Research Article

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Studying how dry extract can affect the aroma release and perception in different red wine styles

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Abstract

BACKGROUND: Four red wine matrices representing different red wine styles with the same VOCs (volatile organic compounds), were obtained by enriching a bleed wine with increasing amounts of deodorized dry extract obtained from the pressed wine of the same vinification. The release of VOCs was determined by solid phase micro-extraction-gas chromatography–mass spectrometry (SPME-GC–MS), in conditions mimicking those applied during sensory assessments.

RESULTS: Results show that even though the perception of the overall odor intensity was not significantly influenced by the matrix, this latter modulated the odor profiles: at rising wine dry extract, fruity, floral odors decreased, while dehydrated fruit, woody-toasty, vegetal-earthy notes increased. These changes cannot be fully explained by the observed significant influence of the matrix on the release of VOCs or by their correlations with the considered matrix components (ethanol, residual sugars, phenolics, pH), but findings suggest that perceptual interactions are involved.

CONCLUSION: This study could be useful in pressing and blending management for wine aroma quality also considering wine compositional trends under the current climate change context.

Supporting information may be found in the online version of this article.

Keywords: red wine matrix; polyphenols; dry extract; aroma release; sensory interactions

INTRODUCTION

The olfactory perception of wine aroma is influenced by the interaction of volatile molecules with other matrix components, impacting their volatility and concentration in the headspace.[1-3] The nature of these interactions differs according to the physical–chemical properties of the aroma compounds (e.g., molecular size, functional group, polarity, solubility, volatility) and the binding that may occur among wine components via covalent, hydrophobic, or hydrogen bonds, or via formation of inclusion complexes.[2] In addition to these physical–chemical interactions, perceptual interactions must also be considered to understand wine sensory perception.[4]

Among the matrix components affecting the partitioning of the volatile organic compounds (VOCs), ethanol, polyphenols, and residual sugars have been reviewed as the most relevant up to date. Indeed, wine sugars have been found to influence VOCs repartition between wine and its headspace under both orthonasal and retronasal conditions, mostly as a consequence of their high water solubility that reduces the solvating capacity of water molecules toward VOCs, and ability to interfere the interactions between VOCs and salivary proteins.[3,5] The influence of ethanol on the release of VOCs and perception in wine and other alcoholic beverages has been widely studied.[6] The studies showed that ethanol concentration affected the solubility of the VOCs and their distribution between gas and liquid phases, due to changes in wine polarity, thus modulating the overall aroma perception in wines.[6] Increasing ethanol contents was negatively correlated with the release of several VOCs, mostly esters, but also terpenes, ketones, methoxypyrazines and volatile phenols, and with the perception of fruity, floral, and herbaceous aromas.[6] Globally, ethanol tends to diminish the intensity of aromas, either by masking their odor or by decreasing their volatility.[7] Regarding the interactions between polyphenols and VOCs and their impact on the release of VOCs and perception, data reported in the literature suggest that increasing polyphenols concentrations lead to a

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greater release (salting-out) of the most hydrophilic volatiles. This is likely because the increase in polyphenols reduces the solvating capacity of water molecules toward VOCs. On the contrary, the most hydrophobic VOCs seem to be more retained at higher phenolic concentrations, probably due to hydrophobic intermolecular interactions between polyphenols and VOCs. As recently reviewed,[8] few experiments have been conducted to measure the sensory impact of polyphenols on aromas perception. Results show a negative impact of increasing polyphenol concentrations on the intensity of perceived fruity, citrus, strawberry, cooked fruit, and floral odors. A tendency towards the accentuation of spicy, herbaceous, and sweet pepper notes was also observed.[9]

Our consideration is that in the current scenario of climate change, grape composition is shifting, and the aforementioned wine matrix components are among the most impacted.[10]

In this context, how the wine VOCs and matrix interact and how these interactions impact sensory perception seems a relevant subject in the current wine research. The subject has been addressed by several studies but as previously highlighted[11] only a few of them coupled sensory and physical–chemical approaches[1,12-14] to test the sensory impact of VOCs–matrix interactions on wine perception and most of them were conducted in wine model solutions. The comprehension of interaction mechanisms and of their sensory implications is not yet fully understood, also due to the chemical complexity of the wine matrix.

Pressing and blending are the main enological practices used to manage the wine style primarily as color and mouthfeel sensations affecting consumer's liking and preferences.[15,16] These winemaking steps impact red wine matrix composition, mainly referring to phenolics and dry extract. These two parameters can be impacted also by climatic abiotic factors in the vine, as for example, water deficit that can lead to an increase in the wine dry extract.[17]

In this frame, our study aims at investigating by chemical and sensory analyses, the matrix effects on wine aroma release and perception, in wine matrices representative of different red wine styles in terms of dry extract. In this optic, through a progressive enrichment, starting from real wines [bleed wine (BW) and pressed wine (PW)], four different wine matrices were obtained; they were made exclusively of endogenous molecules and characterized by a natural constant VOC composition and diverse matrix compositions representative of low, medium, high, and very high total dry extracts. These compositional characteristics allowed to test the release behavior and the sensory impact of VOCs in response to the different matrices and to study the correlations between some specific compositional features and the release of VOCs in conditions that were designed to be as close as possible to real ones.

MATERIALS AND METHODS

Wine samples

A BW and the corresponding PW produced within the same vinification of Aglianico grapes (Mirabella Eclano, Campania, Italy) were used. The BW was obtained by subtracting an aliquot of the fermenting wine from the tank and completing the alcoholic fermentation in absence of the skins. In this way the extraction of phenolics was limited, so that BW was characterized by a poor phenolic non-volatile fraction. The PW was obtained from the pressing of the drained pomace at the end of alcoholic fermentation and therefore it was characterized by high phenolic non-volatile fraction. To get the experimental matrices, BW was progressively enriched with increasing amounts of PW deodorized dry extract, obtained and checked by gas chromatography–mass spectrometry (GC–MS) as previously reported.[18] Briefly, PW wine was treated in an ultrasound bath (20 (±2) °C, 30 min, Transsonic 460 H; Elma, Germany) and then evaporated at 30 (±2) °C under reduced pressure (Rotavapor R-210; Büchi, Switzerland), until a weight loss of approximately 95% was reached (~90 min).

Four red wine matrices were obtained according to the BW/PW ratios in parentheses (*v/v*; where PW volumes refer to the PW volume before deodorization and drying): B (1:0, 3690 mL BW); $B_1P_{0.5}$ (1:0.5, 3690 mL BW + dry extract from 1845 mL PW); $B_1P_{1.5}$ (1:1.5, 3690 mL BW + dry extract from 5535 mL PW); B_1P_2 (1:2, 3690 mL BW + dry extract from 7380 mL PW). In this way, the four matrices were characterized by the same fraction of VOCs (that of BW) and by an increasing non-volatile composition, without the addition of exogenous compounds.

