Sweet Wines Produced by an Innovative Winemaking Procedure: Colour, Active Odorants and Sensory Profile

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The colour, aroma-active compounds and sensory properties of sweet wines from Pedro Ximenez grapes produced by means of an innovative winemaking procedure, based on controlled chamber-drying of grapes, partial fermentation of the must (to 4% or 8% vol ethanol) and subsequent accelerated ageing by contact with oak chips, were studied. Fermentation made the musts less brown and more yellow, whereas ageing made them darker and increased their brown, reddish and yellowish hues. Overall, the musts fermented to 8% vol ethanol exhibited higher odour activity values (OAVs). In addition, the musts aged with oak chips were slightly different from those without chips. Expert tasters gave the highest scores to the musts fermented to 8% (v/v) ethanol with 2 g/L of oak chips added. The winemaking process studied would allow the existing range of sweet wines from dried grapes to be expanded by using a fast, flexible, hygienic procedure.

INTRODUCTION

In recent years, Pedro Ximenez sweet wine has been under high demand from consumer and the volume produced is virtually sold out every season. The first and foremost step in the production process involves sun-drying the grapes, which face a high risk of deterioration from insect attacks, potential rain and nocturnal dew. In addition, these ambient conditions can favour the production of fungal toxins such as ochratoxin A (OTA), which have an adverse impact on the health and safety of Pedro Ximenez. Unquestionably, increased control over the conditions of grape drying result in better organoleptic and sanitary properties of the final musts. In this respect, Ruiz *et al*. (2010) have shown that chamber-drying grapes under controlled thermohygrometric conditions provides raisins of substantially improved quality. Chamber-drying for grapes is a fast and reliable method, independent of meteorological conditions.

The second important issue to consider is the high levels of sugars found in musts from dried grapes. High sugar content alters the metabolic activity of yeast and can delay, or even stop, alcoholic fermentation. Moreover, fermentation in sugar-rich media is known to lead to wines with high volatile acidity and sometimes with organoleptic faults, as outlined by García-Martínez *et al*. (2011). Using yeasts that are tolerant of high sugar and ethanol concentrations allows a rapid and reliable fermentation, reducing the risk of sluggish or stuck fermentation and microbial contamination. Logically, the selected yeast strain and the moment of when the fermentation is stopped can affect the wine quality in terms of composition and sensory profile. Furthermore, the OTA contents in wines can decrease during fermentation (Pérez-Serradilla & Luque de Castro, 2008).

Another important consideration is the oxidative ageing in oak barrels. During this period of several years, wine acquires its characteristic bouquet as a result of significant changes due to different phenomena: esterification/hydrolysis and redox reactions, spontaneous clarification, CO_2 elimination, slow and continuous diffusion of oxygen through wood pores, and the extraction of tannins and aromatic substances from the wood to the wine (Camara *et al*., 2006). The different volatile compounds extracted from wood during this process (lactones, furanic compounds, vanillin derivatives, and phenol derivatives) have important sensory properties and contribute to the overall aroma of the wine.

However, ageing in oak casks takes a long time and is a very expensive process. A more economical alternative is the use of oak wood fragments. This practice first appeared in wines produced in emerging countries and later became authorised in the European Union (EU). Several studies have shown the technical possibilities of this practice (Guchu *et al*., 2006; Rodríguez-Bencomo *et al*., 2008). In addition, a recent study has shown that wines made with oak wood fragments are scarcely rejected by consumers (Pérez-Magariño *et al*., 2011). However, the sweetness, chromatic adjustment, aroma profile and complexity of the finally

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wines depends of the ageing strategy. Therefore, different factors such as doses, size, form and toast level of fragments, ageing time and maceration conditions, must be studied to obtain wines with desirable sensory properties.

In this work, the colour, aroma profile and sensory properties of sweet wines from Pedro Ximenez grapes produced by an innovative winemaking procedure, based on chamber-drying of the grapes, partial fermentation of the must by osmo-ethanol-tolerant yeasts, and subsequent accelerated ageing by contact with oak chips, was studied. The results show that is possible to produce high-quality sweet wines from raisins by the fast, cheap and hygienic method proposed.

MATERIALS AND METHODS

MUST SAMPLES

Figure 1 depicts the experimental procedure described below. In this study, 200 kg of ripe Pedro Ximenez grapes were collected in the Montilla-Moriles region (southern Spain). Three batches of grapes of 25 kg each one were distributed uniformly (14 kg/m^2) in a single layer and dried in a chamber (Frisol Climatronic, Spain) at air temperatures of 40ºC and humidity of 20%. Samples were collected periodically and the sugar content of the grapes was measured by the Luff-Schoorl method (EEC, 1990). This method is based on the reduction of copper (II) ions in alkaline solution by the reducing sugars, followed by back titration of the remaining copper. The drying was concluded when the sugar concentration was around 450 g/L. The grapes were crushed and subsequently pressed in a vertical press similar to those used at the industrial level (EG-250 Sanahuja, Castellón, Spain). The highest pressure reached in each pressing cycle was 300 bars, and each grape batch was pressed in three cycles in a thermostatised chamber at 20ºC.

Inoculation and fermentation

A total of 21 L of must was supplied with SO_2 at a concentration of 100 mg/L, blended and distributed among seven 5 000 mL Erlenmeyer flasks containing 3 L of must each. The samples were inoculated with a Saccharomyces cerevisiae strain X5 (CECT13015) previously isolated during spontaneous fermentation of musts from partially dried Pedro Ximenez grapes and chosen on the grounds of its tolerance to high sugar and ethanol concentrations in a previous experiment conducted at the Department of Microbiology of the University of Córdoba (García-Martínez et al., 2011). The starter cultures were prepared by growing each strain separately in YPD medium at 28ºC for 2 h, which was followed by centrifugation and washing with distilled water. Six flasks (two triplicates) were inoculated with 5 x 106 cells/mL and incubated at 25ºC, and the remaining 3 L of must was fortified to 18% (v/v) ethanol and stored at 4ºC in a cold chamber for use as the control.

