



The aroma of La Mancha Chelva wines: Chemical and sensory characterization



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ABSTRACT

The aim of this study is the chemical and sensory description of the aroma of wines white Chelva through five successive vintages. Solid Phase Extraction (SPE) was used for the isolation of volatile compounds and Gas Chromatography Mass Spectrometry (GC-MS) was employed by their analysis. Aroma Sensory Profile of wines was defined using the Quantitative Descriptive Sensory Analysis (QDA). A total of 77 volatile compounds were identified in Chelva wines. Chelva wines present a great quantity of aromatic compounds of very different chemical nature and with different aromatic descriptors, which will increase its aromatic complexity of these wines. Aroma sensory profile of Chelva wines was defined by higher intensity of fresh and peach aromas with notes citric, green apple, tropical fruit and sweet. This study showed that the Chelva grapes variety cultivated in La Mancha region present a great aroma potential and a complex sensory profile and can be considered a viable alternative to traditional grape varieties cultivated in this region for increasing the offer to the consumer.

1. Introduction

Aroma and flavour are some of the most important sensory characteristic of wine that define the quality (Cadot, Caillé, Samson, Barbeau, & Cheynier, 2010; Maitre, Symoneaux, Jourjon, & Mehinagic, 2010). They are influenced by a large number of volatile compounds, around 1000, of very different chemical classes and character (alcohols, esters, aldehydes, ketons, fatty acids, terpene compounds.) and in a wide range of concentrations (ng/L to mg/L) (Flanzy, 2003).

Moreover, the perception threshold of these compounds can vary from ng/L to mg/L, and, in several cases, they do not show an evident relationship between concentration and sensory impact. Sensory impact is usually related to complex interactions among volatile compounds (Chambers IV & Koppel, 2013). This complexity explains why small differences in the concentration of aromatic compounds can result in an excellent or ordinary wine (Swiegers, Bartowsky, Henschke & Pretorius, 2005). Odour Activity Value (OAV) is a parameter, frequently used to evaluate the contribution of each volatile compound to wine aroma, and can be considered as a useful measure to assess the relative importance of individual chemical components present in a sample.

Nevertheless the aroma characterization of wine generally involves the use of analytical techniques and descriptive sensory analysis (Muñoz-González, Rodríguez-Bencomo, Moreno-Arribas, & Pozo-Bayón, 2011). Analytical methods use techniques of isolation,

separation, identification and quantification of volatile aroma compounds and use the gas chromatography (such as GC/MS). Therefore, a complete chemical profile of the volatile fraction of the wine is obtained. Sensory analysis is based on an expert panel taster, who defines a sensory profile of wine. Tasters use sensory descriptors of perceived wine attributes according to the definition of a common method and consensual flavours lexicons (Drake & Civille, 2003; Esti, González Airola, Moneta, Paperai, & Sinesio, 2010). For these reasons, in order to adequately evaluate the aroma profile of wine and understand which compounds are responsible for the characteristics notes is necessary to correlate volatile composition and sensory data (Francis & Newton, 2005; Green, Parr, Breitmeyer, Valentin, & Sherlock, 2011; Noble & Ebeler, 2002; Vilanova, Escudero, Graña, & Cacho, 2013).

Vitis vinifera L. var. Chelva a minority grape variety is cultivated in La Mancha region in little zone with singular climatologic circumstances (warm summers, cold winters and low rain) that could affect its volatile compounds (Bureau, Razungles & Baumes, 2000). The understanding of the volatile and sensory aroma characteristics of these wines can give possibility for the adaptation of this grape variety to the winemaking sector governed by the consumer's demand. The goals of this research were to characterize the volatile compounds, impact compounds and sensory aroma profile of La Mancha Chelva wines (Spain) over five consecutive vintages.

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2. Material and methods

2.1. Winemaking process

Vitis vinifera cv. Chelva grapes cultivated in La Mancha (central-southeastern region of Spain) were manually harvested at their optimum point of maturity and in good sanitary stage, across five successive harvests (2013–2017).

The wines were elaborated using about 25 kg of grapes and according to the conventional white winemaking method with slight contact of must with solid parts of grapes. After stemming and crushing the grapes, the must obtained by pressing, was added to 100 mg/L of SO₂ as K₂S₂O₇. In order to make the clarification process by natural settling, the must was kept in repose during 24 h at 10 ± 1 °C. Then, the must was transferred to 10 L fermentation vessels. The inoculum used was *Saccharomyces cerevisiae cerevisiae* strain (CECT n° 10835). The alcoholic fermentation (AF) was conducted at 18 °C, controlled by measures of the density and terminated after 10 days as confirmed by the relative density reached a constant value. Then the wines were raked, passed through 0.45 µm filters (Millipore, Bedford, MA, USA), bottled, and stored in controlled conditions at 8–10 °C. All fermentation were carried out in duplicate and the wines were elaborated and analyzed in the year of vintage.

2.2. General analysis of musts and wines

The methods proposed by O.I.V. International Oenological Codex, 2010 were used to determine total acidity, °Brix and pH in musts, and total and volatile acidity, alcoholic strength (% v/v), pH, and total and free SO₂ in Chelva wines. General analysis of wines was carried out 30 days after bottling.

2.3. Analysis of major volatiles compounds

The method proposed by Sánchez-Palomo, Pérez-Coello, Díaz-Maroto, González-Viñas, & Cabezudo, in 2006 was used to determine the major volatile compounds of wines by direct injection into HP-5890 GC equipped with a FID detector using a CP-Wax-57 column (50 m × 0.25 mm × 0.25 µm). Operating conditions were: oven temperature program: 40 °C (5 min) – 4 °C/min – 120 °C, carrier gas He at 0.7 mL/min and injector and detector temperatures were 250 °C and 280 °C respectively.

2.4. Analysis of minor volatiles compounds

Gas chromatography coupled to mass spectrometry (GC–MS) was used to research minor volatile compounds of La Mancha Chelva wines. Extraction of volatile compounds was carried out using the method described by Sánchez-Palomo et al., 2006 using styrene-divinylbenzene cartridges (LiChrolut EN, Merck, 0.5 g of phase) One hundred millilitres of wine were eluted through the LiChrolut EN cartridges (1 mL/min). In order to eliminate sugars and other low-molecular-weight polar compounds, the cartridge was washed with 50 mL of milli-Q water. Ten millilitres (10 mL) of dichloromethane was used to elute minor volatile compounds and then the organic extract was concentrated under nitrogen stream to a 200 µL.

The chromatographic separation of volatile compounds of organic extract was carried out into an Agilent Gas Chromatograph model 6890 N using a BP-21, capillary column (60 m × 0.25 mm × 0.25 µm) using the following oven temperature program: 70 °C (5 min) – 1 °C/min – 95 °C (10 min) – 2 °C/min – 200 °C (40 min). One microlitre (1 µL) was injected in splitless mode using helium at 1 mL/min as carrier gas. Detection of different minor aroma volatile compounds was carried out by mass spectrometry, using a Mass Selective Detector model 5973 inert (Agilent) by electron impact (EI) mode at 70 eV. Transfer line and injector temperatures were 280 °C and 250 °C, respectively. Source

temperature was 178 °C, the scanning rate was 1 scan/s and the mass acquisition were 40–450 amu.

