LWT - Food Science and Technology 204 (2024) 116481

Contents lists available at ScienceDirect

LWT

journal homepage: www.elsevier.com/locate/lwt

Study of the fruity aroma of red wines made from grape varieties potentially adapted to climate change using a semi-preparative HPLC method

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ARTICLE INFO

Keywords:
Aroma volatiles
Fruity notes
Late-ripening varieties
Red wine
Climate change

ABSTRACT

Under climate change, the fruity aroma of Bordeaux red wines is changing, tending toward undesirable aromas like dried and cooked fruits. To adapt to these changes in aroma, a strategy could be to introduce to Bordeaux vineyards late-ripening grape varieties more suited to drier, sunnier and warmer conditions. A comparative analysis involved traditional Bordeaux red wines (Merlot, Cabernet-Sauvignon, and Cabernet franc) and those from late-ripening grape varieties originating from high-temperature climates (Yiannoudin, Syrah, Agiorgitiko, Xinomavro, Touriga Nacional, Grenache, and Tempranillo). Using a semi-preparative HPLC method, aroma extracts from these ten red wines were fractioned, isolating fruity notes for HPLC–Fruity Aromatic Reconstitutions (FAR). Through a free sorting task, three clusters emerged. Targeted GC-MS analysis highlighted nine compounds characterizing sensory clusters, including ethyl 2-methylpropanoate, ethyl hexanoate, ethyl 6-hydroxyhexanoate, linalool, α-terpineol, α- and β-ionones, hexanol and (Z)-3-hexenol. Through the realization of sensory profiles, it has been demonstrated that by incorporating these compounds into a fruity model solution, fresh red- and black-berry fruit notes are intensified.

1. Introduction

A wide range of fruity nuances are associated with the character of red wine, from the desirable notes of fresh red- and black-berry fruits, ripe fruits and jammy fruit to the undesirable cooked fruit and dried fruit notes (Allamy et al., 2023; Pineau et al., 2009; Van Leeuwen et al., 2022). Larousse des Vins (1998) defined the fruity aroma of red wines as being "more akin to red-berry fruits such as red and black cherries, Damas prunes, redcurrants, blackcurrants, blackberries, raspberries, and strawberries." Pineau et al. (2010) described Bordeaux red wines as having a specific fruity aroma characterized by notes of black-berry and jammy fruits.

Over the past decade, changes in the aromatic fruity notes of Bordeaux red wines have been reported and correlated with factors linked to climate change, such as increasing temperatures, drought and light radiation, and changes in rainfall patterns (Van Leeuwen & Destrac-Irvine, 2017). These climatic variations affect the phenological cycle of the vine, shifting harvest to earlier dates and warmer conditions (Van Leeuwen et al., 2020); this impacts the physicochemical parameters of the grapes and in turn the composition of the wines.

Previous works have demonstrated a reduction in fresh fruity notes accompanied by an accentuation of dried fruit notes (e.g., notes of dried figs, dried apricots and prunes) in Bordeaux red wines made from Merlot grapes (Allamy et al., 2017, 2018). Being the most common grape variety in the Bordeaux region, Merlot represents nearly 66% of the planted area (CIVB, 2019–2020). Studies of Allamy et al. (2017, 2018) underscore the need to take precautions to preserve fresh fruity aromas under climate change. In light of this, multiple solutions for adaptation have been proposed: one strategy is the introduction of selected late-ripening grape varieties to potentially modify the vegetative cycle of the grapevine (Van Leeuwen et al., 2019). In response to global warming, this strategy has been initiated by the winegrowers' association of the AOCs Bordeaux and Bordeaux Supérieur, with authorization issued by INAO. As a result, four new red grape varieties, Arinarnoa, Castet, Marselan and Touriga Nacional, are now allowed in Bordeaux (INAO, 2021). A recent study by Garbay et al. (2023) highlighted that

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https://doi.org/10.1016/j.lwt.2024.116481
Received 28 May 2024; Received in revised form 29 June 2024; Accepted 11 July 2024
Available online 14 July 2024

single-varietal red wines made from late-ripening grape varieties planted in regions with hot climates were associated with desirable fruity descriptors; this indicates that, in terms of red grape varieties, several potential candidates exist for use in the preservation of wine quality.

Although many sensory descriptors are mentioned in the literature, knowledge regarding the fruity aroma of red wines in relation to their chemical composition is still lacking; this is partly because aromatic compounds are often present at levels below their detection threshold (Lytra et al., 2013) or even because some compounds do not necessarily exhibit fruity aromas (Antalick et al., 2015; Escudero et al., 2007; Lytra et al., 2016; Rigou et al., 2014). Several studies have demonstrated the sensory impact of esters on the expression of fruitiness, with synergistic effects indicating that esters increase the perception of fruity aromas in red wines (Lytra, Tempere, de Revel, & Barbe, 2012; Lytra, Tempere, de Revel, & Barbe, 2012, 2013, 2015; Cameleyre et al., 2017; Pineau et al., 2009). However, there is evidence in the literature that other compounds have an enhancing impact on the overall fruity aroma of wine, including furaneol and homofuraneol (Kotseridis & Baumes, 2000), vanillin and eugenol (Cameleyre et al., 2020), C13-norisoprenoids, such as β-damascenone and β-ionone (Escudero et al., 2007; Ferreira et al., 2016; Pineau et al., 2007; Tomasino & Bolman, 2021), and sulfur compounds, such as dimethyl sulfide (Beloqui et al., 1996; Segurel et al., 2004; Lytra et al., 2014, 2016). Recent results from Garbay et al. (2023) showed that the addition of compounds like monoterpenes, C13-norisoprenoids and alcohols with 6 carbon atoms (C6 alcohols) contributed to fresh black-berry fruit notes to a red wine initially described as having cooked fruit aromas.

However, the fruity aroma of red wine remains a complex context that has yet to be fully explained due to it involving complex interactions between various volatile and non-volatile compounds (Cameleyre et al., 2021; Dufour & Bayonove, 1999); these compounds can be affected by a number of different terroir-related factors, such as soil, climate and viticultural practices, as well as grape maturity, vinification process, and even wine aging (Allamy et al., 2023; Van Leeuwen et al., 2022).

Given the numerous interactions among volatile compounds themselves and between volatile and non-volatile compounds in red wines, the objective of this study is to work with a less complex matrix than wine to better understand the role of certain compounds in the perception of fruity notes. To achieve this, a semi-preparative method using High Performance Liquid Chromatography (HPLC) was employed to isolate only the fruity fractions from red wines. This study focuses on 10 high-quality red wines made from Bordeaux and Mediterranean grape varieties, selected for their qualitative notes of fresh and jammy red and black fruits. The aim of this work was to determine if it was possible to differentiate these 10 wines based on their fruity nuances and to identify key odorant compounds that characterize these nuances.

