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Comparative study of aromatic compounds in two young white wines subjected to pre-fermentative cryomaceration

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Abstract

An aroma profile, based on the contents of 36 specific compounds, grouped in seven series that contribute to wine odour was developed, allowing wines to be classified according to their sensory characteristics. The proposed profile allows wines of different varieties to be distinguished, identifying the effects of pre-fermentative treatments on the flavour of the resulting wine. The profile revealed significant differences in the solvent, floral, sweet, green and balsamic series of compounds between wines of the Airen and Macabeo grape varieties. Pre-fermentative cryomaceration significantly increases the solvent, floral, fruity and balsamic series in the Airen variety, whereas the solvent series is only affected in the Macabeo variety.

Keywords: Flavour; Odorant series; Odorant activity; Wine; Airen; Macabeo

1. Introduction

The aroma of wine depends on its contents of volatile compounds, more than 680 of which have to date been identified in wines from some white grape varieties (Maarse & Visscher, 1994). However, not all such compounds, contribute to the same extent to wine aroma; in fact, the contribution of a specific compound is related to its odour perception threshold, which is defined as the lowest concentration that can be detected by smelling. The use of powerful techniques, such as GC-MS, allows aroma compounds to be readily identified and quantified; this, together with the knowledge of their respective perception thresholds, has allowed available information in this respect to be systematized. In fact, the concentration/threshold ratio known as the "odour activity value" (OAV) allows one to estimate the contribution of a specific compound to the aroma of wine, yielding a complex aroma profile with all the components quantified in the wine.

The aroma perceived by smelling is a result of the combined contributions of various compounds and can rarely be attached to a specific, individual component. OAVs have been adopted to analyze these interactions and to reveal so-called critical compounds essential to total aroma (Nykänen & Soumalainen, 1983). According to Guth (1997) this concept is therefore necessary to quantify the levels of recognized odorant compounds and for the exact establishment of flavour differences between wines obtained from different grape varieties or origins.

As a rule, the odour of a compound is described in terms of several descriptors agreed upon by experts (Etievant, 1991; Ferreira, Aznar, & Cacho 2001; Guth, 1997; Kotseridis & Baumes, 2000; López, Ferreira, Hernández, & Cacho, 1999). On the other hand, several authors have utilized aroma series to describe the flavour of a wine (Brugirard, Fanet, Seguin, & Torres, 1991; Torres, 1987). Grouping the aroma compounds with similar descriptors into aroma series, gives an organoleptic profile of the wine. This in turn gives the contribution of a specific compound to each series. This procedure allows one to relate quantitative information derived by chemical analysis to sensory

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perceptions, with a view to obtaining an aroma profile for the wine that is more simple and based on more objective criteria than existing alternatives; among other applications, it has recently been used to interpret data obtained in biological aging tests (Moyano, Zea, Moreno, & Medina, 2002).

Pre-fermentative skin cryomaceration is normally used by the winemaker to enhance the varietal character of white wines. This procedure provides acceptable, well-balanced, better-rounded wines, with a stronger body in the mouth; this strengthening effect, however, occasionally alters the wine typicity and introduces heavy, coarse aromas (Delteil, Feuillat, Guilloux-Benatier, & Sapis, 2000).

In this work, we establish one analytical aroma profile with odorant series in young white wines from Airen and Macabeo varieties and the effect of pre-fermentative cryomaceration in such series. The wines are also evaluated and compared by a panel of tasters and their preferences are related to the analytical data.

2. Material and methods

Grapes of the Airen (100 kg) and Macabeo (100 kg) varieties were harvested at an industrial ripeness stage corresponding to wines containing 11% ethanol. Three samples of each variety were processed separately to obtain two batches of must, one by direct pressing of the grapes and the other by pressing the mass resulting from grapes subjected to skin maceration immediately after crushing at 10 °C for 24 h. Potassium metabisulphite (120 mg/kg grape) was added to the grapes prior to pressing. All the musts obtained after pressing were adjusted to pH 3.4, centrifuged at 3000 rpm for 10 min and inoculated with 2% (v/v) of a pure culture of *Saccharomyces cerevisiae* (cerevisiae A) yeast strain (Kurtzman and Fell, 1998) obtained from the collection of the Microbiology Department of the University of Cordoba. Fermentations were conducted by using 10 l glass bottles that were kept at 22 °C throughout. All macerations and fermentations were performed in triplicate. In this way, three samples of control wines and three of macerated wines of each variety were obtained.