Base chemical and polyphenol analyses

Alcoholic strength by volume, reducing sugars, pH, were measured according the OIV *Compendium of International Methods of Wine and Must Analysis*.[19] Total phenolics, BSA reactive tannins, total anthocyanins and free anthocyanins were determined by the Harbertson–Adams assay.[20] The analyses were performed in duplicate.

Chemical analysis of the volatile fraction

Isolation of VOCs by SPME

The isolation of VOCs was carried out by headspace-solid phase micro-extraction (HS-SPME). To carry out the analyses under conditions that were as representative as possible of those occurring during wine tasting in terms of wine volume and temperature, wine VOCs were pre-concentrated directly from an INAO tasting glass, sealed with a silicone lid, which allowed the hermetic sealing of the glass. Then, 30 mL of each sample, added with 2-octanol (Sigma Aldrich, St Louis, MO, USA) as internal standard (357 mg/L/ethanol) were analyzed. During the isolation of the VOCs, the glass containing the sample was maintained at constant temperature in a thermostatic bath at 25 (±2) °C for 10 min. A DVB/CAR/PDMS (divinylbenzene/carboxen/polydimethysiloxane; 50/30 µm thickness, coating phase; 2 cm length) fiber was exposed to the headspace of the samples for 30 min by a SPME manual fiber holder (Supelco, Bellefonte, PA, USA). Each sample was extracted in triplicate.

GC–MS analysis of VOCs

For GC–MS analysis, chromatographic conditions and identification procedure were the same as those reported by Piombino *et al.*[21] The analyses were performed in scan mode using a GC/MS-QP2010 quadrupole mass spectrometer (Shimadzu, Kyoto, Japan) equipped with a DB-WAX column [60 m, 0.25 mm inner diameter (i.d.), 0.25 µm film thickness; J&W Scientific Inc., Folsom, CA, USA]. The injector and the electronic source were kept at temperatures of 250 and 230 °C, respectively. The SPME fiber was desorbed in the injector for 10 min, in splitless mode. The oven temperature was maintained at 40 °C for 5 min, and then increased by 2 °C/min, up to 220 °C and held for 20 min. Helium was used as a carrier gas with a flow of 1.3 mL/min. Electron impact mass spectra were recorded with an ion source energy of 70 eV.

The identification of the compounds was performed by comparison of their retention times and their mass spectra with those of pure reference standards under the same chromatographic

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conditions. They were further confirmed by comparison of the mass spectra obtained for each compound with those stored in the database of the National Institute of Standards and Technology (NIST). The relative concentrations (semiquantitative analysis) of detected compounds were expressed as a ratio of the response (peak area) of each compound against the response of the internal standard.

Sensory analysis

Panel

The jury was composed of 19 subjects (nine males and ten females; 21–49 years) recruited and selected among 24 students and researchers of the Division of Vine and Wine Sciences (Department of Agricultural Sciences, University of Naples Federico II, Avellino, Italy) that were interested and available to participate to the study. They were all expert wine tasters with previous experience in performing sensory assessment and wine tasting. All procedures were conducted in accordance with the ethical standards of the institutional and/or national research committee and with the 1964 Helsinki declaration and its later amendments or comparable ethical standards. The appropriate protocols for protecting the rights and privacy of all participants were utilized during the execution of the research, assuring no release of participant data without their knowledge. All data were collected anonymously. Participation was on a voluntary basis with no coercion, and prior to the experiments, tasters were required to sign a written informed consent form full disclosing the voluntary participation (with the option to interrupt participation at any time), the type of research, the study requirements and risks with agreement to smell reference solutions and wines with no consumption/ingestion.

Procedure

Panel training: the training of the judges with olfactory stimuli was aimed at memorizing, recognizing, describing, and discriminating odor stimuli. It was performed according to procedures and standards recently reported.[18] Briefly, subjects were provided with a list of 11 odor families (fruity; dehydrated fruits; dried fruits: nuts; floral; vegetal; spicy; toasted; woody; earthy; alcoholic; off-odors: phenolic, sulfurous, cork taint, modernized/oxidized) selected from the literature and 24 odor standards representative of different odor families and wine volatiles, detailed in Supporting Information Table S1. Panelists were asked to smell each standard (20 mL of water solution in covered disposable plastic cups served according to a randomized order), and to recognize the corresponding odor descriptor(s) or family(ies).

Judges were also trained in rating the descriptors using the following scale: 1 = very weak, 2 = weak, 3 = medium, 4 = strong, 5 = very strong, with half values allowed. Four training sessions were held on a weekly basis. Subjects were selected based on the training performances on olfactory stimuli recognition. Data collected from the training sessions were used to calculate the frequency of citations for standards correctly matched with proper descriptor(s). Only the terms with an association frequency ≥ 85% (percentage of judges that matched the correct descriptor to a given standard) were considered as consensually associated to the corresponding standards. At the end of each training session, the perceived sensations were discussed with the participants to prevent overlapping and redundancies among terms and to help their memorization.

Sensory assessment: to verify the absence of off-odors and off-tastes in the experimental matrices, an informal check was conducted internally at the laboratory by five judges selected based on the best performances in the training phase. Furthermore, a triangle test was performed by 24 subjects (11 males and 13 females; 20–51 years) according to ISO 4120:2021(E)[22] to check by smell the olfactory efficacy of the deodorization. No significant smell differences ($P < 0.05$) were detected, therefore the four experimental matrices were analyzed applying the Rate-All-That-Apply (RATA) method. The descriptive sensory approach was applied to investigate if the differences detected in the VOCs released affected the aroma sensory profile of the matrices. Judges were asked to smell each wine sample and to rate the intensity of the perceived odor descriptors, picking them from the list considered in the training phase. The same scale on which they had been trained was used to rate the descriptors. A value of zero was attributed by the experimenter to the terms that did not apply to the sample. The analyses were carried out on a weekly basis and performed in duplicate, in two separate sessions. In each of the two sessions, all judges analyzed all the wine samples. At the end of each analytical session, judges were asked to rank wines in terms of increasing overall odor intensity by applying a ranking test according to ISO 8587:2006.[23] The ranking test was applied to assess if the overall odor intensity changed with increasing levels of the dry extract.

All along the sensory assessment, for each sample, 30 mL of wine were served in INAO tasting black glasses coded with three-digit random numbers and presented in a randomized order, to minimize order and carryover effects. Wines were served at room temperature (21 ± 1 °C) and evaluated in individual booths.[24]

Data analysis

Data on basic compositional parameters, phenolics, volatiles and olfactory sensory characteristics were treated by analysis of variance (ANOVA) and multiple comparison Tukey HSD (honestly significant difference) *post hoc* test ($P < 0.05$) to test significant differences among the four wine matrices.