2. Materials and methods

2.1. Chemicals and reagents

Absolute ethanol (purity >99.9%) and anhydrous sodium sulfate were supplied by Merck (Fontenay-sous-Bois, France). Dichloromethane (purity >99.9%) was provided by VWR Chemicals (Fontenay-sous-bois). Microfiltered water was obtained using a Milli-Q purification water system (resistivity = 18.2 MΩ cm, Millipore, Saint-Quentin-en-Yvelines, France). Variable grade compounds were obtained from commercial sources as follows: ethyl propanoate (99%) and ethyl 2-hydroxy-2-methylpropanoate (98%) from Alfa Aesar (Kandel, Germany); 2-methylpropyl acetate (99%), ethyl butanoate (99%), butyl acetate (99%), ethyl hexanoate (99%), hexyl acetate (99%), ethyl octanoate (98%), ethyl 2-methylpropanoate (99%), ethyl 2-methylbutanoate (99%), ethyl 3-methylbutanoate (98%), 2-methylbutyl acetate (99%), ethyl 2-hydroxy-3-methylbutanoate (99%), ethyl 3-hydroxybutanoate (97%), ethyl 2-hydroxyhexanoate (97%), ethyl 6-hydroxyhexanoate (97%), methyl salicylate (99%), ethyl salicylate (99%), 1,4-cineole (95%), 1,8-cineole (99%), menthol (99%), β-citronellol (95%), geraniol (98%), β-damascenone (99%), α-ionone (96%), β-ionone (96%), (*E*)-3-hexenol (97%) and octan-2-ol (97%) from Sigma-Aldrich (Saint-Quentin Fallavier, France); 3-methylbutyl acetate (98%), ethyl 2-hydroxy-4-methylpentanoate (98%), linalool (96%), α-terpineol (95%), terpinen-4-ol (95%), nerol (98%), hexanol (98%) and (*Z*)-3-hexenol (97%) from TCI Europe (Zwijndrecht, Belgium); ethyl butyrate, 4,4,4-d_3, ethyl hexanoate-d_{11}, ethyl octanoate-d_{15}, linalool-d_3, β-ionone-d_3 from Cluzeau (Sainte Foy la Grande, France). Aroma compounds were prepared in absolute ethanol solution. All the compounds used in this study were olfactively pure, as confirmed by a gas chromatography analysis with olfactometric detection (GC-O) of reference compounds. Moreover, the products' purity was checked (>95%) using a flame ionization detector analysis.

2.2. Wine samples

In total, ten commercial single-varietal red wines made from grape varieties with a maturity gradient of a ripening date of two weeks after Chasselas (Tempranillo) to a ripening date of five weeks after Chasselas (Xinomavro) were selected (Table 1). The grape varieties were chosen for their later maturity compared to that of Merlot. The qualitative aromas of these ten red wines were evaluated in a preliminary study proposed by Garbay et al., 2023. These 10 wines exhibit qualitative fruity descriptors, characterized by fresh and jammy red- and black-berry fruits. (Table 1). Seven of the wines were made from grape varieties of the Mediterranean basin: Agiorgitko and Xinomavro (Greece), Yiannoudin (Cyprus), Syrah (France), Touriga Nacional (Portugal), Grenache and Tempranillo (Spain). The other three wines were from the Bordeaux area and made from Cabernet franc, Cabernet-Sauvignon and Merlot grape varieties. All of the wines were from the 2018 vintage and were opened on the same day in 2022.

2.3. Preparation of HPLC–fruity aromatic reconstitutions (FAR) from wine aroma extract

Considering the numerous interactions existing among volatile compounds themselves and between volatile and non-volatile compounds in red wines, a focus on a less complex matrix than wine was chosen in this work in order to better understand the role of certain compounds in the perception of fruity notes. To achieve this, a semi-preparative method using High Performance Liquid Chromatography (HPLC) was employed to isolate only the fruity fractions of red wines and then assemble them together to create fruity aromatic reconstitutions (FAR).

HPLC–FAR from wine aroma extract often single-varietal red wines were prepared using a methodology optimized by Lytra, Tempere, de Revel, and Barbe (2012). A 500 mL wine sample was extracted successively using 80, 80 and 40 mL of dichloromethane for 15 min. The organic phases were collected, dried over sodium sulfate and then initially concentrated using a Rotavapor (Laborota 4010 digital Rotary Evaporator, Heidolph, Germany) to reduce the extract volume, and then further concentrated under a nitrogen flow (100 mL/min) to obtain a final extract volume of 1.25 mL. The aroma extracts of the wines were eluted using a reversed-phase (RP) high-performance-liquid-chromatography (HPLC) methodology developed by Ferreira et al. (1999). The HPLC fractionation was carried out using a Nova-Pak C18 column (300 × 3.9 mm i.d., 4 μm, 60 Å, Waters, Saint-Quentin, France) (Pineau et al., 2009). The HPLC system consisted of an L-6200A pump (Merck-Hitachi, Germany). Chromatographic conditions were as optimized by Pineau et al. (2009): flow rate, 0.5 mL/min; injection volume, 250 μL wine extract; program gradient, phase A, water, phase B, ethanol, 0−2 min, 100% A, linearly programmed until 100% B at min 50. An automated fraction collector was connected to the end of the column, and 1 mL of eluted solvent was collected every 2 min. To obtain the HPLC– FAR, fractions of the fruity pool (fractions 17–21, previously selected by panel 1 as described in

Table 1
List of the 10 selected red wines.

Grape variety	Vineyard area	Country	Budburst[a]	Ripening[b]	Vintage	Olfactory descriptors (Panel 1)
Agiorgitiko	Nemea	Greece	4 days after Chasselas	3 weeks after Chasselas	2018	Fresh and jammy red-berry fruits notes
Xinomavro	Amyndeon	Greece	Same day as Chasselas	5 weeks after Chasselas	2018	Jammy red-berry fruits notes (strawberry)
Merlot	Bordeaux	France	2 days after Chasselas	2 and a half weeks after Chasselas	2018	Jammy red- and black-berry fruits notes (strawberry, blackcurrant)
Cabernet franc	Bordeaux	France	5 days after Chasselas	2 and a half weeks or 3 weeks after Chasselas	2018	Jammy red-berry fruits (strawberry) and jammy black-berry fruits (blackcurrant)
Cabernet-Sauvignon	Bordeaux	France	13 days after Chasselas	3 weeks or 3 and a half weeks after Chasselas	2018	Fresh black-berry fruits (blackcurrant, blackberry) and jammy black-berry fruits
Syrah	Rhône Valley	France	7 days after Chasselas	2 and a half weeks after Chasselas	2018	Freshness, fresh black-berry (blackcurrant, blackberry) and blackcurrant jam
Grenache	Saragossa	Spain	6 days after Chasselas	4 weeks after Chasselas	2018	Fresh red-berry fruits (strawberry, raspberry)
Tempranillo	Valladolid	Spain	7 days after Chasselas	2 weeks after Chasselas	2018	Fresh black-berry (blackcurrant and blackberry)
Touriga Nacional	Douro	Portugal	2 days after Chasselas	3 weeks after Chasselas	2018	Jammy red- and black-berry fruits notes
Yiannoudin	Lemesos	Cyprus	n.f.	n.f.	2018	Freshness, minty and fresh black-berry fruits notes

[a], [b]: PlantGrape database, 2023; n.f.: data not found.

section 1.1 Sensory Analyses.) were blended together to reproduce the initial volume in the original wine. Ethanol and microfiltered water were added to obtain an ethanol level of 12% (v/v) (Lytra, Tempere, de Revel, & Barbe, 2012).