The identification of volatile compounds was based on comparison of the GC retention index and mass spectra with authentic standards when standard were obtainable, for these compounds calibration curves were calculated with the purpose of quantification. When the authentic standards were not existing, the identification was established on comparison with the spectral data of the Wiley A library and with the chromatographic data from the literature; semi-quantitative analyses of these compounds were done, assuming response factors equal to one.

2.5. Analysis of odour activity values (OAV)

In order to estimate the influence of the aroma compounds to the aroma of wine the odour activity value (OAV) was determined. The OAV was established as the ratio between the concentration and the odour threshold of the individual aroma compounds found in bibliography (Francis & Newton, 2005).

2.6. Sensory evaluation

The judges were primarily selected based on their ability to identify the basic tastes in standard solutions containing somewhat above-threshold concentrations of pure sucrose, citric acid, caffeine, sodium chloride and tartaric acid (Meilgaard, Civille & Carr, 1999), their capacity to determine variations in the flavour of white wine samples in the difference-from-control test (Meilgaard et al. 1999); and their aptitude to recognise 12 odours present in the Nez Du Vin for white wines, lemon, grapefruit, pineapple, lychee, muscat, pear, walnut, quince, hawthorn, honey, butter and apricot.

The tasters evaluated commercial young white wines from La Mancha region to represent the full range of sensory characteristics of these wines. In each session, the assessors evaluated three wines, individually described their aroma. Then the panellists generated with the management of a panel leader, through consensus the attributes that most described the aroma sensory profile of the research wines creating a sensory descriptive ballot for the wines and linking each descriptor with a non structured scale of 10 cm where the left-hand end of the scale was “not perceptible” and the right-hand end was “strongly perceptible”. The reference standard used for each attribute evaluated in La Mancha Chelva wines is shown in Table 1 of supplementary material.

When the training period was finished the definitive election of the assessors was performed. Finally 10 assessors were selected as panel members whit aged ranged between 30 and 50 years. For this propose, each panellist evaluated six white wines with five replications using the descriptive ballot, and an analysis of variance (ANOVA) (source of variation: wine and replications) was carried out for each panellist and each attribute. The level of significance (P) of the F value, calculated by the ANOVA for the source of variation ‘wine’ (PFwine), was used as the criterion to estimate the discriminative power of each judge, and the level of significance (P) of the F value calculated for the source of variation ‘replication’ (PFreplication) was used as the criterion to estimate the reproducibility of each judge. Only assessors who showed satisfactory discriminative power (PFwine ≤ 0.30), reproducibility (PFreplication ≥ 0.05) and consensus with the rest of the panel for at least 80% of the attributes showed in the ballot were selected as members of sensory panel (Damásio & Costell 1991). No significant interactions were found in any assessor.

Standard sensory-analysis chamber (UNE-EN ISO 8589, 2010) equipped with separate booths, was used to perform the wine sensory evaluation. Wine samples (20 mL) were presented to each taster in standard wine-testing glasses according to standard (UNE 87022, 1992) at 10 ± 2 °C for odour and aroma detection and presented a randomised order. Each panellist evaluated each wine with two repetitions, for each repetition, a different bottle of wine was tested (two sessions

Table 1Oenological parameters of Chelva musts and wines one month after bottling. Mean concentrations and relative standard deviations ($n = 2$).

	Vintage				
	2013	2014	2015	2016	2017
Must composition					
°Brix	21.69 ^a (0.08)	22.40 ^b (0.08)	20.60 ^c (0.05)	22.59 ^b (0.08)	23.70 ^d (0.08)
pH	3.50 ^a (0.06)	3.71 ^b (0.02)	3.19 ^c (0.01)	3.93 ^d (0.01)	3.47 ^a (0.06)
Titration acidity (g/L) _s	3.60 ^a (0.08)	3.20 ^b (0.04)	4.21 ^c (0.01)	3.11 ^b (0.01)	3.16 ^b (0.06)
Wine composition					
Ethanol (%v/v)	13.38 ^a (0.21)	13.59 ^{a,c} (0.09)	12.95 ^b (0.08)	13.89 ^c (0.16)	13.29 ^{a,b} (0.05)
pH	3.42 ^a (0.01)	3.54 ^b (0.04)	3.15 ^c (0.01)	3.55 ^b (0.01)	3.20 ^c (0.01)
Titration acidity (g/L) _s	4.28 ^a (0.06)	3.65 ^b (0.31)	4.60 ^a (0.11)	3.67 ^b (0.13)	4.64 ^a (0.11)
Volatile acidity (g/L) _{**}	0.30 ^{a,b} (0.01)	0.32 ^{a,b} (0.08)	0.21 ^a (0.02)	0.35 ^{a,b} (0.04)	0.40 ^b (0.02)
Free SO ₂	15.72 ^a (0.69)	13.54 ^{a,b} (1.58)	13.00 ^{a,b} (0.34)	9.78 ^c (0.93)	11.26 ^{b,c} (0.40)
Total SO ₂	75.06 ^a (4.29)	73.50 ^a (1.04)	71.44 ^a (0.93)	74.83 ^a (1.20)	71.81 ^a (0.75)

a, b, c, d Different superscripts in the same row indicate statistical differences at the 0.05 level according to the Student-Newman-Keuls test.

* As tartaric acid.

** As acetic acid.

per year).

2.7. Statistical analysis

The statistical treatments were performed using SPSS 23.0 for Windows statistical package. One way ANOVA test was made in order to determine significant differences in the concentration of volatile compounds and in the mean intensity of aroma sensory attributes of La Mancha Chelva white wines and when the samples exhibited significance between them, Student-Newman-Keuls test was conducted with the level of significance set at $P < .05$. In order to evaluate the relationship between aroma sensory attributes and volatile compounds with OAV > 0.1, partial least squares regression (PLS) was applied using XLSTAT. PLS shows the relationship between X data (volatile compounds) and Y data (sensory descriptor). This is a data reduction method because it reduces the X variables to a set of no correlated factors that describe the variation in the data.

3. Results and discussion

3.1. Oenological parameters of musts and wines

Table 1 shows the general composition of musts and wines one month after bottling. These parameters were inside the normal values for white wines of La Mancha region and were in agreement with a quality vinification (Sánchez-Palomo, Alonso Villegas & González-Viñas, 2015; Sánchez-Palomo et al., 2006). It is important to note that the musts of the 2013 and 2015 vintage showed inferior values of °Brix and pH and higher values of total acidity than the rest of studied vintages. On the other hand, wines elaborated in these vintages presented lower alcoholic grade than the other ones. In all studied wines the concentrations of total SO₂ were under legal limits.