The first three flasks were withdrawn from the chamber

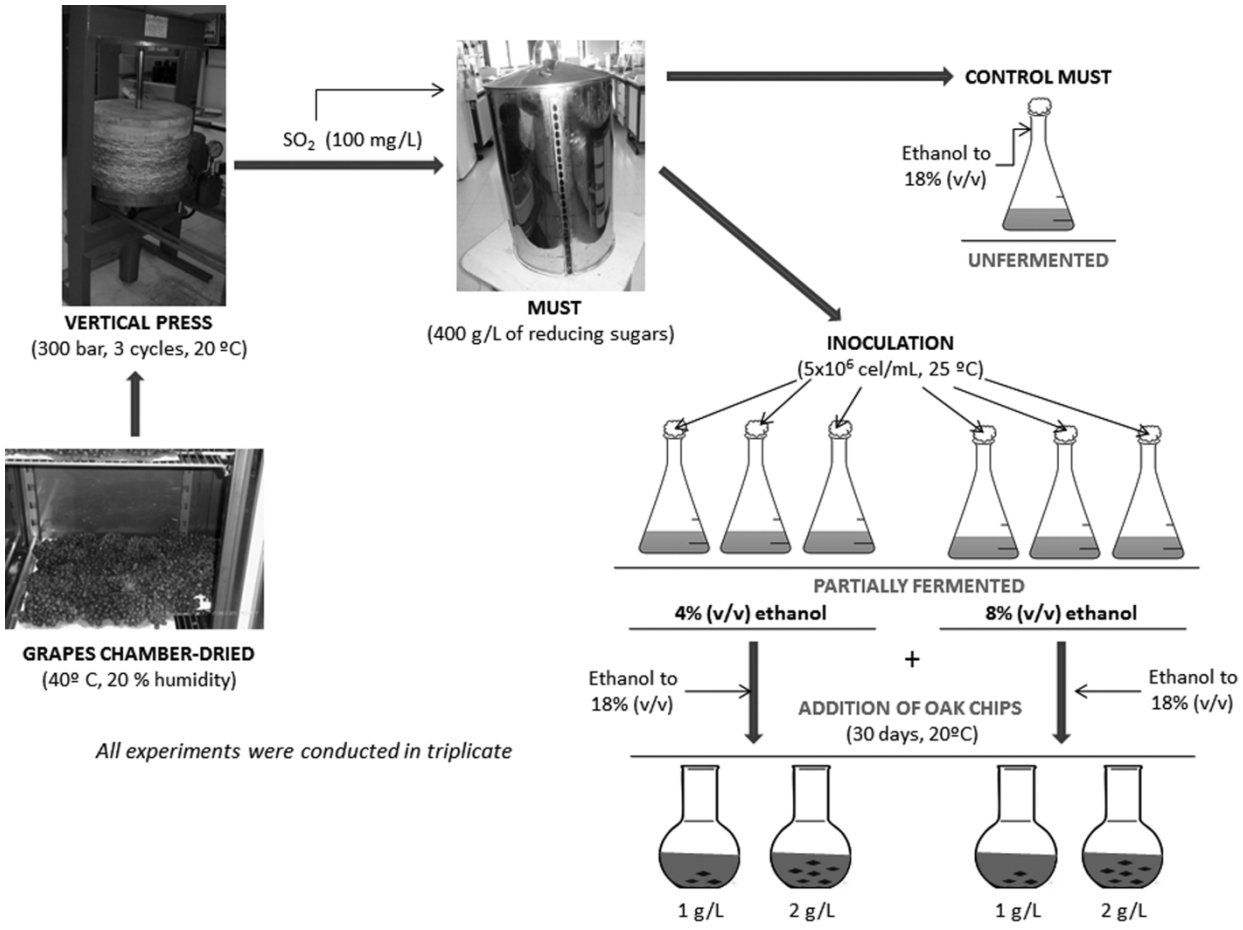

FIGURE 1
Experimental design followed in the study.

when the ethanol content reached 4% (v/v), which occurred after four days of incubation. The other three were withdrawn at an ethanol content of close to 8% (v/v), 14 days after incubation. The fermentation process was monitored by the spectrophotometric method to measure ethanol (Crowell & Ough, 1979). Both batches were immediately fortified to 18% vol with wine alcohol (Alcoholes del Sur, Córdoba, Spain; CE 200-578-6), centrifuged at 3 000 rpm at 4ºC for 10 min and stored in a cold chamber at 4ºC until analysis.

Accelerated ageing

The accelerated ageing experiment was conducted on 3 000 mL of each fermented must, which was distributed among six 1 000 mL flasks to obtain two batches consisting of six 500 mL samples each. Three flasks in each batch were supplied with American oak in the form of medium-toasted chips (Anatride Ibérica SL, Zaragoza, Spain) at a concentration of 1 g/L, and the other three with a 2 g/L concentration of identical chips. The flasks were then stoppered with hydrophobic cotton and allowed to stand in a thermostated room at 20°C for 30 days, with shaking by hand on a daily basis. After the experiment was finished, the wood chips were removed and the samples were stored in a cold chamber at 4°C until analysis.

Conventional analyses

The pH, total and volatile acidities and reducing sugars were determined according to EEC (1990) methods. Glycerine was quantified by direct injection of samples in a gas chromatograph HP 6890 GC System (Agilent Technologies, CA, USA) equipped with a capillary column with molten silica CP-WAX 57 CB (50 m x 0.25 mm x 0.4 µm thickness), and a FID was used as the detector according to Peinado *et al*. (2004).

Browning and colour evaluation

Browning of the samples was measured as absorbance at 420 nm. Colour was determined according to the recommendations of the International Commission on Illumination (CIE, 2004) with the illuminant D65 (daylight source) and 10º standard observer (perception of a human observer). The parameters calculated were a* (red/green values), b* (yellow/blue values), and L* (lightness). From the CIELab space, other psychophysical parameters were calculated, such as C* (chroma or saturation) and h (hue angle). All the respective measurements were carried out in triplicate in a Perkin-Elmer Lambda 25 model spectrophotometer (USA) using 10 mm quartz tray after filtering the samples through a HA-0.45 µm paper (Millipore).

Identification and quantification of aroma compounds

Each aroma compound was identified by means of its retention time, co-eluted with a standard solution of commercial product (Sigma Aldrich, Munich, Germany), and confirmed by mass spectrometry (Hewlett-Packard 5972 MSD, Agilent Technologies, CA, USA). Positive ion electron impact mass spectra were acquired in scan mode, with a range of m⁄z 39 to 300, and a scan rate of 1.6 scans/sec. For each compound the mass spectra were confirmed by comparison with the Wiley mass spectral library. The chromatographic column, injector and oven temperatures, carrier gas and its flow were the same as those used for the quantification, as described below.

For the quantification of the aroma compounds, samples of 100 mL were adjusted to pH 3.5, 150 mg of 2-octanol was added as an internal standard and then extracted with 100 mL of freon-11 (Sigma-Aldrich Química S.A., Madrid, Spain) in a continuous extractor for 24 hours. After concentration of the freon extracts to 0.2 mL in a Kuderna-Danish micro-concentrator, 3 µL were injected into the Hewlett-Packard 5890 series II chromatograph with an HP-INNOWax column of 60 m x 0.32 mm x 0.25 µm thickness (Agilent Technologies, CA, USA), equipped with a split/splitless injector and an FID detector. The oven temperature programme was as follows: 5 min at 45ºC, 1ºC/min up to 185ºC and 30 min at 185ºC. Injector and detector temperatures were 275ºC and 300ºC respectively. The carrier gas was helium at 70 kPa and was split 1:30. The quantification was made by using chromatographic response factors, calculated for each compound in relation to the internal standard, in standard solutions of commercial products (purity > 95%) supplied by Sigma Aldrich (Munich, Germany). The quantification was done in triplicate.

Odour descriptors of aroma compounds

For the determination of odour descriptors, a direct olfaction of the pure reference standards (Sigma Aldrich, Munich, Germany) was conducted on water solutions of each compound with a concentration slightly higher than its perception threshold (10%). The taste panel consisted of 20 trained judges of both sexes (12 female and 8 male) between the ages of 20 and 55 years (ISO 5496:1992) from the University of Cordoba. All judges were trained in preliminary sessions using reference standards taken from Sigma-Aldrich (Munich, Germany) and from "Le nez du vin" (Jean Lenoir, Provence, France) according to ISO 5496:1992. Thirteen judges of the above-mentioned panel had previous experience in the sensory evaluation of sherry wine. During the training, five standards were tasted per session and were discussed by the judges in terms of odour descriptors, and consensus on the terms was reached by eliminating those that were considered irrelevant or redundant. Later, five different odour samples were served at each session and there were 13 evaluation sessions. Samples were prepared 30 min before the test to allow time for the vapour pressure to reach equilibrium at ambient temperature. The odour substances (1 mL) were poured directly into the glass flasks containing a piece of cotton and were closed immediately. Evaluation was conducted in our laboratory in individual booths at room temperature (25ºC). The responses of the judges were compiled for all aroma compounds, and those odour descriptors cited by less than 15% of the panel were eliminated. The odour descriptors are listed in Table 1.