Ethanol was quantified using the method of Crowell and Ough (1979), and reducing sugars, phenolic compounds, volatile acidity, pH, and tartaric, malic and citric acids, were determined using EEC (1990) official methods.

Aroma compounds were quantified on a Hewlett-Packard 5890 Series II gas chromatograph equipped with a split/splitless injector, a flame ionization detector and an SP 1000, 60 m×0.32 mm i.d. capillary column from Supelco, Inc. (Bellefonte, PA. USA). Aroma compounds were collected by using 100 ml of sample and 100 ml of Freon-11 in a continuous extractor for 24 h. The organic extract was concentrated to 0.2 ml in a Kuderna-Danish micro-concentrator and 3 μl were injected into the injector, with a split ratio of 60:1. Each compound was quantified from its response factor, which was obtained by using three standard solutions of known concentration subjected to the same treatment as the samples; 2-octanol at a 481 μg/l concentration was used as internal standard for all samples. The compounds were identified in previous laboratory works by their retention times and confirmed by GC-MS on a Hewlett-Packard 6890 chromatograph equipped with an HP 5972 ion-selective detector (Agilent Technologies. Palo Alto CA. USA). The Wiley mass-spectrum library and pure compounds from Merck, Sigma-Aldrich and Fluka were used for confirmation and preparation of standard solutions of aroma compounds that were quantified.

A single factor analysis of variance (ANOVA) was done using the Statgraphics® Plus v. 2 statistical software package (STCS, Inc., Rockville, MD. USA).

3. Results and discussion

The ethanol contents in all wine were 11% ± 0.2 (v/v) and the residual sugars were less than 5 g/l. Titratable acidity in all types of wine were 6.2 ± 0.8 g/l, expressed as tartaric acid, and volatile acidity was 0.3 ± 0.1 g/l expressed as acetic acid. pH values were 3.1 ± 0.1. These values are characteristic of young white table wines and revealed a good vinification process.

Table 1 shows the volatile aroma compounds quantified and their qualifier ions selected for identification according the Wiley mass-spectrum library, standard compound used and specialized literature on mass-spectrum wine flavour's (Atienza, Esteve, Aragón, & Climent, 1999; Vernin, Boniface, Metzger, Fraisse, Doan, & Alamercery, 1987). This table shows also the perception thresholds and descriptors for each aroma compound studied as previously reported (Brugirard et al., 1991; Ferreira et al., 2001; Guth, 1997; Kotseridis and Baumes, 2000; López et al., 1999). Each compound was assigned to one or several aroma series, depending on its principal odour descriptors; the solvent, floral, sweet, green (vegetal or herbaceous), fatty, fruity and balsamic series were chosen for this purpose on account of their extensive use for describing and distinguishing young table white wines in terms of aroma by specialized journals and tasters (Mijares, 1987; Peris & Masats, 2000; Peynaud, 1987)

The compounds with the highest perception thresholds were 1-propanol, 2-phenylethanol, 1-butanol, ethyl lactate, acetoin, isobutanol, isoamyl alcohols, 2-butanol, γ-butyrolactone, ethyl acetate and octanoic acid. In fact, all have perception thresholds equal to or higher

Table 1
Aroma compounds quantified in wines. Qualifier ions (m/z), perception threshold (in mg l^{-1}), aroma descriptors and assignation of compounds to different odorant series