3.2. Wine volatile compounds

Tables 2 and 3 show respectively the concentrations (µg/L) of 77 varietal aroma compounds and volatile compounds formed principally during alcoholic fermentation positively identified and quantified.

3.2.1. C₆ compounds

C₆ compounds are principally formed by oxidation of the grape skin's unsaturated fatty acids during must processing, and therefore their concentration mainly depends on grape maturity. C₆ compounds are one of the most important group of varietal aroma compounds of La Mancha Chelva wines, with 1-hexanol as compound that showed the major concentration in all studied wines. C₆ compounds are related

with “vegetable” and “herbaceous” notes of wine aroma and generally, when their concentration is above its threshold values, have an undesirable consequence on the quality of the wine. (Ferreira, Fernandez, Peña, Escudero & Cacho, 1995). In this study, the concentration of 1-hexanol and *cis*-3-hexen-1-ol are lower value than their odour threshold (8000 µg/L and 400 µg/L respectively Güth, 1997). The level and ratio between *trans* and *cis*-3-hexen-1-ol could be used as characteristic of the *V. vinifera* grape variety (Versini, Orriols & Dalla Serra, 1994). La Mancha Chelva wines presented lower concentrations of *trans*-3-hexen-1-ol than of the *cis* isomer in all vintages considered. Chelva wines from 2013 and 2015 vintages showed the higher concentration of C₆ compounds, due to a lightly inferior ripening stage than the rest of considered vintages (Flanzly, 2003; Ribéreau-Gayon, Glories, Maujean, & Dubourdieu, 2000).

3.2.2. Terpene and C₁₃-norisoprenoids compounds

Terpene compounds were generally related with floral and citric notes of wine aroma, are typical of aromatic grape varieties, such as Muscat, and their importance are related with their low odour threshold (Etiévant, 1991; Güth, 1997). Linalool, *ho*-trienol, *beta*-citronellol, nerol, geraniol, 2,6-dimethyl-3,7-octadiene-2,6-diol and (*E*)-8-hydroxylinalool were the terpene compounds detected and quantified in La Mancha Chelva wines from all studied vintages. However, the concentrations of these compounds are lower than their odour thresholds and their contribution to the aroma of La Mancha Chelva wine appears insignificant.

Due to their low odour threshold (Güth, 1997) C₁₃-norisoprenoids are a varietal aroma compounds that have key role on the aroma tipicity of wines. β -damascenone can have a great sensorial impact on wines and is normally related with floral and exotic fruit aroma notes (Escudero, Farina, Campo, Cacho & Ferreira, 2007). Chelva wines presented in all studied vintages concentrations of *beta*-damascenone that exceeded their odour threshold (0.05 µg/L) (Güth, 1997). The concentrations of terpene and C₁₃-norisoprenoids compounds in 2013 and 2015 vintages were lower than the rest of studied vintages.

3.2.3. Benzene compounds

The benzene compounds are a quantitatively and qualitatively key group of varietal aroma compounds of La Mancha Chelva wines, which contain aromatic alcohols, aldehydes, volatile phenols and shikimic acid derivatives. The concentrations of benzaldehyde and benzyl alcohol were lower than their odour threshold (350 y 10,000 µg/L, respectively) by Etiévant, 1991, although these compounds can be enhanced the fruity and floral aromas of wines by synergic effect. On the other hand, the concentrations of eugenol and isoeugenol, associated to

Table 2Varietal aroma compounds of Chelva wines one month after bottling. Mean concentration ($\mu\text{g/L}$) and relative standard deviations ($n = 2$).