Sensory analysis of wines

The partially fermented musts treated with oak wood chips were subjected to a quantitative descriptive analysis (QDA) to establish their sensory profile (Stone *et al*., 2012). The tasting panel was composed of five experts (three men and two women, 40 to 50 years old), selected by the Quality Regulation Board of the Montilla-Moriles designation

TABLE 1
Odour descriptors, odorant terms (Wine Aroma Wheel) and perception thresholds (µg/L) of the aroma compounds determined in the sweet wines.

Compound	Odour descriptor	Odorant terms	Threshold
Ethyl acetate	Pineapple, varnish, anise	Tropical fruit, pungent, spicy	7 500 [a]
1,1-Diethoxyethane	Green fruit, liquorice	Tree fruit, spicy	1 000 [a]
Propyl acetate	Glue, Christmas sweet	Chemical, caramelised	65 000 [a]
2,3-Butanedione	Buttery	Caramelised	100 [a]
Ethyl propanoate	Apple	Tree fruit	5 000 [a]
Isobutyl acetate	Sweet, apple, banana	Caramelised, tree fruit, tropical fruit	6 140 [a]
2-Butanol	Vinous	Chemical	1 000 000 [a]
2,3-Pentanedione	Buttery	Caramelised	1 000 [b]
Butyl acetate	Ripe pear, glue	Tree fruit, chemical	4 600 [a]
Hexanal	Green	Fresh	350 [c]
Isobutanol	Alcohol, nail polish	Chemical, pungent	40 000 [a]
Isoamyl acetate	Banana	Tropical fruit	30 [a]
1-Butanol	Medicinal	Phenolic	820 000 [a]
Isoamyl alcohols	Alcohol, nail polish	Chemical, pungent	65 000 [a]
Ethyl hexanoate	Banana, green apple	Tropical fruit, tree fruit	5 [a]
Isoamyl butanoate	Banana	Tropical fruit	1 000 [c]
Hexyl acetate	Apple, banana	Tree fruit, tropical fruit	1 000 [b]
Octanal	Soapy, fatty, honey, grass	Chemical, oily, caramelised, fresh	640 [a]
Acetoin	Buttery, cream	Caramelised	30 000 [a]
Ethyl heptanoate	Sweet, strawberry, banana	Caramelised, berry, tropical fruit	10 000 [d]
3-Methylpentanol	Pungent, vinous, cacao, herbaceous	Pungent, chemical, caramelised, fresh	50 000 [a]
Ethyl lactate	Strawberry, raspberry, buttery	Berry, caramelised	100 000 [a]
1-Hexanol	Green, grass	Fresh	8 000 [a]
E-3-hexenol	Grass, resinous, cream	Fresh, resinous, caramelised	1 000 [c]
3-Etoxypropanol	Overripe pear	Tree fruit	50 000 [a]
E-2-hexenol	Green	Fresh	15 000 [c]
Furfural	Burned almond, floral	Burned, floral, marshmallow	15 000 [a]
Ethyl 3-hidroxybutanoate	Fresh, grape	Fresh, berry, floral	67 000 [a]
Benzaldehyde	Bitter almond, nutty, smoky	Nutty, burned	5 000 [a]
Isobutanoic acid	Rancid butter	Lactic	20 000 [a]
5-Methylfurfural	Bitter almond, spicy	Nutty, spicy	16 000 [a]
γ-Butyrolactone	Coconut, caramel	Tropical fruit, caramelized	100000 [a]
Butanoic acid	Rancid, cheese	Lactic	10000 [a]
Furfuryl alcohol	Medicinal	Phenolic	15000 [a]
Diethyl succinate	Lavender, overripe melon	Caramelized, floral, tropical fruit	100000 [a]
3-Methylbutanoic acid	Parmesan cheese, rancid	Lactic	3000 [a]
α-Terpineol	Lilac	Floral	38000 [a]
γ-Hexalactone	Coconut, almond liqueur, sweet	Tropical fruit, nutty, caramelised	359 000 [d]
Methionol	Cut hay, cooked potato	Fresh	500 [a]
Geranial	Citrus, sweet	Citrus, caramelised	1 000 [c]
Nerol	Herbaceous, lemon balm	Fresh, floral	10 000 [c]
γ-Heptalactone	Coconut, herbaceous, caramel	Tropical fruit, fresh, caramelised	1 000 [c]
2-Phenylethanol acetate	Rose, honey	Floral, caramelised	250 [a]
Hexanoic acid	Cheese	Lactic	3 000 [a]
Guaiacol	Smoky	Burned	20 [e]
Benzyl alcohol	Fruity, walnut	Tree fruit, nutty	900 000 [a]
E-oak lactone	Coconut, burned wood, vanilla	Tropical fruit, burned, spicy	122 [a]

TABLE 1 (CONTINUED)

Compound	Odour descriptor	Odorant terms	Threshold
2-Phenylethanol	Rose, honey	Floral, caramelised	10 000 [a]
Z-oak lactone	Coconut, burned wood, vanilla	Tropical fruit, burned, spicy	35 [a]
Methyleugenol	Clove	Spicy	10 000 [d]
4-Ethylguaiacol	Smoky, clove	Burned, spicy	46 [a]
Diethyl malate	Peach, prune	Tree fruit	760 000 [a]
Pantolactone	Liquorice, smoky	Spicy, burned	500 000 [a]
Octanoic acid	Oily, rancid	Oily, lactic	8 800 [a]
2-Phenylethanol hexanoate	Overripe banana, sweet	Tropical fruit, caramelised	50 000 [c]
Eugenol	Clove	Spicy	5 [a]
γ-Decalactone	Peach	Tree fruit	1 000 [a]
4-Ethylphenol	Spicy	Spicy	140 000 [a]
Syringol	Smoky	Burned	1 700 [e]
Decanoic acid	Rancid, waxy	Lactic, oily	15 000 [a]
Farnesol	Fruity, balsamic, floral, clove	Tree fruit, fresh, floral, spicy	72 000 [c]
Isoeugenol	Clove, burned wood, sweet	Spicy, burned, caramelised	6 [c]
Monoethyl succinate	Burned caramel, coffee	Burned	1 000 000 [c]
Vanillin	Vanilla	Spicy	65 [e]
2,3-Butanediol	Sweet, creamy, butter	Caramelised	668 000 [a]

a = from Zea *et al*. (2007)
b = from Chaves *et al*. (2007)
c = determined by the author in alcoholic solution; data not published. Five solutions of ascending concentration of these compounds were used. Starting from the lowest concentration solution, the judges indicated odorant sensations different to that perceived in the control (distilled water), according to the ISO 5495:1983.
d = from Moreno (2005)
e = from Moyano *et al*. (2012)

of origin (southern Spain). These judges are professional oenologists who know the characteristics of typical wines produced in the region very well, and therefore are sensitive to even small differences.