The ranking test data were analyzed by means of Friedman's test followed by a Nemenyi's multiple comparison ($P < 0.05$) with the aim of testing significant differences between ranks in terms of overall odor intensity.

Relationships between the four wine matrices, basic compositional parameters, phenolics, volatiles and olfactory sensory variables (as supplementary variables), were investigated by a principal component analysis (PCA) (Pearson, $P < 0.05$).

Data were processed with XLStat (version 2019.6), an add-in software package for Microsoft EXCEL (Addinsoft Corp., Paris, France).

RESULTS AND DISCUSSION

The four wine matrices obtained by the applied enriching strategy, and exclusively made of endogenous molecules, had a natural constant VOC composition (i.e., VOCs of BW) with the other parameters such as pH, residual sugars, and phenolics, significantly increasing from B to B_1P_2 (Fig. 1 and Table S2) obtained as described in the section 'Wine samples'. Only the alcoholic degree slightly decreased (up to 1% *v/v*) likely due to a small dilution effect (a very small volume increase occurred when BW was progressively enriched with PW). Ethanol was therefore considered as a compositional variable in the correlation study and not adjusted in order to avoid further volume changes that would have impacted the wine/headspace ratio. Such compositions

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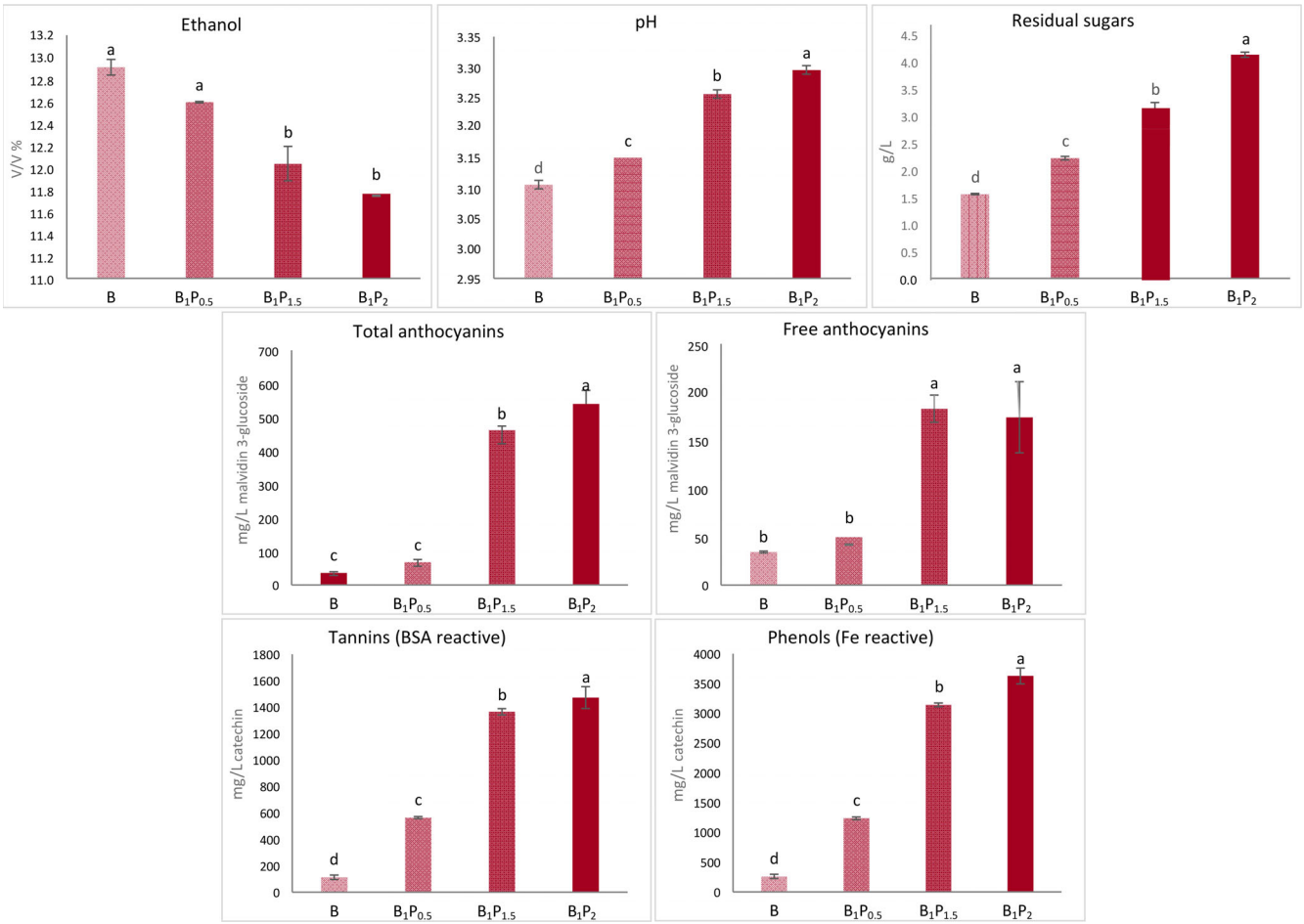

Figure 1. Basic compositional parameters and phenolics of the experimental wine matrices. All the data are expressed as means of two replicates. Different letters refer to significant differences tested by ANOVA followed by multiple comparison Tukey HSD *post hoc* test ($P < 0.05$). Degrees of freedom, *F*- and *P*-values are reported in Supporting Information Table S2.

seem representative of different red wines, indeed all the parameters of the four matrices range within values that can be found in real wines. Particularly, the enriching strategy refers to what enologists do to obtain different types of red wines. Indeed, according to the phenolic content, the four matrices could be considered as four red wines styles characterized by low (B), medium ($B_1P_{0.5}$), high ($B_1P_{1.5}$) and very-high (B_1P_2) levels of dry extract. PWs, compared to free run wines, normally exhibit higher values of reducing sugars, pH, and phenolic compounds (anthocyanins and tannins), as well as lower alcohol content,[25] whereas the link to ethanol and glycerol seems questionable.[17,26,27]

This approach, together with the conditions applied to isolate wine VOCs that were designed for mimicking real tasting conditions, aims to contribute reducing the gap between results obtained in the laboratory and the real consumption setting, in studies investigating the matrix effect on wine aroma quality.

The compositional characteristics of the four experimental matrices represented the starting point to investigate the behavior and the sensory impact of volatiles in response to the different matrices and to study the correlations between specific compositional features and VOC release. The four matrices were characterized only concerning the main non-volatile components (residual sugars, total and free anthocyanins, tannins, and phenols), along with pH and ethanol. Other minor components, as for example organic acids,[28] amino acids,[29] and others, could have contributed to the observed results; however, they have not been considered in the present study.