RI ^a	Source	Compounds	Vintage				
			2013	2014	2015	2016	2017
1282	Fluka	1-hexanol	439.17 ^a (0.34)	339.50 ^c (1.87)	627.00 ^b (3.61)	352.50 ^c (0.20)	308.00 ^d (2.30)
1286	Sigma-Aldrich	(E)-3-hexen-1-ol	7.08 ^a (3.10)	2.34 ^b (0.30)	4.22 ^c (3.35)	5.49 ^d (3.86)	3.44 ^e (0.62)
1296	Sigma-Aldrich	(Z)-3-hexen-1-ol	269.50 ^a (0.79)	323.50 ^b (5.03)	322.50 ^b (0.66)	318.50 ^b (1.11)	100.50 ^c (3.52)
1300	Sigma-Aldrich	(E)-2-hexen-1-ol	3.21 ^a (1.10)	2.50 ^b (1.70)	3.97 ^c (1.07)	1.33 ^d (1.06)	1.26 ^d (0.56)
1394	Sigma-Aldrich	2-ethyl-1-hexanol	3.18 ^a (2.67)	3.23 ^a (0.66)	2.41 ^b (1.17)	2.14 ^c (0.33)	2.52 ^b (0.28)
		Total C6 compounds	722.13	671.06	960.10	679.96	415.71
1529	Fluka	Linalool	2.00 ^a (1.06)	2.97 ^b (1.90)	0.53 ^c (0.27)	3.00 ^b (1.18)	5.77 ^d (2.94)
1650	Tentatively identified	Ho-trienol	3.39 ^a (1.46)	4.27 ^b (1.66)	3.17 ^a (2.01)	5.38 ^c (1.45)	5.58 ^c (3.68)
1703	Sigma-Aldrich	4-oxo-isophorone	Tr	1.91 (1.11)	n.d.	Tr	n.d.
1755	Fluka	β -citronellol	2.31 ^a (0.31)	3.51 ^b (2.22)	Tr	3.89 ^c (0.55)	5.85 ^d (4.11)
1777	Sigma-Aldrich	Nerol	6.04 ^a (0.47)	8.66 ^b (1.63)	6.01 ^a (0.59)	9.01 ^b (0.39)	10.76 ^c (4.34)
1801	Firmenich	β -damascenone	1.92 ^a (1.11)	3.82 ^b (2.22)	2.53 ^c (0.84)	4.50 ^d (2.05)	5.04 ^e (1.54)
1831	Fluka	Geraniol	3.19 ^a (0.89)	4.61 ^b (1.38)	2.02 ^c (0.35)	6.04 ^d (0.47)	4.89 ^c (3.91)
1902	Tentatively identified	2,6-dimethyl-3,7-octadiene-2,6-diol	8.56 ^a (0.74)	10.77 ^b (0.13)	8.37 ^a (0.76)	11.65 ^c (4.68)	8.34 ^a (6.10)
2200	Tentatively identified	(E)-8-hydroxy-linalool	11.18 ^a (1.01)	22.93 ^b (3.79)	10.92 ^a (2.20)	22.42 ^b (8.83)	16.72 ^d (9.05)
2582	Tentatively identified	3-oxo- α -ionol	28.33 ^a (5.49)	31.88 ^b (4.41)	28.91 ^{a,b} (0.56)	27.46 ^{a,b} (8.06)	24.36 ^{a,b} (6.71)
2722	Tentatively identified	3-hydroxy-7,8-dihydro- β -ionol	14.75 ^a (2.49)	18.11 ^b (6.83)	n.d.	21.44 ^c (3.60)	22.54 ^c (9.07)
3170	Tentatively identified	Vomifolol	55.06 ^{a,b} (3.01)	64.47 ^b (9.75)	47.91 ^a (3.01)	61.44 ^b (2.07)	79.17 ^c (2.05)
		Total terpene and C13-norisoprenoids compounds	136.70	175.97	110.34	176.19	189.00
1503	Sigma-Aldrich	Benzaldehyde	5.88 ^a (1.32)	5.56 ^a (3.18)	3.83 ^b (1.85)	4.81 ^c (1.91)	6.60 ^d (5.47)
1505	Tentatively identified	3(2H)-2-methyldihydro-thiophenone	9.54 ^a (2.96)	12.91 ^b (7.78)	8.47 ^a (1.17)	11.31 ^c (0.13)	15.06 ^d (4.65)
1882	Sigma-Aldrich	Guaiacol	1.34 ^a (0.53)	1.28 ^b (0.28)	0.48 ^c (0.88)	1.27 ^b (0.17)	2.35 ^d (1.51)
1895	Sigma-Aldrich	Benzyl alcohol	80.02 ^a (0.38)	117.05 ^b (5.62)	84.22 ^a (5.43)	129.00 ^b (2.19)	162.32 ^c (4.86)
1899	Tentatively identified	1,2-benzothiazole	9.71 ^a (4.44)	5.71 ^b (1.36)	1.53 ^c (0.46)	4.52 ^d (2.82)	5.47 ^b (0.91)
1971	Sigma-Aldrich	Phenol	4.72 ^{a,b} (0.30)	4.74 ^{a,b} (0.60)	4.82 ^b (2.05)	4.58 ^a (1.24)	4.44 ^c (0.32)
2193	Sigma-Aldrich	Eugenol	5.22 ^a (0.27)	7.25 ^b (0.10)	2.75 ^c (0.51)	7.00 ^d (0.20)	7.37 ^e (1.25)
2219	Sigma-Aldrich	Acetophenone	266.00 ^a (6.38)	221.50 ^b (6.07)	215.00 ^b (1.32)	205.50 ^b (3.10)	241.50 ^{a,b} (4.39)
		4-vinylguaiacol	Tr	Tr	Tr	Tr	Tr
2302	Lancaster	Isoeugenol	14.52 ^a (11.64)	17.21 ^a (5.47)	9.07 ^b (6.55)	14.46 ^a (1.13)	21.83 ^c (8.36)
2345	Tentatively identified	2,3-dihydro benzofuran	29.33 ^a (4.22)	57.58 ^b (4.73)	47.07 ^c (4.91)	32.67 ^{a,d} (0.56)	38.56 ^d (10.12)
2378	Sigma-Aldrich	Benzoic acid	5.62 ^a (1.44)	2.44 ^b (5.80)	1.80 ^c (2.76)	2.50 ^b (2.26)	5.59 ^a (1.14)
2511	Panreac	Vanillin	4.39 ^a (5.64)	6.46 ^b (1.75)	3.10 ^c (0.23)	6.90 ^d (2.46)	10.38 ^e (0.75)
2543	Sigma-Aldrich	Ethyl vanillate	2.28 ^a (2.48)	3.22 ^b (0.44)	n.d.	2.17 ^c (2.94)	Tr
2936	Sigma-Aldrich	Zingerone	17.15 ^a (5.36)	23.95 ^b (3.84)	15.85 ^a (4.02)	20.65 ^c (1.71)	24.10 ^b (1.17)
		Total benzene compounds	456.18	486.83	397.97	447.32	545.54

^aLinear retention index on a DB Wax column; n.d., not detected; Tr, Traces.

a, b, c,d,e Different superscripts in the same row indicate statistical differences at the 0.05 level according to the Student-Newman-Keuls test.

sweet spice notes, exceeded in all studied wines their odour threshold (6 $\mu\text{g/L}$ Güth, 1997).

When regarding volatile phenols, guaiacol and 4-vinylguaiacol were detected in La Mancha Chelva wines. These compounds can influence the aroma of wine in a positive or negative way based on their concentrations. Odour threshold of guaiacol (10 $\mu\text{g/L}$) by Güth, 1997 was not exceeded in any studied wines and 4-vinylguaiacol only was detected in trace concentrations.

3.2.4. Acetaldehyde

Acetaldehyde, related with fruity, dried fruits and nutty odours, is the principal aldehyde identified in La Mancha Chelva wines. The concentration of acetaldehyde in wine can be varied with the winemaking conditions, particularly with the quantity of SO_2 added to the medium. In the current study, the winemaking conditions were identical in all considered vintages, consequently the variations on the concentration of these compounds can be due to the changes on composition of the initial musts (Table 1).

3.2.5. Esters

Fermentation-derived esters are related with fruity character of the wines (Dzialis, Park, Steensels, Lievens & Verstrepen., 2017). Between the identified esters, ethyl esters of fatty acids are responsible for the fruity aromas of the wines. Ethyl octanoate, ethyl decanoate, and ethyl hexanoate were the most abundant ethyl esters.

Other group of esters, acetate esters, are formed from acetic acid and higher alcohols, and are considered to have a greater effect on the perceived aroma than the ethyl esters (Dzialis et al., 2017). In this

research, the acetate esters identified were ethyl acetate, hexyl acetate, 2-phenylethyl acetate, and isoamyl acetate. Among the identified and quantified acetate esters were 2-phenylethyl acetate and 3-methyl-1-butanol acetate, associated with the positive attributes, “rose” and “banana” respectively. The concentration of 3-methyl-1-butanol acetate presented values above the threshold value in all the wines studied.

3.2.6. Acids

Fatty acids in wines are formed principally during the fermentative step by microbial organisms, their formation is influenced by the initial composition of the must and by fermentation conditions (Schneider, 1979) and have been related with fruity, cheese, fatty, and rancid aromas (Rocha, Rodrigues, Coutinho, Delgadillo, & Coimbra, 2004). Iso-butyric, isovaleric, hexanoic, octanoic and decanoic acids were presented in higher concentration in all studied wines. The concentration of fatty acids was in agreement with those found by Sánchez-Palomo et al., 2006, 2007, 2015 in white wines made from other grape varieties cultivated in Castilla-La Mancha region and elaborated using same winemaking conditions.

3.2.7. Alcohols

Higher alcohols, known as fusel alcohols, are the main group of volatile metabolites, synthesized by yeast during alcoholic fermentation (Dzialis et al., 2017). In general, regardless of the variety of grapes used in the winemaking process, the yeast produces alcohol levels in concentrations ranging from 239 mg/L to 288 mg/L, below the level of 300 mg/L, so that contribute in a positive way to the aroma of the wine, which enhances their aromatic complexity (Rapp & Versini, 1991).