The tasting session was preceded by another one to reach consensus on the sensory attributes used in the sensory analysis of the sweet musts studied (fermentative aroma, woody, astringency, sweetness, acidity and colour). The performance of the expert tasters was accepted as highly reliable and consistent according to the abovementioned Quality Regulation Board. In three consecutive sessions, the tasters examined the seven samples of wine three times, with the ones being presented in random order each time. Their sensory attributes, sensory balance and global impression were scored using the scale method in accordance with ISO 4121:1987. The scale direction goes from left to right with increasing intensities: 1 (imperceptible), 2 (weak), 3 (moderate), 4 (strong) and 5 (excessive) (Stone *et al*., 2012). Balance and global impression were measured on a five-point hedonic scale, from 1 (unacceptable) to 5 (highly acceptable), with three intermediate points. The results were given as mean ± standard deviation. The means of the sample scores were shown in a "spider web" graph.

Samples were stored in a refrigerator and withdrawn one hour before the sensory test in order to facilitate the adjustment in temperature to that of the tasting room. Evaluation was carried out in a thermostated room with individual booths at 20°C. Twenty millilitres of sample were used in standardised wine glasses (ISO 3591:1992). These were marked with a code and covered to avoid any loss of organoleptic properties. The sequence of sample presentation went from wines with 4% (v/v) to those with 8% (v/v) alcohol content. All the samples were evaluated in a single session, one at a time, with a wait of 3 min between samples.

Statistical treatments

ANOVA (LSD 95% level) was performed on the triplicates of winemaking variables studied. The OAVs for odorant term and the results of the sensory analysis of the wines were calculated as mean ± standard deviation of three samples for each wine type. To find which variables contributed most to the difference in the aroma profiles of the wines, a PCA was performed on the triplicates of OAVs of odorant terms. The values of the variables were standardised by subtracting their means and dividing by their standard deviations. Results were validated by full internal cross-validation.

Software

Statgraphics™ (Version 5.0). STSC Inc, Rockville, MD, USA.

RESULTS AND DISCUSSION

Winemaking variables

Table 2 shows the conventional oenological and colour parameters of the musts. The ANOVA shows that wines with different alcohol levels obtained by fermentation also

TABLE 2

Means and standard deviations (n = 3) for conventional oenological and colour parameters of the control must (unfermented), and the musts partially fermented to 4% (v/v) or 8% (v/v) of ethanol and to which oak chips subsequently were added (1 g/L or 2 g/L) for 30 days.

	Control	Partially fermented		Partially fermented + oak chips			
		4% (v/v)	8% (v/v)	4% (v/v)		8% (v/v)	
				1 g/L	2 g/L	1 g/L	2 g/L
pH[#]	3.93 ± 0.01	3.64 ± 0.01	3.61 ± 0.04	3.53 ± 0.01	3.53 ± 0.02	3.72 ± 0.01	3.74 ± 0.01
Total acidity (meq/L)[#]	51.0 ± 0.3	67.8 ± 0.3	86.9 ± 0.1	71 ± 1	71 ± 1	95 ± 2	94.5 ± 0.8
Volatile acidity (meq/L)[#]	7.9 ± 0.3	18.0 ± 0.3	39.3 ± 0.5	19.8 ± 0.1	19.8 ± 0.1	39.5 ± 0.7	39.5 ± 0.7
Reducing sugars (g/L)[#]	450 ± 2	345 ± 5	301 ± 4	349 ± 1	348 ± 3	302 ± 4	301 ± 4
Glycerine (g/L)[#]	5.2 ± 0.4	18.5 ± 0.9	25 ± 1	19 ± 1	18.7 ± 0.9	25 ± 1	25 ± 1
Absorbance at 420 nm	0.640 ± 0.002	0.560 ± 0.003	0.555 ± 0.003	1.018 ± 0.001	1.065 ± 0.004	1.034 ± 0.006	1.084 ± 0.002
a*	1.62 ± 0.09	0.07 ± 0.02	-0.16 ± 0.02	6.8 ± 0.2	6.76 ± 0.01	7.2 ± 0.1	7.30 ± 0.03
b*	38.14 ± 0.04	33.6 ± 0.7	33.6 ± 0.2	52.22 ± 0.05	53.41 ± 0.01	50.56 ± 0.06	51.72 ± 0.04
L*	89.63 ± 0.06	91.07 ± 0.06	91.53 ± 0.06	81.30 ± 0.01	81.10 ± 0.01	80.63 ± 0.06	80.40 ± 0.01
C^*_{ab}	38.17 ± 0.03	33.6 ± 0.7	33.6 ± 0.2	52.66 ± 0.04	53.84 ± 0.01	51.08 ± 0.08	52.23 ± 0.04
h_{ab}	87.6 ± 0.1	89.8 ± 0.1	90.21 ± 0.09	82.6 ± 0.2	82.78 ± 0.01	81.8 ± 0.1	81.97 ± 0.04

[#] Values different for the wines fermented to 4% and 8% (v/v) ethanol at 95% ANOVA level

presented values of pH, total and volatile acidity, reducing sugar and glycerine that were different in each case at the LSD 95% level. This shows that the fermentations progressed successfully. As can be seen in Table 2, alcoholic fermentation lowered the pH, and increased the total and volatile acidity markedly. This suggests that, as previously found by other authors (Pigeau *et al*., 2002; Erasmus *et al*., 2004; Malacrino *et al*., 2005), the yeasts produced increased amounts of acetic acid in response to the osmotic stress caused by high sugar levels. In general, high levels of volatile acidity are not considered positive for quality of wine. However, for the sweet wines, the increased production of acetic acid countered the overall sweetness in the end-product through its contribution to total acidity (Pigeau *et al*., 2007).

Obviously, the levels of reducing sugars decreased as a result of fermentation, especially in the musts fermented to 8% (v/v) ethanol (about 300 g/L) and irrespective of the ageing procedure. Also, glycerine increased markedly, with average concentrations close to 18 and 25 g/L in the musts fermented to 4 and 8% respectively, even after ageing. These high glycerine levels are the result of the osmotic stress of the yeasts. Glycerine has been proposed by several authors to be one of the most compatible metabolites to equilibrate the osmotic pressure in the cell with the use of glycerine-3-phosphate dehydrogenase.

Fermentation decreased the colour-related parameters, A_{420}, a^*, $b^*$and $C_{ab}{}^*$, and increased L^* and h_{ab}; the resulting sweet wines were thus less brown and more pale. This may have been the result of yeasts adsorbing some coloured compounds during fermentation (Mérida *et al*., 2005) and/or of low β-glucosidase activity in the yeasts. On the other hand, accelerated ageing increased A_{420}, a^*, b^* and $C_{ab}{}^*$, and decreased L^* and h_{ab}, with the resulting wines exhibiting increased brown, dark, reddish and yellowish hues, especially at the higher wood chip concentration. This dissimilar behaviour may have been the result of oxidation, condensation and/or polymerisation reactions, and also of the extraction of mainly phenolic compounds from the chips.