Aroma compound	m/z	Threshold[a]	Odor description	Odorant series[b]
1-Propanol	59, 42, 60, 39	306	Alcohol, ripe fruit	1,7
Isobutanol	41, 43, 39, 42	75	Alcohol, nail polish	1
Isoamyl alcohols	55, 70, 57, 71	60	Solvent	1
Phenethyl alcohol	91, 92, 122, 65	200	Rose, honey	2,3
1-Butanol	56, 41, 43, 39	150	Medicinal	7
2-Butanol	45, 59, 43, 41	50	Medicinal, wine-like	1
1-Hexanol	56, 43, 69, 84	1.1	Herbaceous, grass, woody	4
Z-3-Hexenol	67,41, 39, 82	1	Green, bitter, fatty	4,5
Propyl acetate	43, 61, 73, 87	4.7	Celery	4
Butyl acetate	43, 56, 41, 73	1.8	Fruity	6
Isobutyl acetate	43, 56, 41, 73	1.6	Sweet, fruity, apple, banana	3,6
Isoamyl acetate	43, 70, 55, 41	0.16	Banana, fruity, sweet	3,6
Hexyl acetate	56, 43, 41, 55	0.67	Apple, cherry, pear, floral	2,6
Phenethyl acetate	104, 43, 91, 105	1.8	Flowery	2
Ethyl acetate	43, 45, 42	12	Pineapple, fruity, solvent, balsamic	1,6,7
Ethyl hexanoate	88, 43, 99, 41	0.08	Fruity, green apple, banana, brandy, wine-like	6
Ethyl octanoate	88, 55, 41, 101	0.58	Sweet, floral, fruity, banana, pear, brandy	2,3,5
Ethyl decanoate	88, 101, 41, 43	0.5	Brandy, fruity, grape	6
Ethyl propanoate	57, 74, 75, 102	1.8	Banana, apple	6
Ethyl butanoate	71, 43, 88, 41	0.4	Strawberry, apple, banana	6
Diethyl succinate	101, 129, 55, 73	1.2	Fruity, melon	6
Ethyl lactate	45, 75, 56	150	Fruity, buttery	5,6
Butyl lactate	45, 41, 57, 43	1	Creamy, milky, sweety	3
Hexanoic acid	60, 73, 41, 45	3	Cheese, fatty	5
Octanoic acid	60, 73, 41, 55	10	Fatty, rancid	5
Decanoic acid	60, 73, 41, 55	6	Fatty, rancid	5
Linalool	93, 41, 91, 69	0.015	Citrus, floral, sweet, grape-like	2,3,6
α-Terpineol	93, 68, 67, 59	1	Lilac, floral, sweet	2,3
β-Ionone	43, 91, 135, 177	0.005	Balsamic, rose, violet	2,7
E-Nerolidol	69,41, 93, 91	1	Rose, apple, green, citrus	2,4,6
Z-Nerolidol	69,41, 93, 91	1	Rose, apple, green, citrus	2,4,6
Farnesol	93, 41, 69, 91	1	Floral, oily	2,4
γ-Butyrolactone	42, 56, 86	20	Caramel, sweet	3
Benzaldehyde	106, 77, 105, 51	2	Almond, fragant	2
Acetoin	45, 42, 88, 73	150	Buttery, cream	5
1,1-Diethoxyethane	45, 73, 47, 43	1	Tart, fruity, balsamic	6,7

[a] Determined in 10% (v/v) ethanol solution adjusted to pH 3.5 with tartaric acid.
[b] 1, solvent, 2, floral, 3, sweet, 4, green, 5, fatty, 6, fruity, 7, balsamic.

than 10 mg/l and contributed jointly to the solvent, floral, sweet, fatty, fruity and balsamic series. On the other hand, the compounds with the lowest perception thresholds ($\leqslant 0.5$ mg/l) were β-ionone, linalool, ethyl hexanoate, isoamyl acetate, ethyl butanoate and ethyl decanoate; all except β-ionone were included at least in the fruity aroma series.

Table 2 shows the odour activity value (OAV) for each compound. As can be seen, only isobutanol, isoamyl alcohols, isoamyl acetate, ethyl acetate, ethyl hexanoate, linalool and 1,1-diethoxyethane had OAVs exceeding unity in all types of wine. The OAV for ethyl octanoate was greater than unity in all wines of the Airen variety, whereas that for diethyl succinate only exceeded unity in the control wines of both varieties. Finally, the OAV for hexanoic acid exceeded unity in Airen macerated wines, and β-ionone in all samples except those of Airen control wine.