Table 3

Volatile compounds formed principally during alcoholic fermentation of Chelva wines one month after bottling. Mean concentration ($\mu\text{g/L}$) and relative standard deviations ($n = 2$).

RI ^A	Source	Compounds	Vintage				
			2013	2014	2015	2016	2017
800	Sigma-Aldrich	Aldehydes Acetaldehyde*	76.35 ^a (1.01)	83.04 ^b (5.55)	74.90 ^a (3.02)	62.44 ^c (3.03)	93.29 ^d (0.89)
834	Sigma-Aldrich	Esters Ethyl acetate*	30.34 ^a (2.98)	33.37 ^a (4.96)	41.34 ^a (1.59)	33.89 ^a (4.11)	68.29 ^b (9.71)
1080	Fluka	Ethyl butyrate	16.63 ^a (7.40)	22.73 ^b (4.51)	30.10 ^c (3.74)	19.70 ^{ab} (8.58)	24.09 ^b (9.92)
1145	Sigma-Aldrich	Isoamyl acetate	208.50 ^a (1.02)	239.00 ^{ab} (3.55)	265.50 ^{ab} (12.52)	319.00 ^b (10.20)	238.50 ^{ab} (10.38)
1294	Sigma-Aldrich	Hexyl acetate	9.92 ^a (3.49)	9.80 ^a (4.98)	9.07 ^{ab} (1.48)	7.61 ^b (6.60)	9.23 ^{ab} (9.89)
1326	Sigma-Aldrich	Ethyl lactate	327.00 ^a (7.78)	260.00 ^a (7.61)	222.00 ^a (5.10)	259.00 ^a (15.29)	296.00 ^a (9.14)
1185	Fluka	Ethyl hexanoate	134.00 ^a (11.61)	145.00 ^a (10.73)	181.50 ^a (4.29)	162.00 ^a (2.62)	167.45 ^a (9.67)
1436	Sigma-Aldrich	Ethyl octanoate	366.50 ^a (7.91)	382.50 ^a (2.40)	303.50 ^b (2.56)	301.00 ^b (10.34)	233.00 ^c (0.61)
1655	Fluka	Ethyl decanoate	179.00 ^a (3.00)	198.00 ^a (7.86)	180.50 ^a (5.09)	192.50 ^a (12.12)	168.00 ^a (8.64)
1499	Tentatively identified	3-hydroxy, ethyl butyrate	44.20 ^a (2.24)	31.75 ^b (2.90)	40.00 ^a (11.31)	42.90 ^a (1.65)	24.00 ^c (2.95)
1522	Tentatively identified	Ethyl-dl-2-hydroxycaproate	1.09 ^a (0.65)	1.02 ^b (0.70)	0.66 ^c (0.53)	0.57 ^d (0.74)	0.83 ^c (0.34)
1605	Sigma-Aldrich	Diethyl malonate	0.27 ^a (0.52)	0.28 ^b (0.25)	0.35 ^c (0.40)	0.30 ^d (0.24)	0.31 ^c (0.23)
1702	Fluka	Diethyl succinate	60.24 ^a (1.27)	47.84 ^b (2.78)	61.02 ^a (0.50)	63.00 ^a (7.41)	55.25 ^{ab} (9.84)
1787	Fluka	Methyl Salicylate	1.03 ^a (0.69)	Tr	1.08 ^b (0.39)	Tr	Tr
1827	Tentatively identified	4-hydroxy, ethyl butyrate	467.50 ^a (2.57)	350.50 ^{ab} (14.73)	338.50 ^{ab} (16.92)	310.00 ^b (15.97)	285.00 ^{ab} (11.91)
1936	Fluka	2-phenylethyl acetate	55.15 ^a (3.97)	46.25 ^{ab} (5.05)	38.05 ^{bc} (2.42)	24.25 ^d (3.21)	33.95 ^c (21.87)
2070	Sigma-Aldrich	Diethyl malate	40.55 ^a (10.29)	38.85 ^a (2.00)	43.20 ^a (3.60)	41.20 ^a (4.46)	41.55 ^a (6.98)
2286	Fluka	Ethyl cinnamate	Tr	Tr	Tr	Tr	Tr
2331	Tentatively identified	Monoethyl succinate	325.65 ^a (0.48)	277.34 ^{bc} (5.27)	248.15 ^c (3.50)	244.15 ^c (5.88)	292.95 ^b (2.44)
1426	Sigma-Aldrich	Acids Acetic acid	4.82 ^a (1.62)	3.67 ^b (3.47)	4.34 ^{a,c} (8.15)	4.58 ^a (2.78)	3.88 ^{b,c} (3.83)
1546	Sigma-Aldrich	Propanoic acid	1.44 ^a (2.46)	2.37 ^b (2.09)	1.64 ^c (3.03)	1.43 ^a (0.50)	2.51 ^d (1.69)
1583	Fluka	Isobutyric acid	258.00 ^a (1.10)	294.00 ^a (14.43)	304.00 ^a (9.30)	317.00 ^a (12.05)	320.00 ^a (9.72)
1600	Fluka	Butyric acid	16.90 ^{ab} (7.53)	20.25 ^b (7.33)	19.10 ^b (1.48)	14.67 ^a (10.89)	17.25 ^{ab} (2.87)
1642	Sigma-Aldrich	Isovaleric acid	170.00 ^a (4.16)	214.00 ^a (11.23)	193.50 ^a (6.94)	267.00 ^b (4.77)	196.50 ^a (11.88)
1703	Fluka	Valeric acid	2.24 ^a (4.42)	3.07 ^b (0.46)	3.73 ^c (0.38)	2.81 ^b (10.84)	2.91 ^b (0.97)
1816	Fluka	Hexanoic acid	441.50 ^a (2.08)	360.00 ^a (1.96)	417.00 ^a (13.23)	361.00 ^a (1.57)	404.00 ^a (10.85)
1929	Sigma-Aldrich	(<i>E</i>)-2-hexenoic acid	0.95 ^a (0.45)	0.80 ^b (0.89)	0.87 ^c (0.41)	0.74 ^d (0.48)	0.77 ^e (0.46)
2024	Fluka	Octanoic acid	474.00 ^a (2.69)	381.00 ^b (2.23)	404.00 ^b (1.75)	387.50 ^b (0.55)	398.50 ^b (3.37)
2289	Sigma-Aldrich	Decanoic acid	350.50 ^a (6.25)	253.50 ^b (14.23)	298.00 ^{ab} (4.75)	253.00 ^b (6.71)	262.00 ^b (8.64)
2439	Sigma-Aldrich	Dodecanoic acid	21.27 ^a (2.49)	21.98 ^a (9.97)	15.22 ^b (5.25)	13.22 ^b (3.48)	21.44 ^a (3.03)
879	Sigma-Aldrich	Alcohols Methanol*	21.09 ^a (5.97)	23.42 ^{ab} (0.72)	32.80 ^c (0.41)	24.87 ^b (7.56)	36.12 ^d (1.64)
1060	Sigma-Aldrich	1-propanol*	25.10 ^a (4.51)	15.72 ^b (2.56)	20.24 ^c (3.77)	21.07 ^c (2.45)	33.00 ^d (0.86)
1214	Merck	Isobutanol*	24.13 ^a (3.08)	35.37 ^b (0.26)	42.06 ^{bc} (10.96)	47.13 ^c (4.29)	34.45 ^b (3.90)
1221	Sigma-Aldrich	2-methyl-1-butanol*	31.55 ^a (4.26)	48.05 ^b (7.21)	50.45 ^b (7.71)	32.65 ^c (12.78)	29.65 ^a (8.82)
1221	Sigma-Aldrich	3-methyl-1-butanol*	124.00 ^a (1.14)	138.00 ^b (14.29)	128.50 ^{ab} (6.29)	124.00 ^a (1.14)	138.00 ^a (5.12)
1155	Sigma-Aldrich	1-butanol	1.67 ^a (0.85)	2.72 ^b (0.52)	3.41 ^c (1.87)	1.74 ^a (1.22)	2.10 ^d (0.34)
1260	Sigma-Aldrich	1-pentanol	1.46 ^a (0.19)	1.93 ^b (0.37)	1.77 ^c (1.20)	1.75 ^c (0.41)	1.40 ^d (0.51)
1328	Fluka	4-methyl-1-pentanol	3.01 ^a (1.41)	3.84 ^b (0.55)	3.59 ^c (0.20)	3.49 ^d (1.42)	3.44 ^d (1.03)
1341	Fluka	3-methyl-1-pentanol	7.94 ^{ab} (5.79)	8.72 ^a (0.73)	6.95 ^b (2.14)	8.97 ^a (7.65)	7.75 ^{ab} (0.27)
1472	Fluka	1-heptanol	0.34 ^a (0.21)	0.66 ^b (0.53)	0.71 ^c (0.20)	0.40 ^d (0.18)	0.36 ^e (0.98)
1545	Fluka	2,3-butanediol (levo)	39.97 ^a (1.65)	22.35 ^b (0.32)	29.85 ^c (2.61)	32.17 ^d (1.63)	31.87 ^d (2.97)
1585	Fluka	2,3-butanediol (meso)	5.49 ^a (4.25)	4.19 ^b (0.68)	6.62 ^c (10.68)	5.33 ^c (2.79)	5.60 ^a (1.39)
1725	Sigma-Aldrich	3-(methylthio)-1-propanol,	176.50 ^a (8.41)	238.00 ^a (10.70)	218.50 ^a (6.15)	209.00 ^a (9.47)	212.50 ^a (14.31)
1892	Fluka	2-phenylethanol*	13.17 ^a (5.23)	12.42 ^a (0.87)	14.17 ^a (3.93)	12.32 ^a (0.16)	14.02 ^a (5.62)
1650	Sigma-Aldrich	Lactones γ -butyrolactone	1.90 ^a (0.37)	1.74 ^b (0.41)	1.86 ^a (0.76)	2.66 ^c (0.27)	2.85 ^d (0.99)