Aroma compounds

Table 3 lists the contents of the compounds studied in the sweet wines. The odour activity value (OAV) for each compound was calculated by dividing its concentration in the samples by the concentration corresponding to its odour threshold (Table 1). Over the past few years, numerous authors have proposed an approximation of the importance of a flavour compound in the wine based on the OAV. However, quantifying the perceived intensity of odorants and their contribution to the overall aroma is more complex.

Based on the perception thresholds shown in Table 1, there were only 12 active odorants (OAV > 1) in at least one sample. 2,3-Butanedione (diacetyl) is one of the typical odorants in musts from dried Pedro Ximenez grapes (Ruiz *et al*., 2010), which it endows with buttery notes. The musts fermented to 4% (v/v) ethanol exhibited higher OAVs (approximately 29.5), which suggests that the point at which the fermentation process is stopped influences the final concentrations of this compound. It is most likely that the reductive conditions prevailing at the end of alcoholic fermentation facilitate the reduction of 2,3-butanedione to 2,3-butane-

TABLE 3
Means and standard deviations (n = 3) for the aroma compounds (µg/L) detected in the control must (unfermented), and the musts partially fermented to 4% (v/v) or 8% (v/v) of ethanol to which oak chips subsequently were added (1 g/L or 2 g/L) for 30 days.

Compound	Control	Partially fermented		Partially fermented + oak chips			
		4% (v/v)	8% (v/v)	4% (v/v)		8% (v/v)	
				1 g/L	2 g/L	1 g/L	2 g/L
Ethyl acetate	24 095 ± 572	51 213 ± 8 870	110 324 ± 10 015	38 983 ± 1 277	41 863 ± 2 340	106 666 ± 7 637	113 333 ± 5 773
1,1-Diethoxyethane	1 274 ± 26	2 123 ± 178	4 595 ± 415	1 624 ± 53	1 744 ± 98	4 610 ± 212	4 795 ± 193
Propyl acetate	8.0 ± 0.7	8 ± 1	12 ± 2	11 ± 3	15 ± 4	16 ± 2	14 ± 1
2,3-Butanedione	1 215 ± 153	2 952 ± 100	972 ± 50	4 873 ± 221	5 068 ± 540	1 531 ± 112	1 656 ± 230
Ethyl propanoate	20 ± 3	30 ± 6	45 ± 5	28 ± 2	24 ± 5	40 ± 2	43 ± 6
Isobutyl acetate	40 ± 5	65 ± 9	94 ± 18	nd	nd	nd	nd
2-Butanol	9 ± 2	26 ± 3	7.4 ± 0.6	nd	nd	nd	nd
2,3-Pentanedione	20 ± 5	398 ± 28	47 ± 1	405 ± 30	435 ± 52	46 ± 4	47 ± 8
Butyl acetate	nd	92 ± 14	128 ± 4	154 ± 4	127 ± 9	122 ± 5	134 ± 6
Hexanal	41 ± 7	29 ± 2	6.0 ± 0.7	45 ± 5	35 ± 4	9 ± 1	23 ± 4
Isobutanol	11 396 ± 2 582	30 483 ± 2 659	40 896 ± 2 517	25 170 ± 3 110	28 293 ± 3 827	36 210 ± 3 200	38 330 ± 1 703
Isoamyl acetate	52 ± 6	158 ± 10	208 ± 13	183 ± 10	191 ± 10	286 ± 18	295 ± 26
1-Butanol	461 ± 42	1 067 ± 52	1 509 ± 74	1 768 ± 60	1 121 ± 41	1 110 ± 79	1 246 ± 256
Isoamyl alcohols	18 204 ± 3 273	102 473 ± 7 322	125 593 ± 6 630	89 283 ± 6 315	97 340 ± 3 440	146 721 ± 2 944	132 378 ± 3 951
Ethyl hexanoate	nd	86 ± 8	128 ± 9	101 ± 10	101 ± 19	144 ± 13	147 ± 12
Isoamyl butanoate	87 ± 5	89 ± 4	80 ± 13	82 ± 9	72 ± 4	40 ± 6	44 ± 5
Hexyl acetate	32 ± 5	15 ± 1	35 ± 2	23 ± 2	25 ± 3	14 ± 4	16 ± 2
Octanal	53 ± 4	72 ± 16	49 ± 6	127 ± 5	91 ± 14	71 ± 8	46 ± 2
Acetoin	247 879 ± 18 859	1 228 524 ± 216 045	151 285 ± 6 803	1 415 982 ± 96 303	1 422 045 ± 86 768	253 803 ± 21 226	229 211 ± 16 552
Ethyl heptanoate	46±5	40±5	44±2	29±7	18 ± 2	28 ± 9	16 ± 3
3-Methylpentanol	nd	27±3	24±5	24±7	22±5	28±2	30±2
Ethyl lactate	1225±131	1758±129	4073±460	5239±351	5016±226	14714±1114	14041±1519
1-Hexanol	18±3	28±7	28±6	32±6	31±6	45±6	40±6
E-3-hexenol	5.8 ± 0.7	nd	16 ± 2	nd	nd	9 ± 1	7.6 ± 0.9
3-Ethoxypropanol	9 827 ± 471	11 665 ± 414	12 793 ± 1 951	12 349 ± 194	11 091 ± 544	17 374 ± 2 005	14 382 ± 1 713
E-2-hexenol	7 ± 1	24 ± 2	16 ± 1	33 ± 4	44 ± 5	32 ± 3	22 ± 2
Furfural	40 ± 1	65 ± 7	110 ± 20	1 150 ± 200	1 840 ± 230	1 035 ± 210	1 576 ± 223
Ethyl 3-hidroxybutanoate	46 ± 8	48 ± 10	62 ± 4	27 ± 4	21 ± 3	45 ± 8	48 ± 8
Benzaldehyde	24 ± 2	50 ± 10	17 ± 1	26 ± 2	22 ± 2	18 ± 2	13 ± 4
Isobutanoic acid	820 ± 100	12 555 ± 953	19 593 ± 616	11 019 ± 780	11 834 ± 1 470	18 263 ± 2 344	15 712 ± 2 003
5-Methylfurfural	35 ± 4	45 ± 5	43 ± 5	1 020 ± 83	2 130 ± 106	1 062 ± 175	2 400 ± 360
γ-Butyrolactone	2 436 ± 229	2 596 ± 343	5 821 ± 818	3 808 ± 255	4 381 ± 114	10 185 ± 712	12 200 ± 704
Butanoic acid	97 ± 14	260 ± 15	152 ± 36	137 ± 24	85 ± 14	86 ± 6	70 ± 5

TABLE 3 (CONTINUED)