2.4. Quantitation of aroma compounds in HPLC–fruity aromatic reconstitutions (FAR) made from single-varietal red wines

2.4.1. Sample preparation and aroma compound extraction

The samples were prepared as described by Garbay et al. (2023). A 50 mL HPLC–FAR sample was spiked with 7 different internal standards (IS): 374 μg/L of ethyl butyrate-d_3 (IS for the non-polar esters ethyl propanoate, 2-methylpropyl acetate, ethyl butanoate, butyl acetate and 3-methylbutyl acetate), 171 μg/L of ethyl hexanoate-d_{11} (IS for the non-polar esters ethyl hexanoate and hexyl acetate), 263 μg/L of ethyl octanoate-d_{15} (IS for ethyl octanoate), 300 μg/L of ethyl 2-hydroxy-2-methylpropanoate (IS for substituted and hydroxylated polar esters), 64 μg/L of linalool-d_3 for monoterpene compounds, 40 μg/L of β-ionone-d_3 for C_{13}-norisoprenoid compounds, and 160 μg/L of octan-2-ol for C6 alcohols compounds and methyl and ethyl salicylate. The HPLC–FAR sample was then extracted using 4, 4 and 2 mL of dichloromethane at room temperature (around 25 °C), with magnetic stirring (700 rpm), for 10 min at each volume of dichloromethane. The organic phase was then separated from the aqueous phase using a separatory funnel for 5 min. The organic phases were collected, blended, dried over sodium sulfate and initially concentrated using a Rotavapor (Laborota 4010 digital Rotary Evaporator, Heidolph, Germany) to reduce the extract volume, and then further concentrated under a nitrogen flow (100 mL/min) to obtain a final extract volume of 250 μL.

2.4.2. Gas Chromatography–Mass spectrometry analyses (GC-MS)

Targeted GC-MS analyses were carried out as described by Garbay et al. (2023) using an HP 5890 GC system coupled to an HP 5972 quadrupole mass spectrometer (Hewlett-Packard) equipped with a Gerstel MPS2 autosampler. Samples (2 μL) of aroma extract from HPLC–FAR were injected in split-splitless mode (injector temperature: 200 °C, interface temperature: 240 °C, split flow: 60 mL/min, split ratio: 30:1, total flow: 60 mL/min, saver flow: 20 mL/min, saver time: 2 min). The capillary column was a BP21 (SGE 054469, Ringwood, Australia), 50 m × 0.320 mm i.d., film thickness: 0.25 μm. The oven temperature was programmed as follows: 40 °C for 1 min, then increased to 100 °C at a rate of 1 °C/min, increased to a final isotherm of 180 °C at 3 °C/min, and finally maintained at 180 °C for 3 min. The run lasted 90 min in total. The carrier gas was Helium N55 (Air Liquide, France) with a column head pressure of 9.48 psi and an initial flow of 2 mL/min. The MSD transfer line heater was set to 280 °C. The mass spectrometer was operated with electron ionization at 70 eV and in selected-ion-monitoring (SIM) mode (dwell = 20 msec). The ions were selected according to the previous study by Garbay et al. (2023). The ions that were monitored were chosen based on the best-measured signal-to-noise ratio. MS data were recorded and processed using MSD ChemStation Data Analysis Application software equipped with the NIST 2008 MS library (US National Institute of Standards and Technology, Gaithersburg, MD). The compounds were then characterized by comparing their linear retention index (LRI) and their mass spectra with those of the standards.

2.4.3. Statistical analyses

Statistical analyses of quantitative data were performed using XLSTAT software (Addinsoft, Paris, France). The significance of the variations in concentrations of the aromatic compounds was tested using the sensory clusters obtained in the free sorting task (see Section 2.5.3.) by analysis of variance (ANOVA) with Tukey's test and a level error $\alpha =$ 5% ($p < 0.05$). The homogeneity of the data variance had previously been tested using Levene's test. Finally, the normality of the ANOVA residuals was tested using Shapiro−Wilk's test.

2.5. Sensory analyses

2.5.1. General conditions

The samples were evaluated at controlled room temperature (20 °C) in individual booths. Covered ISO glasses (ISO 3591:1977, 1977; ISO 8589:2010, 2010) were used, containing about 20 mL of liquid and coded with 3-digit random numbers. All the sensory analyses presented in this work were realized via orthonasal perception.

2.5.2. Sensory panels

The panelists were selected from the research laboratory staff at ISVV (Bordeaux University), and all participants provided written and signed informed consent. There were three panels with some overlap in terms of panel members, and each focusing on different tasks as described hereafter:

Panel 1: 5 experts in wine tasting and especially in the fruity aroma assessment of red wines; 2 females and 3 males aged 37.2 ± 8.6 [mean ± standard deviation (SD)] years old; carried out olfactory evaluation of the wines and HPLC fractions; generated fruity olfactory descriptors for all 10 red wines and selected fractions of interest based on their fruity notes.

Panel 2: 19 judges; 15 females and 4 males aged 30.5 ± 5.2 [mean ± standard deviation (SD)] years old; carried out a free sorting task and associated generation of descriptors.

Panel 3: 14 judges; 11 females and 3 males aged 31.4 ± 7.1 [mean ± standard deviation (SD)] years old; received olfactory training for the realization of sensory profiles adapted from the methodology developed by Pelonnier-Magimel et al. (2020) (2 sessions per week for 6 weeks); focused on fresh and jammy red- and black-berry notes in the sensory training in order to be able to clearly define them; 10 fruity descriptors were selected for the training: fresh strawberries, fresh raspberries, fresh blueberries, fresh black-berries, fresh blackcurrants, strawberry jam, raspberry jam, blueberry jam, black-berry jam and blackcurrant jam (reference recipes are given in Table S1).