^aLinear retention index on a DB Wax column; n.d., not detected; Tr, Traces.

* Concentration mg/L; ^a, ^b, ^c, ^d, ^e Different superscripts in the same row indicate statistical differences at the 0.05 level according to the Student-Newman-Keuls test.

Between the aliphatic alcohols 3-methyl-1-butanol showed the highest concentration in all studied wines. 2-phenylethanol, synthesized via the Ehrlich pathway through metabolic reactions that involves transamination of the amino acid L-phenylalanine, could contribute to the wine aroma with a rose notes (Étiévant, 1991). In all studied wines the olfactory threshold of this compound (10,000 $\mu\text{g/L}$) by Güth, 1997 exceeded showing values between 12,320.00 and 14,022.00 $\mu\text{g/L}$.

3.3. Impact compounds of La Mancha Chelva wines

In order to assess the influence of the aroma compounds identified and quantified in La Mancha Chelva wines, the odour activity values (OAVs) were used (Francis & Newton, 2005). Compounds existing in greater concentrations than their odour threshold (OAVs > 1) are

defined as aroma impact compounds nevertheless and contribute individually to the wine aroma. Nevertheless other authors proposed that when the OAV of volatile compound is < 1, it may be contribute to the overall aroma of wines due to synergy effects of the similar compounds (Lopez, Ferreira, Hernández & Cacho 1999).

The odour descriptors, odour threshold obtained from the bibliographic references, aromatic series, OAV minimum, OAV maximum and OAV medium of the volatile compounds with OAV \geq 0.1 of La Mancha Chelva wines are shown in Table 4 (Güth, 1997; Étiévant, 1991; Ferreira, López & Cacho, 2000; Kotseridis & Baumes, 2000; Lambrechts & Pretorius, 2000; Singleton, 1995; López, Ortin, Perez-Trujillo, Cacho & Ferreira, 2003).

La Mancha Chelva wines presented a total of 28 volatile compounds with OAV \geq 0.1. Acetaldehyde, beta-damascenone, ethyl octanoate,

Table 4

Odour descriptors, odour threshold ($\mu\text{g/L}$), aromatic series, minimum, maximum and medium odour activity values of some volatile compounds of La Mancha Chelva wines from 5 consecutive vintages.

Compounds	Odour descriptors	Odour threshold ($\mu\text{g/L}$)	Aromatic series ^a	OAV minimum	OAV maximum	OAV medium
Acetaldehyde	Pungent, ripe apple	500	1,6	124.88	186.57	156.00
β -damascenone	Sweet, fruity	0.05	1,4	38.40	100.80	71.16
Ethyl octanoate	Caramel, fruity	5	1,4	46.60	76.50	63.46
Ethyl hexanoate	Green apple	14	1	9.57	12.96	11.29
Isoamyl acetate	Banana	30	1	6.95	10.63	8.47
Isovaleric acid	Acid, rancid	33	4,6	5.15	8.09	6.31
Ethyl acetate	Fruity, solvent	7500	1,6	4.05	9.11	5.53
3-methyl-1-butanol	Burnt, Alcohol	30,000	4,6	4.13	6.26	4.95
Isoeugenol	Clove	6	5	1.51	3.64	2.57
2-phenylethanol	Floral, rose	10,000	2	1.23	1.42	1.32
2-methyl-1-butanol	Burnt, Alcohol	30,000	4,6	0.99	1.68	1.28
Ethyl butyrate	Fruity	20	1	0.83	1.50	1.13
Eugenol	Spices, clove, honey	6	4,5,7	0.46	1.23	0.99
Hexanoic acid	Sweat	420	6	0.86	1.05	0.94
Isobutanol	Bitter, Green	40,000	3,6	0.60	1.18	0.92
Ethyl decanoate	Caramel, fruity	200	1,4	0.84	0.99	0.92
Octanoic acid	Sweat, cheese	500	6	0.76	0.95	0.82
(Z)-3-hexen-1-ol	Green, cut grass	400	3	0.25	0.81	0.67
Decanoic acid	Rancid fat	1000	6	0.25	0.35	0.28
3-methylthio-1-propanol	Cooked vegetable	1000	6	0.18	0.24	0.21
Linalool	Floral	15	2	0.04	0.38	0.19
2-phenylethyl acetate	Floral	250	2	0.10	0.22	0.16
Geraniol	Rose, geranium	30	2	0.07	0.20	0.14
Isobutyric acid	Rancid, butter, cheese	2300	6	0.11	0.14	0.13
Guaiacol	Medicine, caramel, smoke	10	4,6	0.05	0.23	0.13
Vanillin	Vanillin	60	5,7	0.05	0.17	0.10
Butyric acid	Rancid, cheese, sweat	173	6	0.08	0.12	0.10
Citronellol	Floral	40	2	0.00	0.15	0.08

^a 1 = fruity; 2 = floral; 3 = green, fresh; 4 = sweet; 5 = spicy; 6 = fatty; 7 = others.