To simulate as much as possible the real wine tasting conditions (i.e., wine/headspace *v/v*; temperature), volatiles were preconcentrated by SPME directly exposing the fiber to the static headspace of the wine in a INAO tasting glass. A total of 34 VOCs were identified in the headspace of the four wine matrices: ten esters, among which two acetates, nine alcohols, five acids, five terpenoids, one lactone, two volatile phenols, and two sulfur compounds (Tables 1 and S3).

Except for α-terpineol and nonanoic acid, the analyses revealed significant differences of VOC release from the four wine matrices. Most of the volatile compounds showed progressively increasing levels from B to B_1P_2, suggesting a global salting out effect (+37% of total detected VOCs), going from a matrix with low (B) to very high dry extract (B_1P_2).

Esters are the most impacted class, showing a constant rising total release from B to B_1P_2 (+54%) passing through $B_1P_{0.5}$ (+16%) and $B_1P_{1.5}$ (+21%). Some esters showed percentage variation higher than 100%: ethyl phenylacetate (340%), diethyl succinate (281%), ethyl butanoate (241%), and ethyl lactate (115%). The trend observed for ethyl butanoate (up to +241%) in response to the matrix is in line with previous findings on aroma–

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Table 1. Volatile organic compounds (VOCs) detected by headspace-solid phase micro-extraction-gas chromatography–mass spectrometry (HS-SPME-GC–MS) in the four wine matrices.

		Mean concentration							Odor	
Number	Chemical class and compound	M0 B	M1 $B_1P_{0.5}$	%Variation M1 *versus* M0	M2 $B_1P_{1.5}$	% Variation M2 *versus* M0	M3 B_1P_2	% Variation M3 *versus* M0	Type[†]	Descriptors[30,31†]
	Esters									
1	Ethyl lactate	162.22d	230.46c	42	255.25b	57	348.16a	115	Fruity	Sweet, fruity, acidic, etherial with a brown nuance
2	Diethyl succinate	92.86d	147.86c	59	248.90b	168	353.63a	281	Fruity	Fruity, apple, cooked apple, ylang
3	Isoamyl lactate	22.37d	28.58c	28	29.91b	34	38.85a	74	Fruity	Fruity, creamy, nutty
4	Ethyl butanoate	10.61c	31.52b	197	30.95b	192	36.18a	241	Fruity	Sweet, fruity, tutti frutti
5	Isoamyl acetate	63.75b	75.03ab	18	71.58ab	12	79.74a	25	Fruity	Sweet, fruity, banana, solvent
6	Ethyl phenylacetate	5.07d	8.96c	77	16.17b	219	22.30a	340	Floral	Sweet, Floral, Honey, Rose, Balsamic, Cocoa
7	*β*-Phenylethyl acetate	6.56b	6.91b	5	7.11b	8	10.33a	58	Floral	Sweet, honey, floral, rosy with a slight yeasty honey note with a cocoa and balsamic nuance
8	Ethyl hexanoate	413.35b	423.69b	3	429.48b	4	500.50a	21	Fruity	Sweet, fruity, pineapple, waxy, banana
9	Ethyl octanoate	1112.57d	1222.92b	10	1168.15c	5	1479.01a	33	Waxy	Waxy, sweet, musty, pineapple and fruity with a creamy, dairy nuance
10	Ethyl decanoate	144.13d	174.56c	21	197.88b	37	261.66a	82	Waxy	Sweet, waxy, fruity, apple
	Total esters	2033.49	2350.48	16	2455.38	21	3130.37	54		
	Alcohols									
11	2-Methyl-1-propanol (Isobutanol)	185.43c	201.28b	9	197.34bc	6	220.76a	19	Ethereal	Ethereal, winey
12	1-Butanol	2.39b	3.70a	55	3.52a	47	2.54b	6	Fermented	Fusel, oily, sweet, balsamic, whiskey
13	3-Methyl-1-butanol	4004.88c	4269.16b	7	4100.60bc	2	4554.83a	14	Fermented	Fusel, alcoholic, pungent, ethereal, cognac, fruity, banana, molasses
14	*β*-Phenylethanol	729.94d	944.42c	29	1231.48b	69	1686.32a	131	Floral	Floral, rose, dried rose, flower, rose water
15	(*E*)-3-Hexen-1-ol	1.16c	1.95a	69	1.51b	31	2.09a	81	Green	Green, cortex, privet, leafy, floral, petal, oily, earthy
16	1-Hexanol	152.51d	165.57b	9	160.39c	5	179.45a	18	Herbal	Pungent, ethereal, fusel, oily, fruity, alcoholic, sweet, green
17	1-Heptanol	16.00c	17.76b	11	16.80c	5	19.79a	24	Green	Musty, leafy, violet, herbal, green, sweet, woody, peony
18	2-Ethyl-1-hexanol	5.17b	6.87a	33	7.18a	39	7.64a	48	Citrus	Citrus, fresh, floral, oily, sweet
19	1-Octanol	10.78a	10.82a	0	10.04b	−7	11.22a	4	Waxy	Waxy, green, orange, aldehydic, rose, mushroom
	Total alcohols	5108.25	5621.54	10	5728.87	12	6684.65	31		
	Acids									
20	Acetic acid	101.71d	129.50c	27	148.82b	46	208.26a	105	Acidic	Sharp, pungent, sour, vinegar
21	Hexanoic acid	82.96ab	93.44a	13	76.75b	−7	89.07ab	7	Fatty	Sour, fatty, sweat, cheese
22	Octanoic acid	120.29a	119.70a	0	95.39b	−21	112.07ab	−7	Fatty	Fatty, waxy, rancid, oily, vegetable, cheesy
23	Nonanoic acid	20.45a	21.36a	4	25.87a	27	17.37a	−15	Waxy	Waxy, dirty, cheesy, dairy