2.5.3. Free sorting task: olfactory comparison of HPLC–fruity aromatic reconstitutions (FAR)

Ten HPLC–Fruity Aromatic Reconstitutions (HPLC–FAR) were all presented randomly to Panel 2 for the assessment of the fruitiness of the samples. A free sorting task was carried out to explore perceptive similarities or dissimilarities (Maitre et al., 2010). Each assessor was instructed to combine or separate each HPLC–FAR depending on their fruity character. No constraints were applied in this test and the panelists could create any number of clusters they wanted (with a minimum of 2 clusters and at least 2 samples per cluster). The HPLC–FAR clusters were reported in the dissimilarity matrix (Faye et al., 2004) for all the judges, which was analyzed using Hierarchical Cluster Analyses (HCA, XLSTAT software, Addinsoft, Paris, France). The panelists freely described each cluster they formed (NF ISO 11035:1995). Each HPLC–FAR received a description from each judge according to the clusters made; thus, a free description was associated with the HPLC-FAR. The attributes were gathered according to the grouping methodology to facilitate data interpretation (Lawrence et al., 2013; Sester et al., 2013; Spinelli et al., 2014; Tournier et al., 2007): first, terms or expressions with similar meanings, such as nouns and adjectives, were put in the same semantic group (e.g., fruity, fruits and fruit were put in the same group fruity); then, attributes that belong to the same lexical field were grouped together (e.g., red-berry fruits includes descriptors like strawberry and raspberry, and black-berry fruits includes attributes like blueberry, blackberry and blackcurrant). The percentage citation frequency of the sensory attributes was calculated for each HPLC–FAR based on their occurrence frequencies according to the following formula: CF (%) = (NO/N)*100 (NO = the number of occurrences of the term for each HPLC–FAR and N = the total number of aromatic terms for each HPLC–FAR). In addition, the percentage citation frequencies of the descriptors of HPLC–FAR that belonged to the same cluster were summed to obtain percentage citation frequencies of descriptors for each cluster.

2.5.4. Sensory profiles

Panel 3 carried out a comparative sensory profile evaluation to determine the intensity of six descriptors (fresh red-berry fruits, fresh black-berry fruits, jammy red-berry fruits, jammy black-berry fruits, freshness and overall intensity) (NF ISO 13299: 2016). Three glasses were presented to the panelists for the sensory evaluation of their contents. The first glass contained a hydroalcoholic solution (12% of ethanol and adjusted to pH 3.5 using 5 g/L of tartric acid) supplemented with a mixture of esters comprising 15 esters (5 acetates, 8 ethyl esters and 2 cyclic esters) whose concentrations were fixed based on the average concentrations determined in each HPLC–FAR (Table 2). The second glass contained the same mixture of esters combined with four compounds added at the specific average concentrations found in a cluster that had been formed in the free sorting task: linalool (38 μg/L), α-ionone (0.26 μg/L), β-ionone (0.30 μg/L) and hexanol (150 μg/L). The last glass contained the same mixture of esters combined with four other

Table 2
Sample compositions for sensory profiles.

Chemical family	Compounds	Compounds added to hydroalcoholic solution (12%; pH 3.5)		
		Glass 1 (μg/)	Glass 2 (μg/L)	Glass 3 (μg/L)
Ethyl esters and Acetates	ethyl 2-methylpropanoate	21	21	21
	2-methylpropyl acetate	6	6	6
	ethyl butanoate	52	52	52
	ethyl 2-methylbutanoate	22	22	22
	butyl acetate	1	1	1
	ethyl 3-methylbutanoate	28	28	28
	2-metylbutyl acetate	20	20	20
	3-methylbutyl acetate	226	226	226
	ethyl hexanoate	340	340	340
	hexyl acetate	1	1	1
	ethyl 2-hydroxy-3-methylbutanoate	2	2	2
	ethyl octanoate	14	14	14
	methyl salicylate	5.5	5.5	5.5
	ethyl salicylate	1.8	1.8	1.8
	ethyl 6-hydroxyhexanoate	6	6	6
Monoterpenes	linalool	–	38	–
	α-terpineol	–	–	95
C13-norisoprenoids	α-ionone	–	0.26	0.24
	β-ionone	–	0.30	–
C6 alcohols	Hexanol	–	150	145
	(*Z*)-3-hexenol	–	–	1.8

compounds added at specific average concentrations found in another cluster formed in the free sorting task: α-terpineol (95 μg/L), α-ionone (0.24 μg/L), (*Z*)-3-hexenol (1.8 μg/L) and hexanol (145 μg/L) (Table 2). Each panelist evaluated the samples using a 0–100 mm unstructured scale, where 0 indicated that no odor was perceived and 100 indicated high intensity.

Statistical analyses were performed using XLSTAT software. The sensory data were analyzed using non-parametric tests: Kruskall-Wallis test followed by Dunn post hoc test with level error α = 5% ($p < 0.05$).

3. Results and discussion

3.1. Aromatic descriptions of wine samples

The five experts of Panel 1 generated olfactory attributes to describe the 10 red wines, such as freshness and fresh and jammy red- and black-berry fruity descriptors (Table 1). The wines made from Grenache and Xinomavro were described as having fresh red-berry fruits notes (strawberry and raspberry) and jammy red-berry notes (strawberry) respectively by the experts (80%, 4/5). The Cabernet franc, Merlot and Touriga Nacional wines were described as having jammy red- and black-berry fruit notes (strawberry and blackcurrant) (60%, 3/5) and Agiorgitiko as having jammy strawberry notes (4/5, 80%). Meanwhile, the wines made from Cabernet-Sauvignon, Syrah, Tempranillo and Yiannoudin were characterized as having fresh black-berry fruits notes (60%, 3/5) and jammy black-berry fruits notes (40% 2/5). Finally, Yiannoudin and Syrah were described as having a minty and fresh aroma respectively (60%, 3/5).

3.2. Fraction selection and chemical distribution of aromatic compounds during HPLC fractionation

The semi-preparative HPLC fractionation methodology proposed by Ferreira et al. (1999) was optimized by Pineau et al. (2009) and applied to each red wine aroma extract. The five experts from Panel 1 evaluated

25 fractions (F1 to F25) obtained from the 10 wine aroma extracts. The five fractions F17 to F21 (referred to as HPLC-FAR in this study) exhibited intense fruitiness and were thus selected for the qualitative study of the fresh fruity aroma of wine. This fruitiness is consistent with findings by Lytra, Tempere, de Revel, and Barbe (2012) and Pineau et al. (2009), who identified the presence of such fruity fractions in red wine aroma extracts. F17 to F21 were described using the descriptors fresh and jammy red- and black-berry fruits, amylic notes and fresh. After these fractions had been identified for their aromatic interest, their chemical composition in terms of aroma-related compounds was analyzed using targeted GC-MS. Table 3 shows the elution of 34 aroma compounds in these HPLC-FAR fractions, comprising 19 ethyl esters and acetates, 7 monoterpenes, 3 C13-norisoprenoids and 3 C6 alcohols. A good separation of the aroma compounds based on their polarity was achieved by fractionation using a C18 column. The results of this instrumental analysis showed, as expected and previously demonstrated by Lytra, Tempere, de Revel, and Barbe (2012), that ethyl esters and acetates with low molecular weight and higher polarity were collected in fractions F17–F18, while those with high molecular weight and lower polarity were distributed in fractions F20–F21. Hydroxylated ethyl esters were mainly eluted in the first fractions (F17 and F18) due to their high polarity (Table 3). Methyl and ethyl salicylates were distributed within fractions F19–F20 due to their high molecular weight and relatively low polarity, corroborating a previous study by Pelonnier-Magimel et al. (2022). Additionally, monoterpenes, C13-norisoprenoids and C6 alcohols were present in the HPLC–FAR fractions, and their elution correlated with their polarity and molecular weight. These results are consistent with other work using the same HPLC methodology: Picard et al. (2016) demonstrated the presence of piperitone in fraction F17, which contributed to the minty and fresh aromas of the red wines; more recently, Siebert et al. (2021) showed the presence of α-terpineol, linalool and β-damascenone in fractions F38 and F39 (corresponding to F19 in the HPLC fractionation method of Ferreira et al. (1999) (74% EtOH and 26% water)), with 0.5 mL of eluted solvent collected every minute. Regarding the fruity aroma described in fractions F17 to F21, the studied monoterpenes, C13-norisoprenoids and C6 alcohols potentially contribute to the fruity aroma of HPLC–FAR.