By combining the OAV for each individual compound in an aroma series, the global OAV for each series (Fig. 1) was obtained. The results were subjected to a single-factor analysis of variance (Table 3) in order to identify series exhibiting significant differences between varieties and those affected by the pre-fermentative skin cryomaceration within each variety.

The control wines of the Airen variety showed the highest contribution of the fruity series (followed by the balsamic, sweet, solvent, floral, fatty and green series) to the global aroma. This sequence was also observed in the Airen macerated wines, with the sole exception of the sweet series, which was the second most marked contributor to the wine aroma, followed by the balsamic series (Fig. 1).

The effect of maceration is an increase of the solvent ($P \leqslant 0.05$), floral ($P \leqslant 0.001$) and sweet series ($P \leqslant 0.01$), and a decrease of the fruity ($P \leqslant 0.05$)

Table 2
Odour activity values (OAVs) for the aroma compounds in wines from Airen and Macabeo grapes

Aroma compound	Airen control	Airen macerated	Macabeo control	Macabeo macerated
1-Propanol	0.012±0.005	0.032±0.006	0.030±0.003	0.052±0.006
Isobutanol	1.7±0.4	3.7±0.8	3.0±0.4	9±1
Isoamyl alcohols	4.5±0.7	6.9±0.4	8.2±0.7	12.1±0.4
Phenethyl alcohol	0.22±0.03	0.34±0.02	0.6±0.1	0.8±0.1
1-Butanol	0.0033±0.0008	0.0054±0.0008	0.0089±0.0004	0.0097±0.0005
2-Butanol	0.043±0.004	0.080±0.005	0.075±0.006	0.067±0.004
1-Hexanol	0.29±0.06	0.43±0.04	0.74±0.06	0.9±0.1
Z-3-Hexenol	0.34±0.06	0.110±0.004	0.09±0.01	0.08±0.01
Propyl acetate	0.0082±0.0009	0.018±0.003	0.0069±0.0006	0.004±0.001
Butyl acetate	0.12±0.01	0.35±0.04	0.0±0.0	0.0±0.0
Isobutyl acetate	0.10±0.01	0.14±0.02	0.12±0.01	0.15±0.02
Isoamyl acetate	17±2	26±2	20±2	19±1
Hexyl acetate	0.11±0.01	0.062±0.003	0.26±0.05	0.0±0.0
Phenethyl acetate	0.31±0.03	0.62±0.07	0.53±0.10	0.52±0.09
Ethyl acetate	2.8±0.5	3.1±0.4	5.7±0.6	5.2±0.5
Ethyl hexanoate	11±1	7±1	11.5±0.5	3.9±0.8
Ethyl octanoate	1.1±0.1	1.7±0.2	0.9±0.1	0.5±0.1
Ethyl decanoate	0.11±0.01	0.11±0.01	0.05±0.01	0.0±0.0
Ethyl propanoate	0.061±0.008	0.060±0.005	0.16±0.01	0.13±0.02
Ethyl butanoate	0.42±0.04	0.59±0.07	0.57±0.05	0.49±0.06
Diethyl succinate	1.4±0.1	0.6±0.2	4.0±0.3	1.5±0.2
Ethyl lactate	0.039±0.008	0.077±0.009	0.14±0.02	0.23±0.03
Butyl lactate	0.25±0.04	0.19±0.01	0.51±0.02	0.60±0.07
Hexanoic acid	0.8±0.1	1.4±0.2	0.8±0.1	0.7±0.1
Octanoic acid	0.072±0.005	0.34±0.03	0.07±0.01	0.09±0.01
Decanoic acid	0.024±0.004	0.063±0.005	0.052±0.004	0.061±0.004
Linalool	1.4±0.3	3.21±0.04	4±1	9±1
α-Terpineol	0.080±0.008	0.30±0.07	0.12±0.01	0.038±0.007
β-Ionone	0.0±0.0	5.0±0.8	16±2	18±2
E-Nerolidol	0.035±0.009	0.048±0.004	0.042±0.004	0.081±0.006
Z-Nerolidol	0.07±0.01	0.14±0.02	0.24±0.05	0.29±0.05
Farnesol	0.028±0.002	0.0±0.0	0.079±0.008	0.0±0.0
γ-Butyrolactone	0.06±0.01	0.19±0.02	0.14±0.02	0.26±0.01
Benzaldehyde	0.0±0.0	0.0±0.0	0.0069±0.0006	0.014±0.002
Acetoin	0.10±0.02	0.012±0.001	0.055±0.004	0.025±0.003
1.1-Diethoxyethane	39±5	9±1	36±5	30±2