Compound	Control	Partially fermented		Partially fermented + oak chips			
		4% (v/v)	8% (v/v)	4% (v/v)		8% (v/v)	
				1 g/L	2 g/L	1 g/L	2 g/L
Furfuryl alcohol	4.7 ± 0.7	nd	nd	nd	nd	nd	nd
Diethyl succinate	183 ± 19	198 ± 25	501 ± 21	289 ± 17	268 ± 24	809 ± 94	733 ± 54
3-Methylbutanoic acid	130 ± 11	2 207 ± 92	2 495 ± 150	1 968 ± 162	1 255 ± 121	2 208 ± 346	1 557 ± 74
α-Terpineol	12 ± 1	15 ± 2	14 ± 2	9.1 ± 1	10 ± 1	15 ± 1	16 ± 3
γ-Hexalactone	19 ± 4	8 ± 1	2.7 ± 0.7	8.3 ± 0.6	7 ± 1	8 ± 1	10 ± 2
Methionol	67 ± 2	65 ± 10	70 ± 9	nd	nd	nd	nd
Geranial	56 ± 7	34 ± 6	20 ± 1	13.0 ± 0.2	14 ± 2	20 ± 4	15 ± 1
Nerol	13 ± 2	nd	nd	nd	nd	nd	nd
γ-Heptalactone	66 ± 7	67 ± 12	86 ± 27	36 ± 3	43 ± 7	108 ± 26	120 ± 28
2-Phenylethanol acetate	20±2	21±3	29±3	34±4	28±3	45±5	32±7
Hexanoic acid	15±2	64±4	82±7	75±7	68±8	70±20	72±8
Guaiacol	0	0	0	6±1	9±2	5.3±0.6	9.4±0.8
Benzyl alcohol	86±10	152±18	159±23	173±17	143±28	219±11	188±15
E-oak lactone	nd	nd	nd	3.7±0.7	8±1	4.4±0.5	9±2
2-phenylethanol	11 458 ± 1 323	31 036 ± 4 703	69 932 ± 3 229	35 574 ± 4 911	26 260 ± 2 789	78 891 ± 5 066	75 253 ± 4 949
Z-oak lactone	nd	nd	nd	11 ± 1	19 ± 2	13 ± 1	28 ± 6
Methyleugenol	nd	nd	nd	50 ± 8	68 ± 5	44 ± 4	53 ± 4
4-Ethylguaiacol	nd	nd	nd	11 ± 1	19 ± 3	20 ± 2	33 ± 2
Diethyl malate	61 ± 6	129 ± 14	248 ± 23	168 ± 4	183 ± 8	531 ± 51	324 ± 17
Pantolactone	7 ± 1	18 ± 2	20 ± 2	20.8 ± 0.7	17 ± 3	65 ± 4	61 ± 12
Octanoic acid	2.9 ± 0.7	84 ± 8	99 ± 14	62 ± 11	49 ± 5	200 ± 16	183 ± 9
2-Phenylethanol hexanoate	7 ± 1	15 ± 2	11 ± 1	14 ± 1	15 ± 1	18 ± 1	10.9 ± 0.8
Eugenol	nd	nd	nd	16 ± 3	24 ± 3	15 ± 1	23 ± 3
γ-Decalactone	20 ± 2	25 ± 3	27 ± 5	32 ± 4	36 ± 2	39 ± 3	33 ± 2
4-Ethylphenol	nd	nd	nd	11 ± 1	10 ± 1	30 ± 2	29 ± 2
Syringol	nd	nd	nd	58 ± 6	69 ± 3	43 ± 3	56 ± 7
Decanoic acid	nd	107 ± 8	117 ± 14	140 ± 10	160 ± 15	175 ± 15	185 ± 20
Farnesol	nd	7.5 ± 0.5	8 ± 2	18 ± 2	13 ± 2	19 ± 3	21.2 ± 0.7
Isoeugenol	nd	nd	nd	1.9 ± 0.3	2.4 ± 0.4	4.4 ± 0.6	4.3 ± 0.9
Monoethyl succinate	nd	51 ± 7	36 ± 6	70 ± 12	53 ± 7	47 ± 1	29 ± 2
Vanillin	nd	nd	nd	51 ± 10	180 ± 30	48 ± 5	185 ± 20
2,3-Butanediol[a]	0.25 ± 0.03	1.49 ± 0.08	5.03 ± 0.11	1.68 ± 0.08	1.75 ± 0.05	5.05 ± 0.18	4.9 ± 0.7

nd = not detected

a = the concentration is expressed as g/L

diol, thereby diminishing its impact on the aroma (Martineau *et al*., 1995). The odorant activity of 2,3-butanediol increased markedly during fermentation and the compound reached its highest OAVs (≈7.5) in the must fermented to 8% (v/v) ethanol. This compound is associated with sweet, creamy, buttery notes.

The higher alcohols isobutanol, isoamyl and 2-phenylethanol only reached their perception thresholds at the end of fermentation; however, they exhibited near-unity OAVs, so it is reasonable to assume that they can hardly have contributed to the overall aroma of the sweet wines studied.

Ethyl hexanoate and isoamyl and ethyl acetates exhibited the highest odorant activity in the partially fermented musts and thus were major contributors to their aroma profile, which they enriched with fruity, anise and varnish notes. Ruiz *et al*. (2010) previously found ethyl acetate to increase during the drying of Pedro Ximenez grapes, both in the sun and in chambers, through the effect of its involvement in anaerobic metabolism in the grape berries during drying. Also, this compound has been deemed a useful marker for metabolism in drying grape berries (Chkaiban *et al*., 2007).

1,1-Diethoxyethane (diacetal) slightly surpassed its perception threshold in the unfermented must, and its concentration increased, also slightly, from the effect of fermentation. The most important compound in odorant terms was acetoin (3-hydroxy-2-butanone), with OAVs of about 40 in the musts fermented to 4% (v/v) ethanol. Like 2,3-butanedione, acetoin is a typical component of musts from Pedro Ximenez cv. grapes dried in the sun or in a chamber, and increases during drying. This compound behaved similarly to 2,3-butanedione, but differed even more markedly between the two types of fermented must. Although acetoin is a typical product of alcoholic fermentation, it can also come from other sources, including yeasts and bacteria, or malolactic bacteria. However, sweet dessert wines fortified halfway through fermentation were found to contain more acetoin than identical wines allowed to ferment completely. This has been ascribed to the high levels of acetoin present in the middle of the process, which subsequently decreases from the effect of its conversion to 2,3-butanediol (Herraiz, 1990).

1-Butanol, ethyl lactate, 3-ethoxypropanol, isobutanoic acid, γ-butyrolactone and 3-methylbutanoic acid were present at high concentrations as a result of the alcoholic fermentation, but exhibited no odorant activity in most of the wines studied. None of the other compounds studied reached its perception threshold.

As can be seen from Table 3, the compounds exhibiting odorant activity at the end of partial fermentation of the musts remained active during the 30 days of accelerated aging. This was particularly so with 2,3-butanedione and isoamyl acetate, which were the most interesting compounds in terms of OAVs at this stage, irrespective of the wood chip concentration used; both compounds increased in the two types of fermented must. 2,3-Butanedione also increased during the oxidative ageing of Pedro Ximenez sweet musts (Chaves *et al*., 2007), probably as a result of the oxidation of acetoin or the gradual decrease in the levels of SO_2 because of its addition to the carbonyl groups in diacetyl. The reversible and exothermal nature of this reaction could change the "buttery" flavour of wines (Bartowsky & Henschke, 2004), an effect that can also be observed in sweet wines. The increase in isoamyl acetate may be related to the high values of acetic acid in the wines in the presence of oak chips, measured as volatile acidity (Table 2).

Among the compounds not present in the fermented musts but extracted from the wood chips, only eugenol and vanillin surpassed their perception threshold, and only in a few samples. In this sense, vanillin was only active in the samples treated with a 2 g/L concentration of oak chips, with OAV ≈ 3 irrespective of the alcohol content reached by the wines. This compound is one of the most phenolic aldehydes in wine and is responsible for the typical vanilla notes of wines aged in wood casks (Singleton, 1995). Most phenol aldehydes come from the wood and are present at negligible concentrations in the base wine. Therefore, drying and toasting the wood used to age wine influences the extent to which these aldehydes (and, especially, vanillin) are extracted from it. Eugenol was active in the sweet wines treated with a 1 or 2 g/L concentration of oak chips, which imparted a typical clove aroma.