3.3. Olfactory discrimination and classification of HPLC–fruity aromatic reconstitutions (FAR)

The olfactory discrimination of the fruity expression of HPLC–FAR made from 10 different single-varietal red wines was carried out by Panel 2 in a free sorting task. The results of the free sorting task are presented in Fig. 1 as a Hierarchical Cluster Analysis (HCA) applied to a dissimilarity matrix. Fig. 1 highlights three distinct HPLC–FAR clusters. In terms of fruitiness as rated by Panel 2, the three clusters show dissimilarities relative to each other, while within a single cluster similarities can be observed. Cluster 1 (in green) comprises HPLC–FAR made from Yiannoudin, Syrah, Touriga Nacional, Tempranillo and Cabernet-Sauvignon red wines. The HPLC–FAR made from the Bordeaux Cabernet-Sauvignon wine shows a fruity sensory similarity with that made from the other wines belonging to Cluster 1 (Yiannoudin, Syrah, Touriga Nacional and Tempranillo). Cluster 2 (in pink) comprises HPLC–FAR made from Agiorgitiko and Merlot red wines. The HPLC–FAR produced from the Bordeaux red wine made from the Merlot grape variety shows a fruity sensory similarity with HPLC–FAR made from Agiorgitiko red wine. Cluster 3 (in brown) comprised HPLC–FAR made from Grenache, Xinomavro, and Cabernet franc red wines. HPLC–FAR made from the Bordeaux Cabernet franc shows a fruity sensory similarity with HPLC–FAR from the Grenache and Xinomavro red wines.

Fig. 2 shows the citation frequencies of the olfactory descriptors generated and associated with each cluster by Panel 2. The desirable fruity aromas of the ten red wines (i.e., fresh and jammy red- and black-berry fruit aromas) as perceived by Panel 1 were also perceived in

Table 3
Distribution (presence or absence) of aromatic compounds with fruity or non-fruity notes in 5 fractions which composed the 10 HPLC – FAR [a] [a:] F: fraction number; [b]: olfactory descriptors of compounds found in The GoodScent Company databases, 2021; "x" indicates the presence of listed compounds eluted in the various fractions; "-"indicates the absence of listed compounds.

Aroma compounds	Olfactory descriptors[b]	F17	F18	F19	F20	F21
Ethyl esters						
ethyl propanoate	Sweet, fruity, juicy	x	–	–	–	–
ethyl butanoate	Fruity, juicy, pineapple	x	–	–	–	–
ethyl hexanoate	Sweet, fruity, pineapple	–	–	–	x	–
ethyl octanoate	Fruity, waxy, banana	–	–	–	–	x
ethyl 2-methylpropanoate	Strawberry, kiwi, fruity, solvent	x	x	–	–	–
ethyl 2-methylbutanoate	Fruity, kiwi	–	x	x	–	–
ethyl 3-methylbutanoate	Cheesy, fruity	–	x	x	–	–
ethyl 2-hydroxy-3-methylbutanoate	Fruity, pineapple, strawberry	x	x	–	–	–
ethyl 3-hydroxybutanoate	Fruity, strawberry	x	–	–	–	–
ethyl 2-hydroxy-4-methylpentanoate	Blackberry	x	–	–	–	–
ethyl 2-hydroxyhexanoate	Blackberry	x	x	–	–	–
ethyl 6-hydroxyhexanoate	Fruity	x	x	–	–	–
Acetates						
2-methylpropyl acetate	Sweet, fruity, banana	x	–	–	–	–
butyl acetate	Solvent, fruity, banana	–	x	x	–	–
2-methylbutyl acetate	Sweet, fruity banana	–	x	x	x	–
3-methylbutyl acetate	Sweet, fruity, banana	–	x	x	x	–
hexyl acetate	Apple, fruity, pear	–	–	–	–	x
Cyclic esters						
methyl salicylate	Wintergreen, mint	–	–	x	x	–
ethyl salicylate	Sweet, wintergreen, mint	–	–	x	x	–
Monoterpenes						
1,4-cineole	Cooling, pine, minty	–	–	x	x	–
1,8-cineole	Eucalyptus, herbal, camphor	–	–	x	x	–
linalool	Citrus, flowery, sweet	–	–	x	x	–
terpinen-4-ol	Peppery, woody, earthy	–	–	x	x	–
menthol	Peppermint, cool, woody	–	–	x	x	–
α-terpineol	Pine, terpene, lilac	–	–	x	x	–
β-citronellol	Flowery, rose, citrus	–	–	x	x	–
nerol	Sweet, neroli, citrus	–	–	x	x	–
geraniol	Flowery, fruity, rose	–	–	x	x	–
C13-norisoprenoids						
β-damascenone	Sweet, fruity, rose	–	–	x	x	x
α-ionone	Flowery, sweet, violet	–	–	–	x	x
β-ionone	Violet, fruity, blackberry	–	–	–	x	x
C6 alcohols						

(*continued on next page*)

Table 3 (*continued*)

Aroma compounds	Olfactory descriptors[b]	F17	F18	F19	F20	F21
hexanol	Ethereal, fusel, oil	x	x	–	–	–
(*Z*)-3-hexenol	Fresh, fruity, grassy	x	x	–	–	–
(*E*)-3-hexenol	Green, leafy, flowery	x	x	–	–	–

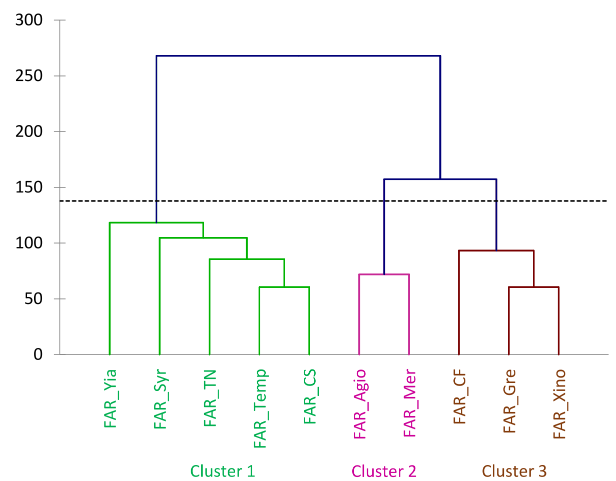

Fig. 1. Dendrogram obtained by HCA using a dissimilarity matrix applied to free sorting data resulting from the evaluation of HPLC–Fruity Aromatic Reconstitution (FAR) (Agio: Agiorgitiko; CF: Cabernet franc; CS: Cabernet-Sauvignon; Gre: Grenache; Mer: Merlot; Syr: Syrah; Temp: Tempranillo; TN: Touriga Nacional; Xino: Xinomavro; Yia: Yiannoudin).