Table 1. Continued

Number	Chemical class and compound	Mean concentration M0 B	M1 $B_1P_{0.5}$	%Variation M1 *versus* M0	M2 $B_1P_{1.5}$	% Variation M2 *versus* M0	M3 B_1P_2	% Variation M3 *versus* M0	Odor Type[†]	Descriptors[30,31†]
24	Decanoic acid	10.26a	10.16a	−1	7.66b	−25	9.50ab	−7	Fatty	Rancid, sour, fatty, citrus
	Total acids	335.67	374.16	11	354.50	6	436.26	30		
	Terpenoids									
25	Linalool	16.91c	19.95c	18	24.72b	46	28.31a	67	Floral	Citrus, orange, floral, terpenic, waxy, rose
26	α-Terpineol	5.55a	5.44a	−2	5.10a	−8	5.61a	1	Terpenic	Lilac, floral, terpenic
27	β-Citronellol	4.36ab	4.62ab	6	4.05b	−7	4.69a	8	Floral	Floral, rose, sweet, citrus, green, fatty, terpenic
28	Isobornyl acetate	40.66a	29.25b	−28	12.30c	−70	11.84c	−71	Balsamic	Balsamic, camphor, herbal, woody, sweet
29	Geranylacetone	12.54d	15.71c	25	20.03b	60	24.57a	96	Floral	Fresh, green, fruity, waxy, rose, woody, magnolia, tropical
	Total terpenoids	80.02	74.97	−6	66.19	−17	75.02	−6		
	Lactones									
30	γ-Butyrolactone	7.44d	15.38c	107	30.01b	304	40.57a	446	Creamy	Creamy, oily, fatty, caramel
	Total lactones	8.44	16.38	107	30.01	304	40.57	446		
	Phenols									
31	4-Ethylguaiacol	9.81c	12.74a	30	11.71b	19	10.09c	3	Spicy	Spicy, smoky, bacon, phenolic, clove
32	4-Ethylphenol	14.64a	14.40a	−2	9.57b	−35	6.91c	−53	Smoky	Phenolic, castoreum, smoke, guaiacol
	Total phenols	24.45	27.14	11	21.29	−13	17.00	−30		
	S-Compounds									
33	3-(Methylthio)-1-propanol (Methionol)	6.46d	9.28c	44	13.94b	116	19.37a	200	Meaty	Sulfurous, onion, sweet, soup, vegetable
34	Benzothiazole	10.05a	8.00b	−20	2.96c	−71	0.00d	−100	Sulfureous	Sulfury, rubbery, vegetable, cooked, nutty, coffee, meat
	Total S-compounds	16.51	17.28	5	16.90	2	19.37	17		
	Total detected VOCs	7606.82	8481.94	11	8673.14	14	10 403.23	37		

Note: Compound concentrations were reported semiquantitatively as internal standard equivalents in µg/L to compare relative concentration changes among the studied samples. Degrees of freedom, *F*- and *P*-values are reported in Supporting Information Table S3. In each row different letters refer to significant differences tested by ANOVA followed by multiple comparison Tukey HSD *post hoc* test ($P > 0.05$). B [1:0, 3690 mL bleed wine (BW)]; $B_1P_{0.5}$ [1:0.5, 3690 mL BW + dry extract from 1845 mL pressed wine (PW)]; $B_1P_{1.5}$ (1:1.5, 3690 mL BW + dry extract from 5535 mL PW); B_1P_2 (1:2, 3690 mL BW + dry extract from 7380 mL PW).
[†] From: http://www.thegoodscentscompany.com/.

polyphenol interactions, while this is not the case for ethyl decanoate that, as other hydrophobic esters, resulted in a lower variation at rising polyphenol content ranging from 230 to 2142 mg/L.[31] Here a wider range (from around 250 to more than 3500 mg/L) has been tested, that together with other compositional differences could at least partially explain the observed behavior of ethyl decanoate (up to +82%). Increased release of ethyl butanoate and decanoate was recently observed[31,32] in a pressed wine compared to red and white wines of different styles. The release of isoamyl acetate was barely impacted by changes in the matrix composition (up to +25%). Compared to other esters, it is considered the one able to break the aroma buffer of the wine, meaning that in a pool of 27 wine volatiles, its addition or subtraction significantly impacted the wine fruity perception differently from the other 26 volatiles.[33] According to this, it could be hypothesized a scarce sensory impact of the observed variations of the release of esters through the four wine matrices.

Total alcohols release rose up to 31% in the matrix very rich in dry extract (B_1P_2) with β-phenylethanol showing the most relevant increase reaching +131%. A salting-out effect was already observed for β-phenylethanol at raising tannins (1–10 g/L) and at low ethanol concentrations (10% *v/v*) in wine model solution.[34] Indeed, in these conditions of high tannins and low ethanol concentrations, more tannins self-aggregation could occur, leading to a decrease in the potential binding tannins sites for the odorants.[12,35] Among the four wine matrices, B_1P_2 represents the one that mostly tends to these conditions, even if the variation in ethanol percentage is narrow. Differently from β-phenylethanol, the other alcohols detected did not show a progressively increasing trend from B to B_1P_2, in accordance with data recently reviewed.[8]

Among acids, only acetic acid showed a clear trend: it constantly and significantly increased from B to B_1P_2. Differently from the other detected volatile acids, this compound is characterized by a very low hydrophobicity ($\log P_{o/w} = -0.170$) that could explain the observed salting out effect at increasing concentration of the dry extract in the matrix. This result suggests that within the legal range of volatile acidity, the contribution of acetic acid in the perception of acescence taint could be modulated by the red wine style referring to dry extract. Very little information is available in the literature about the matrix effect on the release of this class of volatiles. Therefore, it could be interesting to perform more studies focusing on acids response to different matrix compositions, since it has been reported that fatty acids and isoacids can positively contribute to wine fruitiness perception.[33]

Among terpenes, linalool, and geranyl acetone release constantly raised at increasing matrix concentration (up to +67 and + 96%, respectively), while isobornyl acetate showed the opposite trend (up to −71%). γ-Butyrolactone showed the highest significant increase from B to B_1P_2 (up to +446%), suggesting that in red wines, the perception of this volatile molecule can be favored at rising dry extract. This molecule is characterized by a low hydrophobicity ($\log P_{o/w} = -0.64$) that could be the reason why its release is importantly impacted when the concentration of the non-volatile wine components rise in the hydroalcoholic means. It has been already reported that VOCs with $\log P_{o/w} < 1$ independently from the wine matrix type, show a salting-out with possible sensory implications: sotolon ($\log P_{o/w} = -0.29$), furaneol ($\log P_{o/w} = -0.08$), and ethyl furaneol ($\log P_{o/w} = 0.43$), were indeed characterized by higher GC-O scores in the presence of a red wine non-volatile extract compared to a white one.[1,31] 4-Ethylguaiacol did not significantly vary when comparing B with B_1P_2, while 4-ethylphenol showed a significant decrease (up to −51%). This result partially confirms previous findings on aroma–polyphenol interactions[13] and is of oenological interest for the management of one of the main worldwide spread wine fault, namely the 'Brett character', for which 4-ethylphenol is a key molecule. A similar behavior was reported in a recent article,[32] where the release of 4-ethylphenol significantly decreased in pressed red wine compared to white wines. Opposite release trends from B to B_1P_2 were observed for two sulfur compounds: 3-(methylthio)-1-propanol (methionol) increased (up to +200%) while benzothiazole significantly decreased (up to −100%). This observation could also be of interest in the management of wine aroma quality, as benzothiazole could give rise to sulfur notes impacting wine quality.[36]

Globally, considering that the small dilution effect observed for ethanol at progressive addition of the deodorized dry extract ($B_1P_{0.5}$ *versus* B = 6,58%; dilution $B_1P_{1.5}$ *versus* B = 6,74%; dilution B_1P_2 *versus* B = 8,83%) likely occurred for the whole volatile fraction of the BW, the results on the release of VOCs could underestimate the global salting-out effect. However, the percentage dilutions fall in the range of standard deviations.