Table 3
ANOVA of the aroma series in the control wines and those obtained by prefermentative cryomaceration of grapes of the Airen and Macabeo varieties[a]

Series	Maceration effect		Variety effect	
	AC-AM	MC-MM	AC-MC	AM-MM
Solvent	*	**	**	***
Floral	***	NS	***	***
Sweet	**	NS	*	NS
Green	NS	NS	*	**
Fatty	NS	NS	NS	**
Fruity	*	NS	NS	**
Balsamic	**	NS	*	***

[a] AC, Airen control; AM, Airen macerated; MC, Macabeo control; MM, Macabeo macerated; NS, No significative interaction. *$\leqslant$0.05, **$\leqslant$0.01, ***$\leqslant$0.001.

and balsamic series ($P\leqslant0.01$), in the aroma of the Airen wines (see Fig. 1 and Table 3). The decrease in the contributions of the fruity and balsamic series are due mainly to the decrease in the content of 1,1-diethoxyethane ($P\leqslant0.001$), which contributed, with an OAV higher than unity, to both series. Likewise, the increased values for the other aroma series can be ascribed to the substantially increased contents of compounds with OAV$\geqslant$1 in each series (Table 4); in contrast, diethyl succinate had its OAV decreased by maceration (Table 2).

Control and macerated wines of the Macabeo variety had the same sequence in the aroma series; in fact, the greatest aroma contribution was that of the fruity series, followed by the balsamic, sweet, floral, solvent, fatty and green series (Fig. 1).

The pre-fermentative cryomaceration for production of Macabeo wines had significant effects ($P\leqslant0.01$) only on the OAV for the solvent series (Table 3), which increased through an increase in the OAVs for isoamyl alcohols ($P\leqslant0.01$) and isobutanol ($P\leqslant0.001$) (Table 4). Although maceration significantly decreased the OAVs of diethyl succinate, ethyl hexanoate (both with $P\leqslant0.001$) and ethyl octanoate ($P\leqslant0.01$), and increased those for linalool ($P\leqslant0.01$), the differences did not