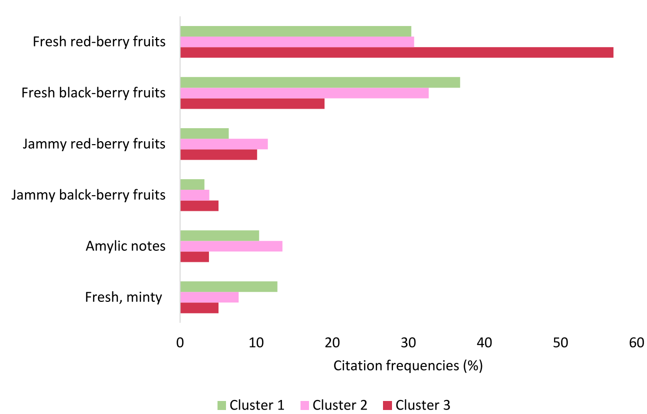

Fig. 2. Citation frequencies of olfactory descriptors generated by Panel 2 (19 judges) during the sensory evaluation of HPLC–Fruity Aromatic Reconstitution (FAR). Cluster 1: HPLC–FAR made from Yiannoudin, Syrah, Tempranillo, Touriga Nacional and Cabernet-Sauvignon wines; Cluster 2: HPLC–FAR made from Merlot and Agiorgitiko wines; Cluster 3: HPLC–FAR made from Cabernet franc, Grenache and Xinomavro wines.

HPLC–FAR by Panel 2: Cluster 1 had the highest citation frequency for the descriptors fresh black-berry fruits (36.8%) and fresh and minty aromas (12.8%), Cluster 2 for the descriptors amylic (13.5%) and jammy red-berry fruit notes (11.5%), and Cluster 3 for fresh red-berry fruit notes (57%). It was thus possible to characterize all three clusters as having olfactory fruity descriptors: fresh and jammy red- and black-berry fruit notes, amylic notes, and fresh and minty aromas; these are in agreement with those found in the red wines by the five experts of Panel 1. HPLC–FAR made from grape varieties belonging to the same cluster showed aromatic similarities. HPLC–FAR made from the three "traditional" red grape varieties from the Bordeaux vineyards (Cabernet-Sauvignon, Cabernet franc and Merlot) falls into three distinct clusters containing "non-traditional" grape varieties. These results open up perspectives for the potential use and replacement of a Bordeaux variety by other varieties within the same cluster, and for evolving the red wine blending strategy.

3.4. Chemical discrimination of the sensory clusters

To better understand Panel 2's sensory discrimination of the ten HPLC–FAR, their chemical composition was studied. The concentrations of each compound in all the HPLC–FAR, as well as the sum of the total esters, monoterpenes, C13-norisoprenoids and C6 alcohols, were determined (Table S2). Furthermore, the concentrations of each compound in the three clusters, resulting from the Panel 2 sorting task, were determined and compared using ANOVA. The results show that nine compounds out of 27 discriminate the three clusters (Table 4). Cluster 1 comprises the highest levels of linalool, α- and β-ionones, hexanol, ethyl hexanoate and ethyl 6-hydroxhexanoate. The literature provides evidence of ethyl hexanoate and octanoate contributing to red-berry aromas (Pineau et al., 2009), while ethyl 6-hydroxyhexanoate has been reported to boost the fruity aroma of red wines as a result of perceptual interactions (Pineau et al., 2009). As regards methyl salicylate, Pelonnier-Magimel et al. (2022) have shown that its addition to red wines at 60 μg/L increases the coolness attribute. Studies on the olfactory impact of C13-norisoprenoids, such as β-ionone or β-damascenone, on the fruity aroma have shown that these compounds increase the red- and black-berry aromas in red wines (Escudero et al., 2007; Tomasino & Bolman, 2021). In a recent study by Garbay et al. (2023), specific concentrations of a pool of compounds comprising linalool, α-terpineol, α- and β-ionones and hexanol were added to a red wine with cooked fruit aromas; this had the effect of increasing the intensity of the fresh black-berry notes while reducing the perceived intensity of the cooked fruit aromas. Cluster 2 showed the highest amount of ethyl 2-methylpropanoate. In the literature, this compound is known to contribute to the amylic and fruity aroma of red wines (Lytra, Tempere, Revel, & Barbe, 2012; Pineau et al., 2009). Finally, Cluster 3 comprised the highest levels of ethyl hexanoate, α-terpineol, α-ionone, (*Z*)-3-hexenol and hexanol. To our knowledge, there is no reference in the literature to the sensory contribution of (Z)-3-hexenol, which individually presents fresh, fruity, and grassy notes (according to The GoodScent Company databases, 2021), to the fruity nuances of red wines.

3.5. Olfactory impact on fruity aroma of the addition of monoterpenes, C13-norisoprenoids and C6 alcohols to a model wine solution

The sensory profile task conducted by Panel 3 aimed to investigate the role of the aromatic compounds that significantly discriminated the clusters obtained previously in the free sorting task, specifically examining the potential contribution of monoterpenes, C13-norisoprenoids and C6 alcohols to the perception of fruitiness. The objective of the sensory evaluation was to determine if the aroma compounds (i.e., linalool, α-terpineol, α- and β-ionones, hexanol and (*Z*)-3-hexenol) that discriminated the three clusters from a chemical point of view could also impact the intensity of the perceived fruity aroma. The olfactory contribution of esters in fruity aroma perception has been extensively studied in the literature, and these compounds are known to enhance the fruity aroma in red wines (Pineau et al., 2009; Lytra, Franc, Cameleyre, & Barbe, 2017). The choice of compounds added to glasses 2 and 3 aimed to vary only the concentration of linalool, α-terpineol, α- and β-ionones, hexanol and (*Z*)-3-hexenol according to the composition of Cluster 1 and 3. A fixed concentration of esters was maintained in all

Table 4
Average concentrations (μg/L) of the aroma compounds in the 3 HPLC – FAR clusters and *p* value of the ANOVA [a] [a]: letters in each row indicate significant differences between clusters per compound by ANOVA Tukey test ($p < 0.05$). Numbers in italics represent the $p < 0.05$. Numbers in bold represent the highest value for compounds with a significant level. Cluster 1: HPLC–FAR made from Yiannoudin. Syrah. Tempranillo, Touriga Nacional and Cabernet-Sauvignon wines; Cluster 2: HPLC–FAR made from Merlot and Agiorgitiko wines; Cluster 3: HPLC–FAR made from Cabernet franc, Grenache and Xinomavro wines.