To test the global olfactory impact of the observed variations, a trained panel was asked to rank the four wine matrices according to the overall odor intensity. Results show that despite a positive trend to higher values, the differences between the matrices were not significant (Friedman; 3° of freedom; $P < 0.05$) suggesting a limited impact of the diverse matrix compositions on the overall odor intensity (Fig. 2).

However, significant variations of the relative intensity of specific olfactory notes have been detected (Fig. 3 and Table S4) meaning that, despite a constant VOC fraction of the four matrices, their olfactory perception was modulated by the diverse matrix composition affecting the release of VOCs. In Fig. 3 only the olfactory descriptors that showed significant differences (ANOVA, $P < 0.05$) among the four wine matrices, namely fruity, floral, dehydrated fruit, woody-toasty and vegetal-earthy, are represented. Fruity and floral notes decreased while the others significantly increased from B to B_1P_2. According to results, while

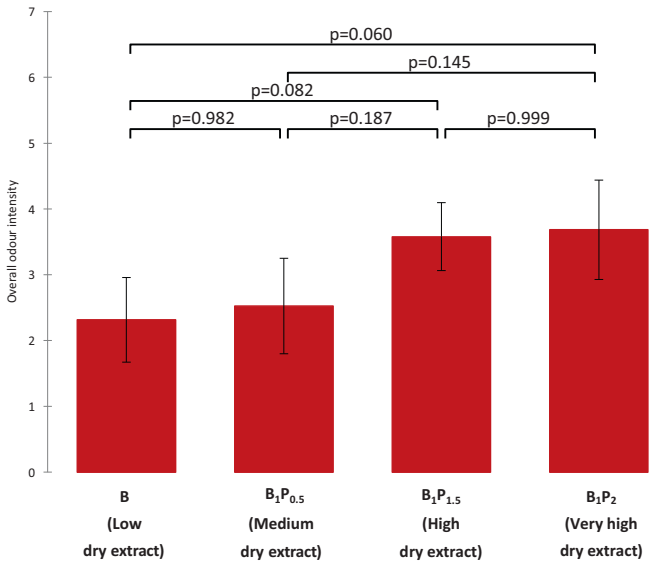

Figure 2. Ranking of the overall odor intensity in the four matrices. Differences were analyzed by a Friedman's test followed by a Nemenyi's multiple comparison ($P < 0.05$).

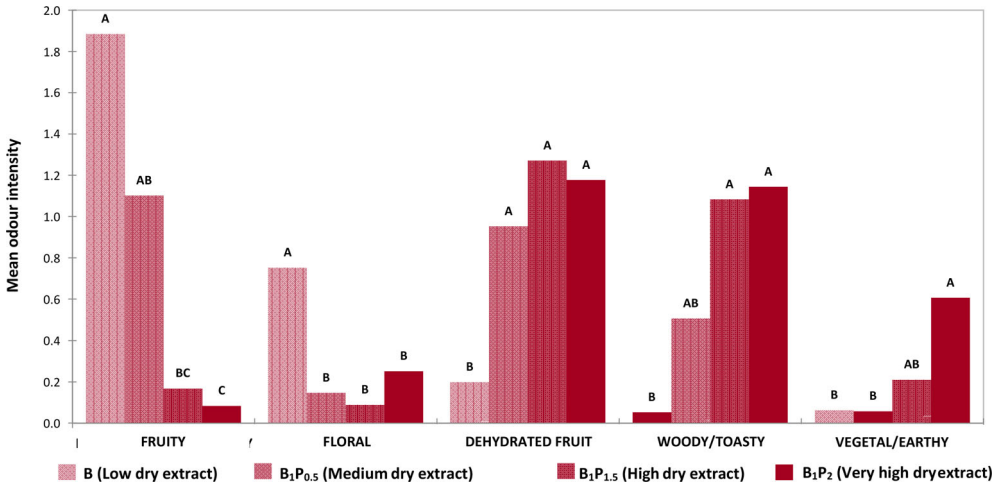

Figure 3. Wine matrices olfactory profile. Only descriptors with significant differences among the four wine matrices are shown. For each descriptor, different letters refer to significant differences tested by ANOVA followed by multiple comparison Tukey HSD *post hoc* test ($P < 0.05$). Degrees of freedom, *F*- and *P*-values are reported in Supporting Information Table S4.

the fruity, woody-toasty, and vegetal-earthy notes gradually varied from B to B_1P_2, the floral and dehydrated fruit notes significantly changed as soon as the BW (B) was enriched with PW deodorized dry extract (from B to $B_1P_{0.5}$).

To test the correlations among volatiles, matrix components and olfactory descriptors, a PCA (Pearson; $P < 0.05$) was performed with olfactory descriptors as supplementary data. Only VOCs and significant sensory variables (ANOVA, $P > 0.05$) were included in the computation. Furthermore, 86% of the total variance is represented with almost 76% on the first component (F1) and 10% on the second component (F2) (Fig. 4).

The four matrices are positioned in different areas of the biplot, with F1 essentially opposing the poorest matrix (B) to the richest one (B_1P_2), and F2 separating the $B_1P_{1.5}$ matrix to the others, with $B_1P_{0.5}$ occupying the middle of the chart. Most of the chemical variables, including VOCs and phenolic parameters, together with residual sugars and pH, show a strong projection onto the positive semiaxis of F1 and are well correlated with the matrix B_1P_2 highlighting the general salting-out effect on volatiles from the matrix enriched with the pressed fraction. In contrast, the BW matrix (B) correlates with ethanol and a few volatiles that cannot directly explain the correlation with fruity and floral sensory variables. Isobornyl acetate is reported as a balsamic terpene, while both 4-ethylphenol and benzothiazole are linked to phenolic and sulfureous wine taints, respectively (Table 1). This could suggest the involvement of perceptual interactions in the odor perception of the matrices. Indeed, molecules known to be involved in the fruity (isoamyl acetate and ethyl-butanoate, -hexanoate, -octanoate and -decanoate) and floral (linalool, phenylethyl acetate) wine aroma vectors[33] are well projected on the opposite area of the plot together with phenolics, residual sugars and pH as well as with the sensory descriptors of dehydrated fruit and woody/toasty. In this area the matrix with very high dry extract (B_1P_2) is well correlated with several volatiles among which the earlier-mentioned esters composing the fruity aroma vector, alcohols of the fusel alcohol aroma vector (e.g., isobutanol, isoamyl alcohol, β-phenylethanol, methionol) and those known to be characterized by a green flavor (1-hexanol,1-heptanol, 3-hexen-1-ol). Globally, based on the correlations shown in the PCA (Fig. 4) and on the variations detected in the sensory profiles (Fig. 3) it could be hypothesized that the loss in fruitiness at increasing dry extract could be linked to the higher release of fusel alcohols (e.g., isobutanol, isoamyl alcohol, β-phenylethanol, methionol) that can have a suppression power on that specific note, as previously demonstrated.[37-39] Further, despite the general increase in the release of esters, the minimal variation observed for isoamyl acetate, reported as a key ester impacting on fruitiness intensity by omission/addition tests[40,41] may also explain our evidence. Finally, the detected lower perception of fruitiness, as well as of floral notes and the higher perception of vegetal/earthy notes (Fig. 3), has been already highlighted at increasing polyphenolic concentrations and in the presence of different phenolic fractions.[9,11,14,42]

