 1.414 + 0.142 \cdot \sqrt{Wy} + 0.133 \cdot \sqrt{Gua} + 0.133 \cdot \sqrt{Eug} + 0{,}130 \cdot \sqrt{Fur} + 0.072 \cdot \sqrt{Ava} + 0.071 \cdot \sqrt{EVa} - 0.248 \cdot \sqrt{E2M} - 0.037 \cdot \sqrt{Van}$ Explained Variance: 69.1 %; RMSE: 0.454 (17.0 % scale); 2PCs
(woody + clove + caramel + vanilla + coconut)	$Y = 3.448 + 0.464 \cdot \sqrt{Fur} + 0.267 \cdot \sqrt{Wy} + 0.242 \cdot \sqrt{Gua} + 0.203 \cdot \sqrt{Eug} + 0.190 \cdot \sqrt{EVa} + 0.166 \cdot \sqrt{Ava} - 0.387 \cdot \sqrt{E2M}$ Explained Variance: 82.4 %; RMSE: 0.81 (13.0 % scale); 2PCs

E2M: ethyl 2-methylbutyrate; *Wy*: whyskylactone; *Eug*: Eugenol; *Gua*: guaiacol; *Ava*: acetovanillone; *EVa*: ethyl vanillate; *Fur*: Furaneol; *Van*: vanillin; RMSE: root-mean-square error; OAV: Odour aroma value.

CONCLUSION

In summary, our findings show that woody compositions confer to the wine a generic woody character which, in general, competes with fruity notes, particularly with the “dried fruit” note emerging in unwooded models. However, the specific composition of the woody vector has remarkably differential effects on wine aroma, mostly a consequence of perceptual interactions with fruity esters. Only woody compositions containing high levels of whiskylactones and furaneol (HAO vector) communicate to the wine their specific “coconut” nuances, and this happens at the expense of decreasing “strawberry” and other fruity notes as the consequence of competitive perceptual interactions. However, models with woody vectors containing average concentrations of woody odorants keep the original “strawberry” and “apple” fruity notes. At certain specific fruity-woody vector ratios, particularly in the model richest in vanillin and poorest in eugenol, guaiacol and furaneol (MFO vector), “blackcurrant” notes emerge, which can be considered a creative perceptual interaction.

This result may be of interest to the wine industry as it provides the winemaker with tools to choose the type of barrel for wine ageing, and also to the general food industry since it shows how perceptual interactions modulate aroma perception.

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AUTHOR CONTRIBUTION

A.D.L.F.B.: Methodology, investigation, formal analysis, writing—original draft, writing—review and editing. M.P.S.N.: conceptualisation, investigation, writing—review and editing, formal analysis. E.F.: Investigation, Formal analysis. J.B.: methodology, formal analysis, writing—review and editing. V.F.: conceptualisation, project administration, writing—original draft, funding acquisition. D.V.: conceptualisation, project administration, writing—review and editing.

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