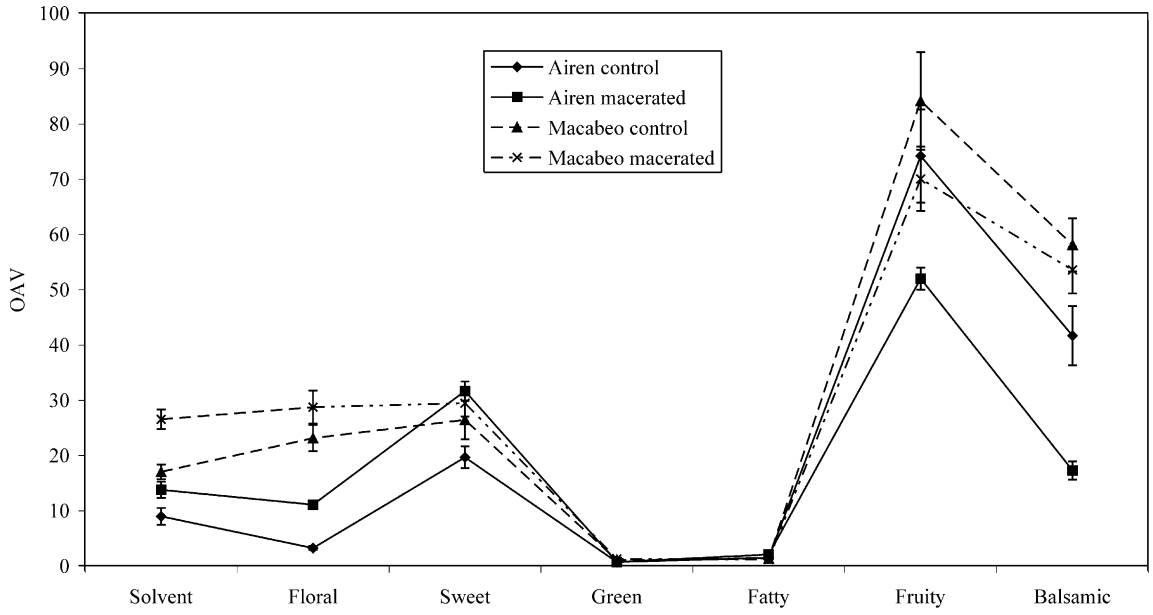

Fig. 1. Odour activity values of odorant series macerated and unmacerated young white wines of Airen and Macabeo varieties.

reflect their respective aroma series as they were offset by the other components of the series.

The Airen and Macabeo control wines exhibited significant differences ($P \leqslant 0.05$) in the solvent, floral, sweet, green and balsamic series (Table 3); the Macabeo variety had the greater values because the compounds with OAVs above unity were present in higher concentrations in this variety -by exception, ethyl octanoate was present in greater amounts in the Airen variety. The fatty and fruity series exhibited no significant differences as their main components (viz. hexanoic acid for the fatty series, and 1,1,-diethoxyethane, isoamyl acetate and ethyl hexanoate for the fruity series) exhibited none either (Table 4).

The differences between the Airen and Macabeo wines were strengthened by pre-fermentative skin maceration, which resulted in significant differences at the $P \leqslant 0.01$ level in the solvent, floral, green, fatty, fruity and balsamic series (Table 3). All aroma series, except the fatty one, exhibited higher OAVs in macerated Macabeo wine than in macerated Airen wine.

Finally, in order to establish a first approach between chemical and sensory evaluation in quality control situations, all the wines were subjected to sensorial analysis by an expert panel of tasters. Macerated and unmacerated wines of each variety were compared, as well as the best wine from each variety selected by the tasters.

Unmacerated Macabeo wine was preferred to its macerated counterpart which can be attributed to the smaller contribution of the solvent series in the former. On the other hand, the tasters preferred cryomacerated Airén wine to unmacerated wine of the same variety, as a likely result of the significantly increased OAVs for the floral and sweet series, and the decreased OAVs for the balsamic series. Finally, the tasters preferred unmacerated Macabeo wine to macerated Airen wine, which can ascribed to the increased OAVs for the floral and fruity series which substantially improved sensory characteristics of this young white table wine.

Table 4
Aroma compounds with OAV $\geqslant 1$ that allow distinction between Airen and Macabeo varieties and show significant differences because the prefermentative cyomaceration[a]

Compounds	Series[b]	Maceration effect		Variety effect	
		AC-AM	MC-MM	AC-MC	AM-MM
Isobutanol	1	*	***	*	**
Isoamyl alcohols	1	**	**	**	***
Isoamyl acetate	3,6	**	NS	NS	**
Ethyl acetate	1,6,7	NS	NS	**	**
Ethyl hexanoate	6	**	***	NS	*
Ethyl octanoate	2,3,5	*	**	*	**
Diethyl succinate	6	**	***	***	**
Hexanoic acid	5	*	NS	NS	**
Linalool	2,3,6	**	**	*	**
β-ionone	2,7	***	NS	***	***
1,1-Diethoxyethane	6,7	***	NS	NS	***

[a] AC, Airen control; AM, Airen macerated; MC, Macabeo control; MM, Macabeo macerated; NS, No significant interaction.

[b] 1, solvent, 2, floral, 3, sweet, 4, green, 5, fatty, 6, fruity, 7, balsamic. * $\leqslant 0.05$; ** $\leqslant 0.01$; *** $\leqslant 0.001$.

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