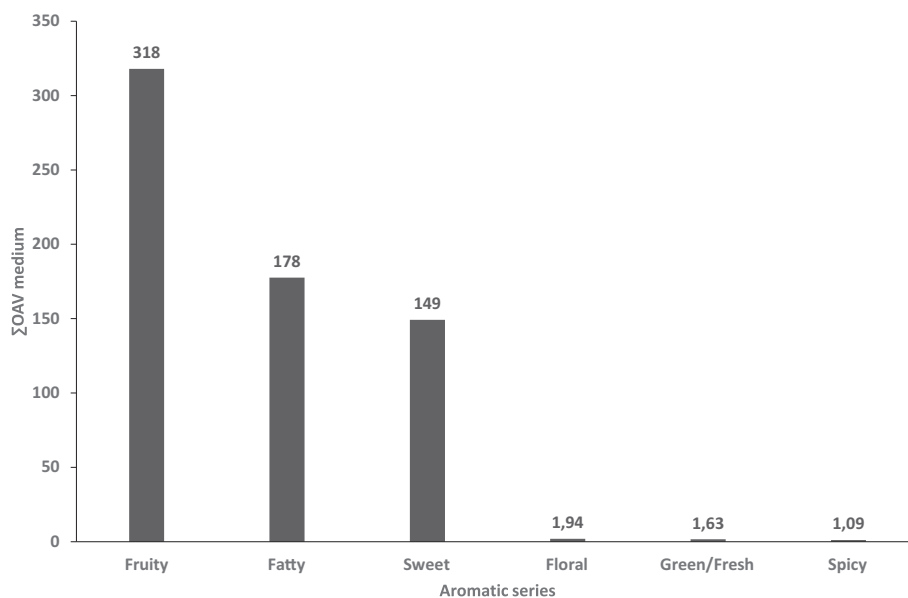


Fig. 1. Aromatic series in La Mancha Chelva wines ($\Sigma\text{OAV}_{\text{medium}}$ over five vintages).

ethyl hexanoate, isoamyl acetate, isovaleric acid, ethyl acetate, 3-methyl-1-butanol, isoeugenol, 2-phenylethanol, 2-methyl-1-butanol, ethyl butyrate, eugenol, hexanoic acid and isobutanol presented $\text{OAV} > 1$ in La Mancha Chelva white wines and could be considered as aroma impact compounds of these wines.

In order to relate chemical composition to sensory profile the odour descriptors of volatile compounds were grouped into several aromatic series. The aromatic series used to define the aroma of Chelva white wines were fruity, floral, green/fresh, sweet, spice, fatty and other. Due to the great difficulty to define the olfactory perceptions, there are some volatile compounds assigned into one or more aromatic series (Lorenzo,

Pardo, Zalacain, Alonso & Salinas, 2008; Gómez-Míguez, Gómez-Míguez, Vicario & Heredia, 2007).

Fig. 1 graphed the total intensity of each aromatic series used to define the aroma of La Mancha Chelva wines (without aromatic series 7 defined as other odours) calculated as the sum of the $\text{OAV}_{\text{medium}}$ of each volatile compounds assigned to each series. As can be observed fruity, sweet and fatty were the main series of the aroma of La Mancha Chelva wines.

It is important to note that although the aromatic series 6 (pungent, chemical, fatty and dry) is one of the principal aromatic series in the aroma of Chelva wines from Castilla-La Mancha, these aromas notes

Table 5Aroma sensory profile of wines Chelva wines one month after bottling. Mean scores of 10 judges and standard deviations ($n = 5$).

	Vintage				
	2013	2014	2015	2016	2017
Odour intensity	7.35 ^a (0.49)	7.50 ^a (0.71)	6.80 ^a (0.42)	8.35 ^b (0.49)	6.80 ^a (0.42)
Fresh	7.50 ^{a,b} (0.42)	7.60 ^{a,b} (0.57)	8.52 ^c (0.64)	6.85 ^a (0.21)	7.97 ^b (0.15)
Peach	7.20 ^a (0.71)	7.85 ^{a,b} (0.21)	6.96 ^a (0.36)	8.05 ^b (0.21)	7.12 ^a (0.13)
Citric	2.30 ^a (0.42)	1.80 ^b (0.28)	3.59 ^c (0.27)	1.10 ^d (0.14)	2.65 ^a (0.62)
Green apple	2.55 ^a (0.64)	2.05 ^b (0.07)	4.05 ^c (0.21)	1.95 ^b (0.21)	3.55 ^d (0.49)
Tropical fruit	3.05 ^a (0.78)	3.75 ^a (0.35)	1.71 ^b (0.52)	5.05 ^c (0.78)	2.09 ^b (0.01)
Floral	3.10 ^a (0.14)	3.60 ^b (0.57)	2.05 ^c (0.07)	4.00 ^b (0.28)	2.30 ^c (0.28)
Sweet	3.10 ^a (0.71)	3.85 ^a (0.21)	1.72 ^b (0.37)	3.80 ^a (0.28)	1.95 ^b (0.21)

a, b, c Different superscripts in the same row indicate statistical differences at the 0.05 level according to the Student-Newman-Keuls test.