Although furfural and 5-methylfurfural form during the grape-drying process (Ruiz *et al*., 2010), oxidation and the presence of wood chips in the medium increased their contents markedly, albeit below their perception thresholds. However, these compounds might be useful as markers for the ageing process, since their levels are highly correlated with the ageing time (Camara *et al*., 2006). The (E) and (Z) isomers or oak lactone (β-methyl-γ-octalactone) were only detected in the samples treated with oak chips; however, both exhibited OAV < 1. Other volatile phenols, including guaiacol, 4-ethylguaiacol, 4-ethylphenol, syringol and isoeugenol, exhibited increased contents after the accelerated ageing of the wines.

Odorant terms

To compare the aroma profiles of the wines studied by considering a small number of variables, we used the OAV for the compound grouped into nine odorant terms according to their similar odour descriptors (caramelised, tropical fruit, tree fruit, spicy, pungent floral, chemical, toasted, and lactic). The remaining terms listed in Table 1 are not significant. The addition of the OAVs of the compounds to each term cannot be interpreted as an arithmetical addition of odorant sensations. Several authors have used odorant terms and aromatic series to establish aroma profiles for musts and wines from different winemaking processes (Zea *et al*., 2007; Ruiz *et al*., 2010; Gómez García-Carpintero *et al*., 2012; López de Lerma *et al*., 2012). In any case, the proposed method is valid for comparing wines of the same type (sweet wines in this work), because the odorant terms always comprise the same compounds. However, this method of studying the aroma profile has the advantage that it strongly reduces the number of variables to be interpreted, preserving their relative importance according to the OAVs of the compounds assembled.

Figure 2 shows the aroma fingerprint of the samples as obtained from the components with OAV > 1. As can be seen, the profile was altered considerably by the fermentation process. Overall, the musts fermented to 8% (v/v) ethanol exhibited higher OAVs; and the caramelised term had a

significantly higher OAV in the musts fermented to 4% (v/v) ethanol. After the addition of oak chips, the terms caramelised and also, to a lesser extent, spicy, exhibited an increase in the OAVs in both types of wines, as did the tropical fruit term in those wines with the higher alcohol content. Also, ageing introduced the term toasted, which was absent from the samples to which no wood chips had been added.

A principal component analysis (PCA) on the OAVs of the odorant terms considered was conducted in order to identify those with the greatest influence on the aroma profile of sweet wines (Fig. 3). The first two principle components (PCs) jointly accounted for 87% of the overall variance. PC1 explained 72% of the variance and encompassed the tropical fruit, tree fruit, pungent and floral terms, which afforded discrimination according to the alcohol content reached by the samples. The musts fermented to 8% (v/v) ethanol aged with oak chips were slightly different from those without chips. Since these wines exhibited the highest scores in this

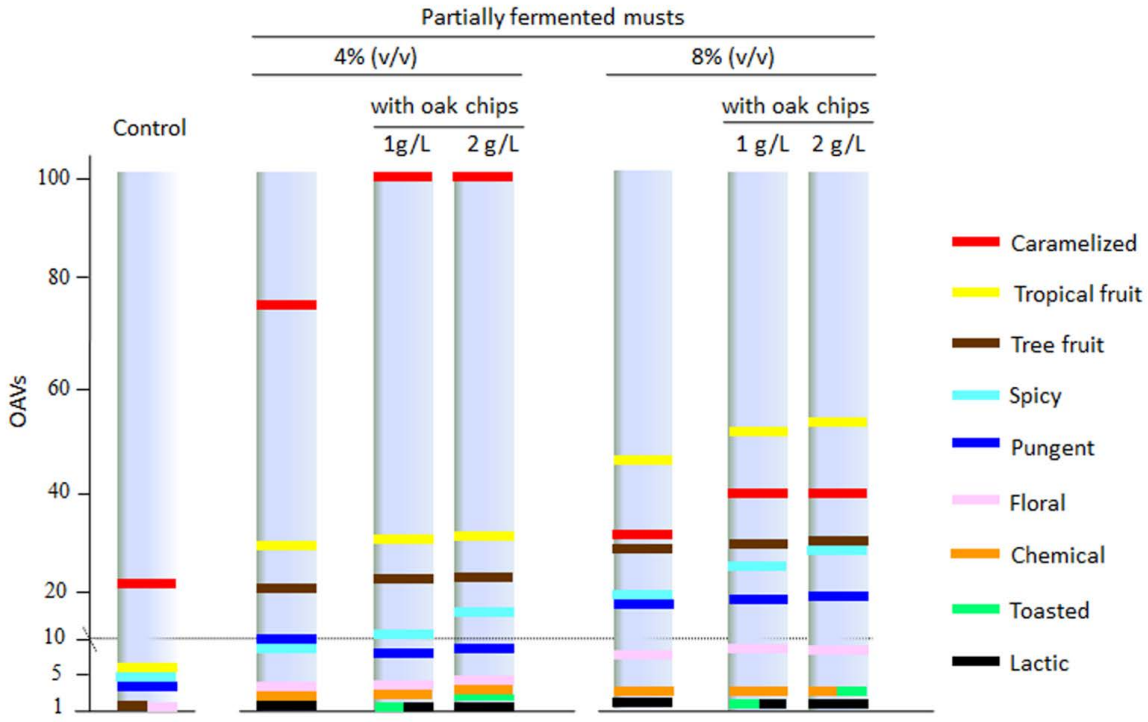

FIGURE 2

Aroma fingerprint of the control must (unfermented), and the musts partially fermented to 4% (v/v) or 8% (v/v) of ethanol and to which oak chips subsequently were added (1 g/L or 2 g/L) for 30 days.

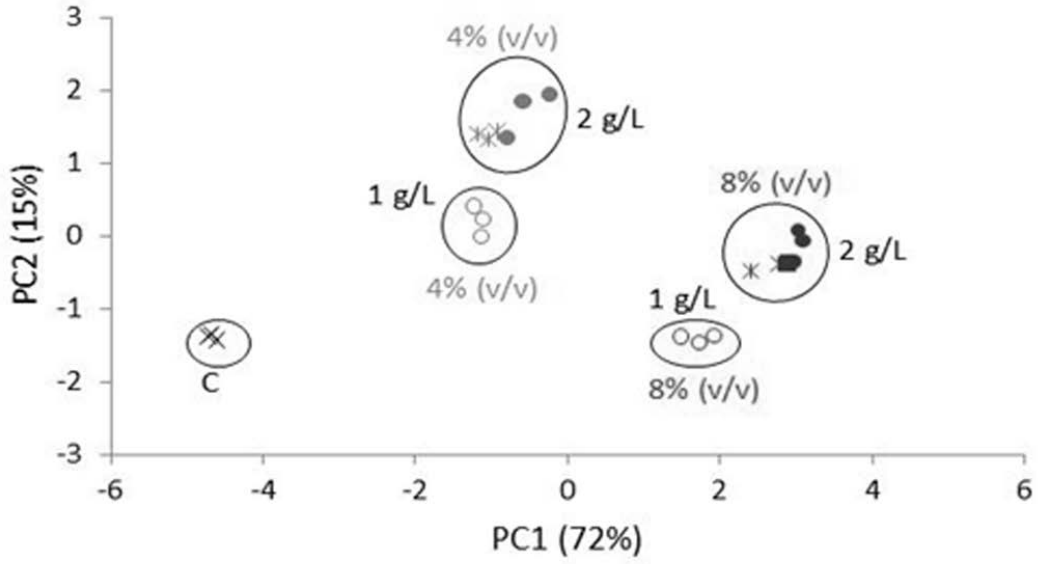

FIGURE 3

Principal component analysis carried out on the OAVs of the odorant terms of the control must (unfermented, C), and the musts partially fermented to 4% (v/v) or 8% (v/v) of ethanol and to which oak chips subsequently were added (1 g/L or 2 g/L) for 30 days.