Several highly significant correlations were found ($r > 0.9$) (Table 2). An opposite behavior of ethanol compared to the other wine matrix components was found in relation to the release of VOCs. Most of the less hydrophobic aromas (i.e., β-phenylethanol, isoamyl lactate, diethyl succinate, methionol, ethyl lactate, acetic acid and γ-butyrolactone; $-0.64 \leq \text{Log}P_{o/w} \leq 1.36$) are negatively correlated to ethanol and positively to residual sugars and pH, suggesting that the alcohol content may act in opposition to sugars and acids on the release of these aromas. We previously found that both in orthonasal and in retronasal olfactory conditions, the release of volatiles with $0 < \log P_{o/w} < 2$ is the most affected by the wine matrix, particularly referring to residual sugars.[5] Moreover, the observed higher release of acetic acid and γ-butyrolactone at increasing dry extract could be a further element explaining the loss of fruitiness. Indeed, supplementing a fruity fraction with several compounds, among which also acetic acid, and γ-butyrolactone, Lytra *et al.*[43] showed that these volatiles, through perceptual interactions, had a significant attenuating effect on fresh-fruit aroma intensity. For the same less hydrophobic VOCs, some positive correlations with the diverse phenolic parameters were also found, except for free anthocyanins, which were the least correlated matrix parameter. This latter result could be related to the significant role of hydrophobicity of the matrix components on the release of these VOCs, as free anthocyanins are more hydrophilic than total anthocyanins and BSA reactive tannins, which consisted of more complex structures with higher hydrophobicity.[44] For the other VOCs, the

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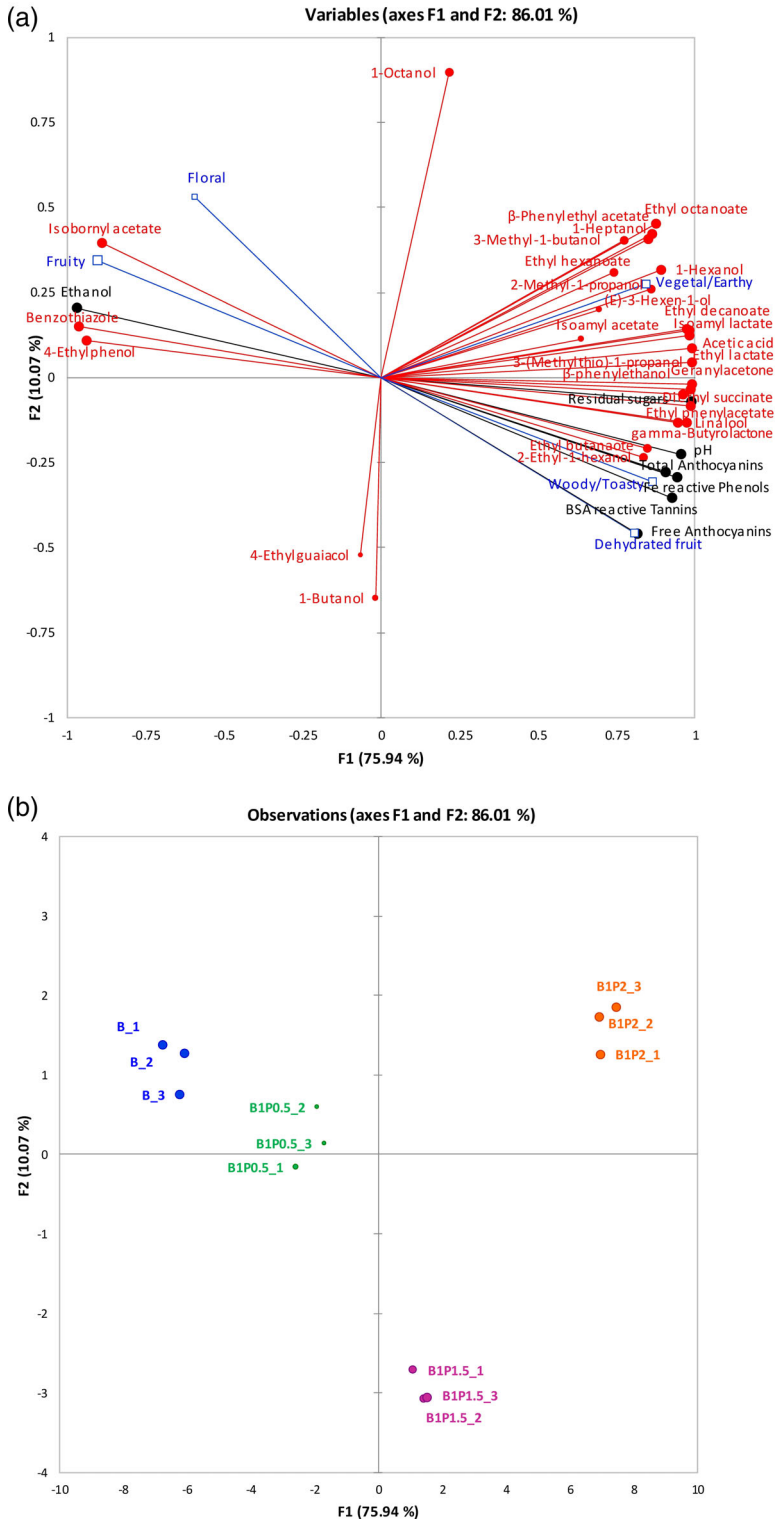

Figure 4. Principal component analysis (PCA) plots, (a) volatiles, matrix components, and olfactory descriptors (as supplementary data) – variables and (b) experimental wine matrices – observations (B, $B_1P_{0.5}$, $B_1P_{1.5}$, and B_1P_2). Only VOCs and sensory significant variables (ANOVA, $P > 0.05$) were included in the computation.