Aroma compounds	Pr > F (p-value)	HPLC–FAR Cluster 1			HPLC–FAR Cluster 2			HPLC–FAR Cluster 3		
		Mean	Min	Max	**Mean**	Min	Max	**Mean**	Min	Max
Ethyl esters and acetates										
ethyl 2-methylpropanoate	*< 0.0001*	20 (b)	8.7	39	**42 (a)**	35	49	8.1 (b)	–	23
2-methylpropyl acetate	*0.005*	2.7 (b)	–	8.3	12 (a)	–	23	6.2 (ab)	–	10
ethyl butanoate	*0.002*	40 (b)	31	50	77 (a)	35	119	55 (ab)	42	64
ethyl 2-methylbutanoate	0.094	25 (a)	5.5	31	29 (a)	12	47	26 (a)	17	35
butyl acetate	0.662	1.9 (a)	–	10	0 (a)	–	–	0 (a)	–	–
ethyl 3-methylbutanoate	0.345	37 (a)	10	44	45 (a)	21	68	28 (a)	22	32
2-metylbutyl acetate	0.320	19 (a)	11	26	26 (a)	22	31	19 (a)	12	26
3-methylbutyl acetate	*0.002*	239 (ab)	187	319	303 (a)	148	457	152 (b)	110	227
ethyl hexanoate	*0.0003*	**368 (a)**	234	508	202 (b)	170	235	**387 (a)**	172	541
hexyl acetate	0.353	1.0 (a)	–	2.7	1.2 (a)	–	2.3	0.2 (a)	–	0.4
ethyl 2-hydroxy-3-methylbutanoate	0.689	2.4 (a)	2.2	2.8	2.4 (a)	2.3	2.4	2.4 (a)	2.3	2.6
ethyl octanoate	0.088	29 (a)	–	74	0 (a)	–	–	0 (a)	–	–
methyl salicylate	*0.047*	7.9 (a)	3.5	12.5	5.8 (ab)	3.6	7.9	2.8 (b)	2	3.6
ethyl salicylate	0.349	2.7 (a)	0.9	8	1.3 (a)	1	1.7	1.3 (a)	0.9	1.7
ethyl 6-hydroxyhexanoate	*0.014*	**2.5 (a)**	2	3.4	1.9 (b)	1.8	2	1.9 (b)	1.9	1.9
Monoterpenes										
1,4-cineole	0.761	2.4 (a)	–	6.1	2.2 (a)	2.2	2.2	2.3 (a)	2	2.8
1,8-cineole	0.423	2.6 (a)	–	6.6	0 (a)	–	–	0.8 (a)	–	2.5
Linalool	*0.002*	**38 (a)**	12	57	12 (b)	12	16	17 (b)	5.5	30
terpinen-4-ol	0.133	8.9 (a)	6.7	12	10 (a)	10	10	10 (a)	8.2	13.6
α-terpineol	*< 0.0001*	40 (b)	24	55	29 (b)	28	30	**95 (a)**	24	139
Nerol	0.117	2.9 (a)	2.4	3.9	2.6 (a)	2.5	2.6	2.6 (a)	2.5	2.8
Geraniol	0.318	4.8 (a)	2.4	11	2.7 (a)	2.3	3	2.9 (a)	2.7	3.1
C13-norisoprenoids										
β-damascenone	0.512	1.1 (a)	1	1.2	0.9 (a)	0.8	0.9	1 (a)	0.9	1.1
α-ionone	*0.0002*	**0.26 (a)**	0.2	0.3	0.17 (b)	0.15	0.18	**0.24 (a)**	0.17	0.33
β-ionone	*< 0.0001*	**0.31 (a)**	0.2	0.4	0.19 (c)	0.17	0.20	0.26 (b)	0.19	0.34
C6 alcohols										
Hexanol	*0.0004*	**150 (a)**	48	331	47 (b)	41	53	**147 (a)**	75	282
(*Z*)-3-hexenol	*0.008*	0.4 (b)	–	1.8	0.9 (b)	–	1.7	**1.8 (a)**	1.7	1.8

glasses to study only the impact of these 6 compounds in a simplified fruity matrix (Table 2). Sensory profiles applied on a 100 mm-scale of intensity revealed significant differences between glasses 1, 2, and 3 in overall fresh red- and black-berry fruits, jammy black-berry fruits and freshness notes (Fig. 3). However, the average score for jammy black-berry fruit notes was the same. Fig. 3 shows that the addition of linalool, α- and β-ionones and hexanol at 38, 0.26, 0.30, and 150 μg/L respectively in glass 2 significantly increased overall intensity, fresh black-berry fruit notes and freshness compared to glass 1. These findings align with a recent study by Garbay et al. (2023), in which the addition of linalool, α-terpineol, α- and β-ionones and hexanol, as well as other monoterpenes, C6 alcohols, and cyclic esters, at specific concentrations to a red wine was found to increase the perception of fresh black-berry fruit notes and decreased the perceived intensity of cooked fruit aromas. In addition, the role of β-ionone as an enhancer of red- and black-berry fruit notes is well-known in the literature (Tomasino & Bolman, 2021;