component, the tropical fruit, tree fruit, pungent and floral terms showed the highest OAVs, distinguishing them even more clearly from the unfermented musts. Thus, the musts partially fermented to 8% (v/v) ethanol displayed a more intense aroma than the unfermented musts, which were reminiscent of the aroma of commercial sweet wines from raisins. PC2 explained 15% of the variance and encompassed the terms caramelised and toasted. It discriminated between the musts according to whether they were subjected to accelerated ageing, with those fermented to 4% (v/v) ethanol exhibiting the highest scores.

Sensory analysis

To evaluate the different conditions of the winemaking process used in sensorial terms, the sweet wines obtained were subjected to sensory analysis. Using the opinion of the judges it was possible to estimate the acceptability of sweet wines for the consumer and compare the products with other typical wines from the Montilla-Moriles region.

Fig. 4 shows the primary differences established by the sensory analysis of the partially fermented musts, namely differences in acidity, sweetness, balance and global impression. The musts fermented to 4% (v/v) ethanol were judged sweeter and less acidic than the others. The tasters distinguished between the musts fermented to identical ethanol content but treated with different concentrations of oak chips; thus, the musts fermented in the presence of a 2 g/L concentration were better scored for attributes such as woody and astringent. These results are somehow related to the balance and global impression scores, which were higher for the musts fermented to 8% (v/v) ethanol and aged in the presence of a 2 g/L concentration of oak chips, probably as a result of their reduced sweetness/acidity ratio. This, in combination with the perception of woody and astringent notes, led to higher global impression scores for these sweet wines.

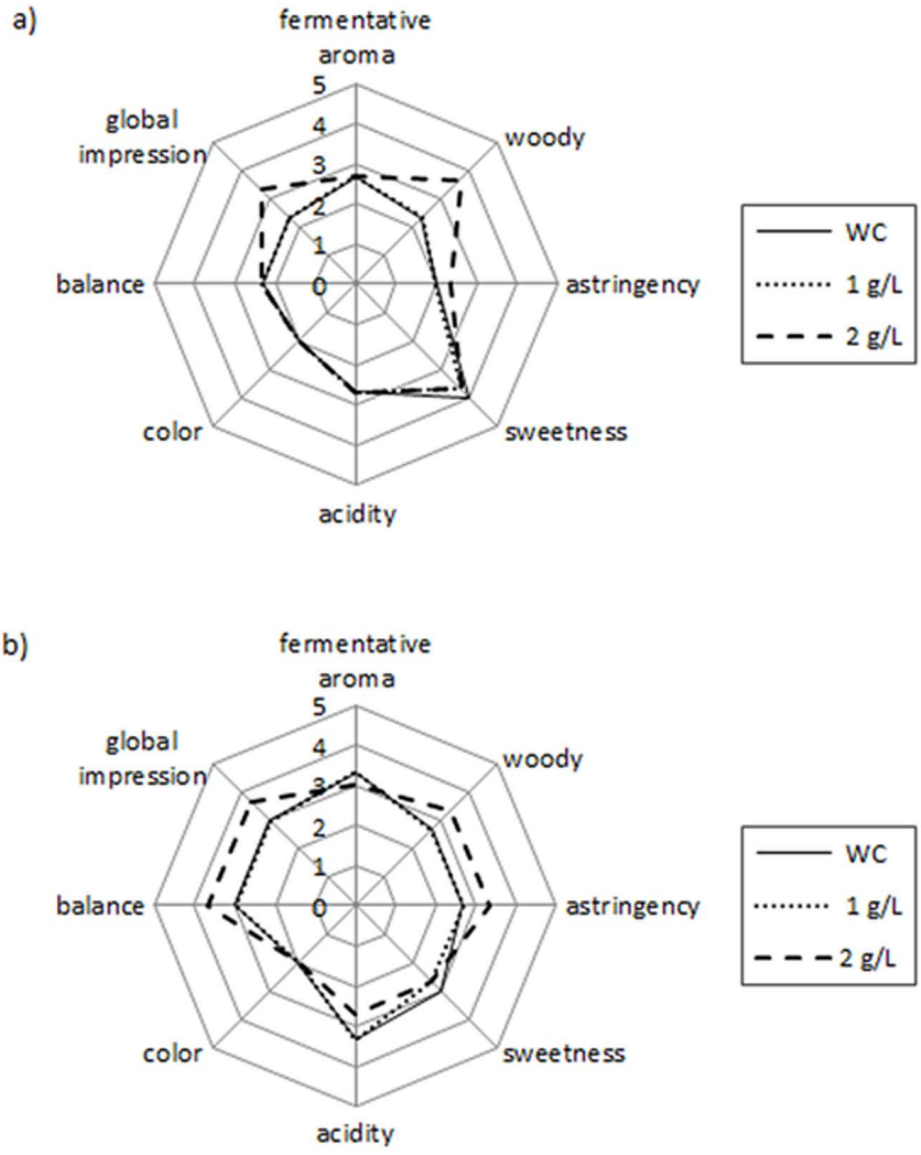

FIGURE 4

Spider web graph for the musts partially fermented to a) 4% (v/v) or b) 8% (v/v) of ethanol and to which oak chips subsequently were added (1 g/L or 2 g/L) for 30 days and without chips (WC).

CONCLUSIONS

The oenological parameters (Table 2) demonstrate that the partial fermentation of musts has developed normally. Furthermore, these parameters differed markedly between unfermented and partially fermented musts. The results reveal that fermentation reduced brownness, while ageing increased it. In this study, the OAVs of the compounds were grouped into nine odorant terms to compare the aroma profile of wines; this provided a simple and practical method by reducing the number of variables representing the aroma fingerprint of the samples. The tropical fruit term had higher OAVs in the musts fermented to 8% (v/v) ethanol; also, the presence of wood chips in the medium introduced the toasted term. The caramelised term showed the highest OAVs in the musts fermented to 4% (v/v) ethanol. The opinion of the judges was that the musts fermented to 8% (v/v) ethanol and aged in the presence of wood chips at a concentration of 2 g/L received the best balance and global impression scores, probably as a result of their low sweetness/acidity ratio, their woody and astringent notes and the increased levels of glycerine. Partial fermentation of the musts from chamber-dried grapes and subsequent ageing with oak chips provided a viable procedure to obtain high-quality sweet wines from raisin, and thus to achieve a diversification to meet market demands.

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