r coefficients seem not to indicate hydrophobicity as a driving factor for their release, suggesting that other molecular features, likely structural ones, can be impactful on these correlations. According to the literature,[45,46] this could be the case of the two benzenoids 4-ethylphenol and benzothiazole, for which the π-π stacking between the galloyl ring of the phenolic compounds with their aromatic rings could be responsible for the observed lower release in wines rich in dry extract and polyphenols (Table 1). Despite similar chemical characteristics of 4-ethylguaiacol compared to 4-ethylphenol (i.e., molecular

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Table 2. Correlation matrix coefficients (Pearson) between base compositional parameters, polyphenols, and volatile organic compounds (VOCs)

Log$P_{o/w}$	VOCs	Ethanol	pH	Residual sugars	Total anthocyanins	Free anthocyanins	Tannins (BSA reactive)	Phenols (iron reactive)
4861	Ethyl decanoate	**−0.930**	**0.927**	**0.972**	0.871	0.758	0.873	**0.901**
4129	Geranylacetone	**−0.939**	**0.942**	**0.959**	**0.918**	0.861	**0.923**	**0.937**
3842	Ethyl octanoate	−0.746	0.727	0.829	0.641	0.484	0.647	0.688
3600	Isobornyl acetate	**0.924**	**−0.943**	**−0.903**	**−0.915**	**−0.910**	**−0.962**	**−0.956**
3000	1-Octanol	−0.011	−0.007	0.142	−0.073	−0.223	−0.112	−0.061
2823	Ethyl hexanoate	−0.663	0.625	0.706	0.593	0.462	0.571	0.608
2820	2-Ethyl-1-hexanol	−0.825	0.810	0.818	0.725	0.714	0.833	0.826
2670	Linalool	**−0.952**	**0.946**	**0.958**	**0.915**	0.838	**0.925**	**0.940**
2620	1-Heptanol	−0.724	0.684	0.794	0.581	0.446	0.626	0.660
2580	4-Ethylphenol	**0.954**	**−0.972**	**−0.964**	**−0.970**	−0.896	**−0.931**	**−0.951**
2434	4-Ethylguaiacol	0.029	−0.061	−0.092	−0.144	−0.010	0.072	0.014
2260	*β*-Phenylethyl acetate	−0.782	0.754	0.847	0.719	0.572	0.672	0.716
2300	Ethyl phenylacetate	**−0.984**	**0.985**	**0.996**	**0.961**	0.890	**0.958**	**0.975**
2280	Isoamyl acetate	−0.611	0.530	0.615	0.459	0.377	0.537	0.548
2030	1-Hexanol	−0.765	0.749	0.839	0.633	0.493	0.690	0.720
2010	Benzothiazole	**0.981**	**−0.976**	**−0.979**	**−0.959**	−0.895	**−0.957**	**−0.971**
1804	Ethyl butanoate	−0.821	0.794	0.820	0.685	0.651	0.826	0.817
1612	(*E*)-3-Hexen-1-ol	−0.603	0.541	0.641	0.416	0.338	0.532	0.542
1360	*β*-Phenethylethanol	**−0.961**	**0.958**	**0.991**	**0.920**	0.823	**0.915**	**0.939**
1333	Isoamyl lactate	**−0.917**	**0.900**	**0.960**	0.826	0.713	0.857	0.881
1260	Diethyl succinate	**−0.976**	**0.977**	**0.995**	**0.950**	0.871	**0.943**	**0.963**
1020	3-Methyl-1-butanol	−0.615	0.634	0.716	0.504	0.361	0.553	0.587
0.880	1-Butanol	−0.034	0.050	−0.026	−0.032	0.106	0.174	0.114
0.760	2-Methyl-1-propanol (Isobutanol)	−0.732	0.757	0.816	0.647	0.524	0.693	0.720
0.417	3-(Methylthio)-1-propanol (Methionol)	**−0.977**	**0.971**	**0.995**	**0.939**	0.847	**0.934**	**0.955**
−0.039	Ethyl lactate	**−0.934**	**0.924**	**0.972**	0.851	0.742	0.883	**0.906**
−0.170	Acetic acid	**−0.921**	**0.903**	**0.959**	0.851	0.735	0.854	0.883
−0.640	*γ*-Butyrolactone	**−0.986**	**0.984**	**0.990**	**0.952**	0.870	**0.956**	**0.973**

Note: VOCs have been displayed from the highest to the lowest log$P_{o/w}$ value. Values in bold are different from 0 with a significance level $P = 0.05$ (in gray $P = 0.01$; in green positive correlations with $r > 0.9$; in red negative correlations with $r < 0.9$).

structure, log$P_{o/w}$), a clear release trend was not observed for the former compound (Table 1), and no correlations were detected with any of the compositional parameters. This could suggest an amount effect, but specific experiments are needed. This correlation study considered only some of the main compositional fractions of a red wine dry extract and therefore is lacking in considering the possible contribution of other non-volatile compounds that were not analyzed. The presented results, however, represent a starting point for future in-depth sensometabolomic studies.

CONCLUSIONS

In conclusion, the applied enriching strategy allowed to consider wine matrices with a wider range of phenolic concentration and a narrower range of ethanol, compared to similar previous studies. Except for free anthocyanins, the results showed significant correlations of the considered wine matrix components (ethanol, pH, residual sugars, total anthocyanins, tannins, and phenols) with the release of volatiles. The different matrices were able to modify the olfactory profile of the wine, even not impacting the perceived overall odor intensity. The fruity character seemed the most impacted by the red wine style likely because of both physical–chemical and perceptual interactions. A general negative correlation of ethanol with the release of VOCs was found, with results suggesting that it may occur already at around 1% *v/v* variation, acting in opposition to the other analyzed non-volatile matrix components on release of aromas. Negative correlations were found for 4-ethylphenol, and benzothiazole, two molecules responsible for important wine taints, that could become less perceivable in wines with high polyphenol content, but specific trials are necessary. On the contrary, the release of acetic acid constantly and significantly increased at higher dry extract concentrations suggesting that it could be more or less perceivable also depending on the wine style referring to dry extract. The latter result could be of particular interest in the management of wine off-odors during red winemaking. Despite the limited parameters of the matrix considered, the study

contributes to the understanding of wine matrix variables affecting its aroma quality under conditions closely mimicking the real ones. Specifically, the results could be useful in winemaking to guide pressing and blending, a practice recently proposed to enhance the sensory properties and consumer acceptance of warm climate red wines,[47] in an optic of aroma quality management. The results could also be considered for questioning the possible contribution of an increasing dry extract on the loss of fruitiness/freshness and appearance of cooked/dehydrated fruit notes in red wines as effects of raising air temperature and radiation, and vine water deficit[48] linked to the current climatic change.

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DATA AVAILABILITY STATEMENT

Data available on request from the authors.

SUPPORTING INFORMATION

Supporting information may be found in the online version of this article.

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