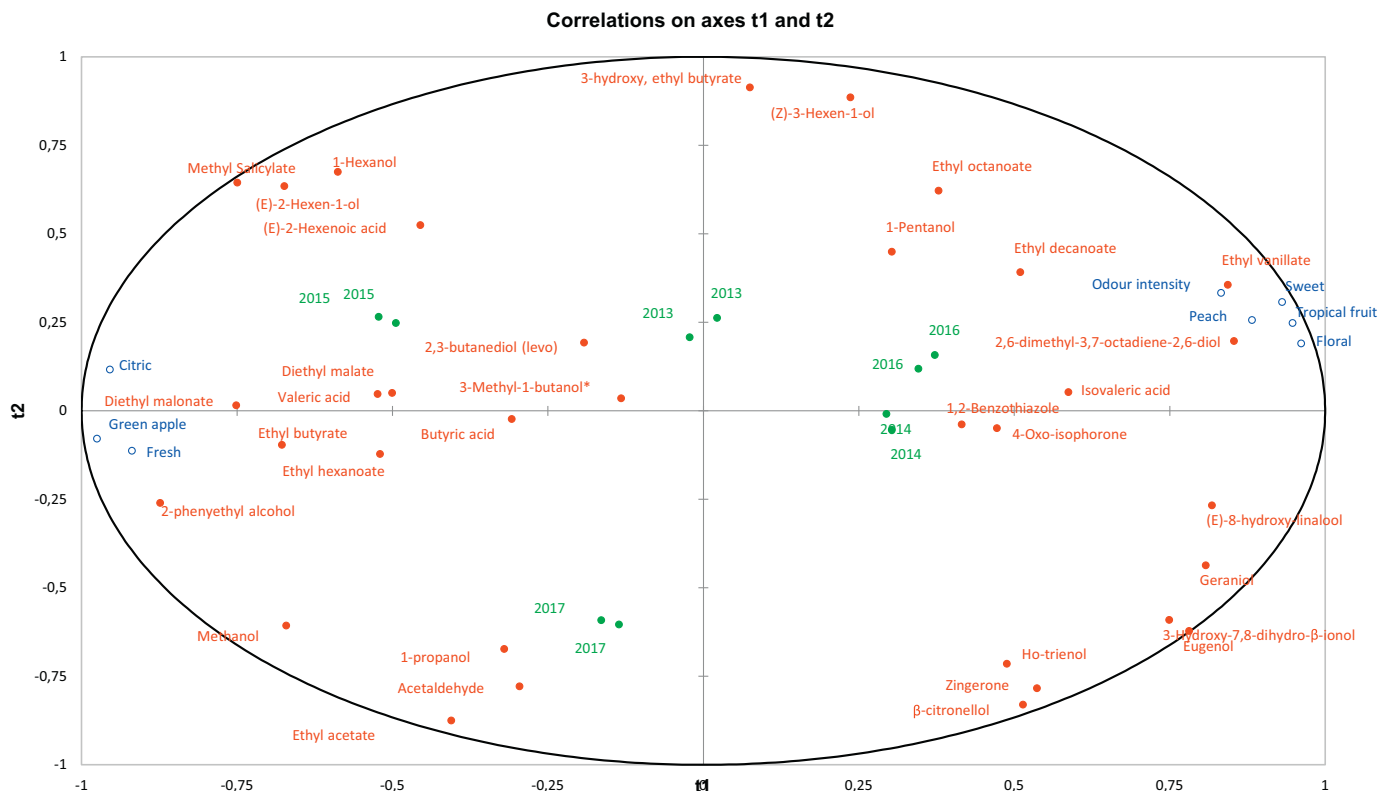


Fig. 2. PLS loading for t1 y t2 for sensory attributes (Y variables) and volatile compounds of La Mancha Chelva wines (X variables) from five vintages considered.

were not detected on the sensory aroma profile of wines. On the other hand, aromatic series 2 (floral) and 3 (green/fresh) were the minor aroma series, and these attributes were used by the tasters to define the sensory profile of La Mancha Chelva wines. These results can be attributed to the fact that the total intensity of the aromatic series were calculated as the sum of the individual OAVs of each volatile compound without taking into account the rest of the compounds present in the wine matrix. However, when considered, the synergy, suppression and effects of the wine matrix can vary the intensity of the sensory attributes of the wine aroma, so that the descriptors of some aromatic series 6 are not perceived while the intensity of those of others is increased.

3.4. Aroma sensory profile of Chelva wines

Table 5 show the mean aroma-intensity attributes of Chelva wines. In order to discriminate between the means of attributes of aroma sensory profile Student-Newman-Keuls test was applied. These results shows that the aroma of La Mancha Chelva wines was defined by the assessors as fresh, peach, tropical fruit, citric, green apple, floral and

sweet aromas. As can be see some variations in the aroma sensory profiles of Chelva wines was detected between all studied harvests. In 2015 harvest the wines showed a more different aromatic characteristic that the rest of studied wines, being more pronounced notes of fresh, green apple and citric aromas and lower intensity of peach, tropical fruit and sweet aromas.

3.5. PLS modeling relationship between sensory descriptors (aroma) and volatile compounds of wines

Partial least squares regression (PLS) was employed to establish correlations between the aroma sensory attributes of the wines and the volatile compounds identified and quantified by GC–MS.

Fig. 2 shows PLS made taking into account aroma sensory attributes and volatiles compounds identified in La Mancha Chelva wines. Based on proximity on the left side of the plot, it can be observed that the odour attributes citric, fresh and green apple were co-varied positively to the 1-hexanol, (E)-2-hexen-1-ol, ethyl butyrate, diethyl malonate, methyl salicylate, isovaleric acid, valeric acid, (E)-2-hexenoic acid and

2-phenylethanol with a high correlation coefficient (r mainly between 0.60 and 0.85). In contrast, odour intensity, sweet, peach, tropical fruit, and floral, located on the right side of the plot, were mainly correlated with some terpene compounds, ethyl esters of fatty acids and benzene compounds such as vanillin derivative and eugenol.

PLS component 1 contributed to the spread of samples on the left side of the plot mainly based on the intensity of citric, green apple and fresh; as opposed to samples on the right with general more odour intensity, tropical fruit, peach, floral and sweet aromas. Wines elaborated in 2015 and 2017 vintages are located on the left side of the plot and showed higher intensity of fresh, citric and green apple. These attributes are positively correlated with the C_6 compounds, alcohols, acetates and esters such as diethyl malate, ethyl butyrate, ethyl hexanoate, methyl salicylate, ethyl acetate, 2-phenylethyl alcohol, propanol and methanol. On the other hand wines from 2014 and 2016 vintages are located on the right side of the plot by their aroma intensity and their high intensity of tropical fruit, apricot, floral and sweet aromas. These aromas are influenced by the concentration of certain terpenes such as geraniol, 8-hidroxylinolool, 2,6-dimethyl-3,7-octadiene-2,6-diol, beta-citronellol y ho-trienol, esters such as ethyl octanoate and ethyl decanoate, ethyl vanillate, and benzene compounds such as zingerone and eugenol related principally with the sweet aromas.

4. Conclusions

This research shows the first characterization of the chemical aroma composition and sensory aroma characteristics of La Mancha Chelva white wines. The aroma of Chelva wines from La Mancha region is characterized by great concentrations of C_6 and benzene compounds, nevertheless, is also important to highlight the presence of a large number of terpene compounds. The aroma sensory profile of Chelva white wines was characterized by fresh and peach aromas with notes to citric, green apple, tropical fruit and sweet. This work displayed that Chelva grape variety, cultivated in La Mancha region, present a complex aroma characteristic and can be considered as alternative to white grape varieties used in winemaking sector in this region.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.foodres.2019.01.049>.

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