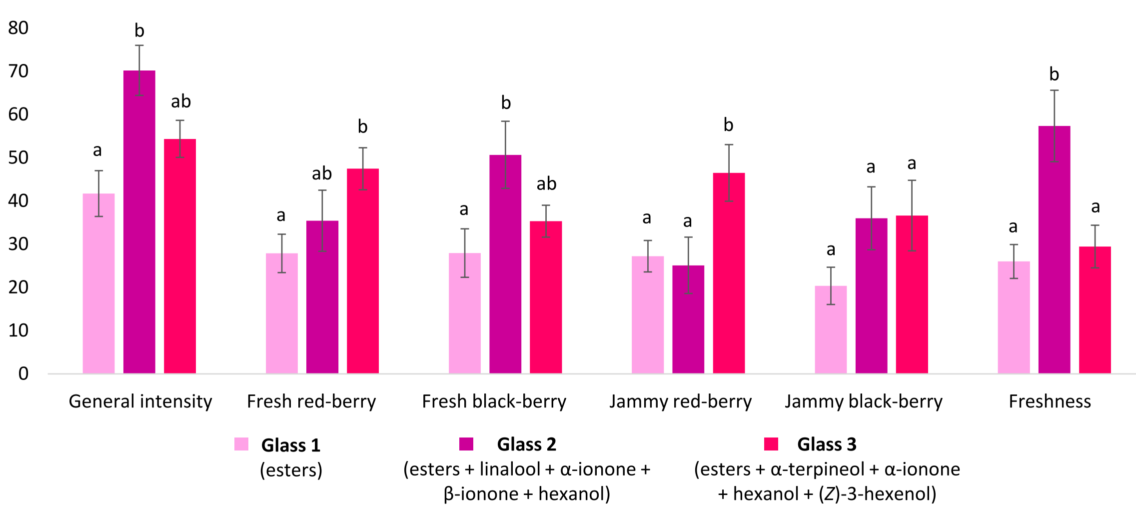

Fig. 3. Aromatic impact of the addition of linalool, α-terpineol, α- and β-ionones, hexanol and (*Z*)-3-hexenol at specific concentrations (Table 1) in hydroalcoholic solution mixed with esters. Letters indicate significant differences between samples (Glass 1, 2 or 3) per average mean intensity of descriptors determined by Kruskall-Wallis test followed by Dunn post hoc test.

Escudero et al., 2009). The addition of α-terpineol, α-ionone, hexanol and (*Z*)-3-hexenol at 95, 0.24, 145, and 1.8 μg/L respectively in glass 3 showed a significant increase in fresh red-berry and jammy red-berry fruit notes. These results highlight the sensory impact of linalool, α-terpineol, α- and β-ionones, hexanol and (*Z*)-3-hexenol on the fruity aromas in a model solution containing esters, suggesting that these compounds can modulate the red- and/or black-berry fruit nuances in red wine. Moreover, the fruity modifications perceived by Panel 3 in the sensory evaluation of glass 2 corresponding to the descriptors generated for Cluster 1; i.e., fresh black-berry fruit notes and freshness. Additionally, the intensity of the fruity notes of glass 3 corresponding to the descriptors generated for Cluster 3, such as red-berry fruit notes. The main results obtained during the sensory analyses performed by Panel 2 and Panel 3 are summarized in Table 5. These findings provide new options for blending strategies involving Bordeaux wines. The fact that the 3 HPLC–FAR made from the "traditional" grape varieties of Bordeaux were categorized into three distinct clusters suggests that they had aromatic similarities with the other "non-traditional" varieties. Therefore, in further analyses regarding Cluster 1, Cabernet-Sauvignon red wine could potentially be blended with a low percentage of red wines made from Tempranillo, Syrah, Touriga Nacional and even Yiannoudin, with the aim of modulating the increase of linalool, α- and β-ionones and hexanol, and thus potentially enhancing the fresh black-berry fruit aromas. This could also be applied to Clusters 2 and 3 to modulate the red-berry aromas by blending Merlot red wine with Agiorgitiko red wine, and Cabernet franc red wine with a small amount of Xinomavro or Grenache red wines.

4. Conclusion

The fruity aroma of red wine is a complex topic due to the diverse interactions between volatile compounds and it is described by a wide range of fruity nuances. In the present study, three sensory clusters with distinct fruity aromas were identified via sensory evaluations of the fruity fractions of ten commercial red wines made from single grape varieties characterized by an increasing maturity gradient. The results of the targeted GC-MS analysis showed variations in concentration of compounds among the HPLC–FAR, and nine aroma compounds were identified as discriminating the sensory clusters. These compounds comprised three esters but also linalool, α-terpineol, α- and β-ionones, hexanol and (*Z*)-3-hexenol, and they likely contributed to the olfactory discrimination of the sensory clusters; i.e., their addition to the fruity model solution in the studied concentrations contributed to the increase in the intensity of fresh red- and/or black-berry fruit notes. These findings indicate that there is potential to develop viticultural practices by introducing late-ripening grape varieties into the "traditional" Bordeaux blend. In the future, it would be pertinent to consider introducing these grape varieties into Bordeaux wine blends. For instance, replacing Merlot with other varieties and examining whether the new blends maintain a similar fruity typicity to that of traditional Bordeaux wines. One other perspective related to this work would be to understand whether the variations in the concentrations of the highlighted compounds are typical of the studied grape varieties and whether they can be used to differentiate them. To this end, it would be beneficial to introduce additional references of red wines from the grape varieties studied in this work, sourced from different regions, to enhance the initial approach. Furthermore, given the vast diversity of red grape varieties, it would be essential to explore other grape varieties not included in this study, whose fruity aroma profiles warrant further investigation. Moreover, this study opens up new oenological avenues regarding the modulation of the release of specific compounds during the vinification process to enhance the fresh fruit notes in red wines. Further analyses also need to be conducted to determine the role of the studied chemical families and/or compounds in the fruity aroma of a complex matrix such as wine; for example, by adding them separately to a real red wine.

Table 5
Summary of the olfactory descriptors of the 3 clusters generated by Panel 2 and the corresponding aroma compounds which discriminated each cluster. The results of the sensory profiles are also reported in this table.

Grape varieties of HPLC – FAR	Sensory clusters obtained from the free sorting task (Panel 2)	The most cited descriptors for HPLC–FAR (F17-21) (Panel 2)	Discriminating aroma compounds for each cluster[a]	Sensory profiles of a fruity simplified matrix (Panel 3)
Yiannoudin Syrah Touriga Nacional Tempranillo Cabernet-Sauvignon	Cluster 1	Fresh black-berry fruits	**linalool, α-ionone, β-ionone, hexanol**, ethyl hexanoate and ethyl 6-hydroxyhexanoate	**Glass 2** ↑ overall intensity ↑ fresh black-berry ↑ freshness
Agiorgitiko Merlot	Cluster 2	Amylic, jammy red-berry fruits	ethyl 2-methylpropanoate	–
Cabernet franc Grenache Xinomavro	Cluster 3	Fresh red-berry fruits	**α-terpineol, α-ionone, hexanol, (*Z*)-3-hexenol** and ethyl hexanoate	**Glass 3** ↑ fresh red-berry ↑ jammy red-berry

[a] Compounds in bold are those added at specific concentrations to Glasses 2 and 3 for the sensory profiles (cf. Table 1).

CRediT authorship contribution statement

Justine Garbay: Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis. **Margaux Cameleyre:** Writing – review & editing, Methodology. **Nicolas Le Menn:** Writing – review & editing, Methodology, Formal analysis. **Laurent Riquier:** Methodology. **Jean-Christophe Barbe:** Writing – review & editing. **Georgia Lytra:** Writing – review & editing, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Acknowledgements

This work received financial support from Bordeaux Sciences Agro (France), the Region of Nouvelle-Aquitaine (Région Nouvelle-Aquitaine, France), the Union des Œnologues de France, the Bordeaux Wine Council (C.I.V.B - Conseil Interprofessionnel du Vin de Bordeaux, France) and the GPR (Grands Programmes de Recherche) Bordeaux Plant Sciences (Université de Bordeaux, France).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2024.116481.

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