Chemical content and sensory changes of *Oloroso* Sherry wine when aged with four different wood types

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ARTICLE INFO

ABSTRACT

Oloroso Sherry Wine is a fortified Sherry wine obtained by oxidation and ageing in American oak barrels of 500 L–600 L. In this work, the study of the suitability of other types of woods for the ageing of these wines was carried out. To compare the characteristics of the alternative woods, an *oloroso* wine was aged in four groups of 16 L barrels made of French oak, Spanish oak, chestnut, as well as American oak as control, with intense and medium toasting. Phenolic and furanic compounds, organic acids, volatile compounds, color characteristics, total polyphenol index and sensory analysis of wines aged for two months were analyzed. The results confirmed that the aged samples could be differentiated on the basis of their chemical composition, and that the use of alternative woods to age *oloroso* Sherry wines, and the level of wood toasting, had the potential to provide products with specific differences to the traditional aged in American oak. Furthermore, the organoleptic characteristics of these alternative wines were valued above a standard Sherry wine.

1. Introduction

Sherries are all fortified wines aged in American oak casks of 500 L–600 L by the traditional dynamic system called *Solera y Criaderas* under the Designation of Origin *Jerez-Xérès-Sherry* (southwest of Spain). *Oloroso* Sherry wine is a type of Sherry obtained by oxidative ageing: young white wines are oxidized by transferring them from one cask to another at regular intervals and this process forces air to rush into (BOJA, 2013). The chemical composition and organoleptic characteristics of barrel-aged wines are affected by a large number of factors related to the ageing process. Among these factors are those related to the nature of the barrel wood (type of wood, origin of the wood, etc.), with the characteristics of the process of making the barrel (intensity and duration of the wood toasting process, size of the barrel, etc.), and with the ageing process itself carried out (ageing time, filling volume of the barrel, previous preparation operations of the barrel, etc.) (Canas, 2017).

Wine ageing in different woods has been proved to produce significant differences in volatile compounds (Herrera et al., 2020; Pérez-Prieto, López-Roca, Martínez-Cutillas, Pardo Mínguez, &

Gómez-Plaza, 2002), or polyphenols (Alañón et al., 2013; Fernández de Simón et al., 2014; García-Moreno et al., 2020) in diverse kinds of wine. The most widely used botanical species to make casks are the American oak (*Quercus alba*) and the French oak (*Quercus petraea, Quercus robur*). Nonetheless, there are species such as cherry, acacia, ash or chestnut whose suitability for wine ageing has been tested (De Rosso, Panighel, Dalla Vedova, Stella, & Flamini, 2009).

In recent years, Spanish coopers and producers have started to offer Spanish oak (*Quercus pyrenaica*) as a low cost alternative to French oak. Several studies were carried out to characterize the phenolic composition of this species (Alañón et al., 2011; Cadahía, Muñoz, De Simón, & García-Vallejo, 2001; Castro-Vázquez, Alañón, Ricardo-da-Silva, Pérez-Coello, & Laureano, 2013) and wines aged therein. Aroma compounds have also been profiled both in wood (Cadahia, de Simon, Vallejo, Sanz, & Broto, 2007) and wine (Cadahia et al., 2007; Herrera et al., 2020). The main conclusions of these studies confirm the suitability of Spanish oak to age wine as it provides intermediate or similar organoleptic features to French and American oaks (Fernández De Simón, Cadahía, & Jalocha, 2003).

Another traditional wood for cooperage in the Iberian Peninsula is

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chestnut (*Castanea sativa*). Recently, researchers and winemakers have focused their interest on the suitability of chestnut for ageing. The characterization of chips of such wood demonstrated that gallic and ellagic acids are heavily released by this wood when wine is stored in it (Garcia, Soares, Dias, Freitas, & Cabrita, 2012). The volatile compounds released by chestnut to red wine are positively valued, being described as vanillin, nuts or clove, although if ageing is extended over six months then off-odor compounds such as 4-ethylguaiacol or 4-ethylphenol may be produced (Alañón, Schumacher, Castro-Vázquez, Díaz-Maroto, Díaz-Maroto, et al., 2013).

Most of these studies were carried out on red wines (Alañón, Schumacher, Castro-Vázquez, Díaz-Maroto, Díaz-Maroto, et al., 2013; De Rosso et al., 2009), vinegars (Chanivet, Durán-Guerrero, Barroso, & Castro, 2020) or wine brandies (Caldeira, Belchior, Clímaco, & Bruno De Sousa, 2002; Canas, Leandro, Spranger, & Belchior, 1999; Garcia et al., 2012), and, therefore, available references to white wines are scarce. Furthermore, research has not been carried out on *oloroso* Sherry wine ageing by means of other than traditionally used wood types. Therefore, this study intends to determine the compounds profile of *oloroso* wines aged in different wood-type barrels and to determine any significant differences between them as well as to confirm the suitability of this alternative woods for Sherry ageing purposes.

2. Materials and methods

2.1. Samples

The wine used for the tests was an unaged *oloroso* Sherry wine, a dry wine made from the *Palomino fino* variety, fortified to 17% alcoholic strength by volume (AS_v). 16 L casks were used for the research. The casks were supplied by Tonelería J. L. Martínez, a cooper from Montilla (Spain). Four groups of new casks were purchased, one of each kind of wood to be used: American oak (*Quercus alba*, AM), French oak (*Quercus robur*, FR), Spanish oak (*Quercus pyrenaica*, SP), and chestnut (*Castanea sativa*, CH). Each group comprised four casks: two of them intensely toasted (INT) and two medium toasted (MED), so each type of wood was tested four times. For the intense toasting procedure, the cask had remained in an oven for 10 min–12 min at 130 °C–140 °C, the medium toasted barrels stayed in the oven for 5 min–7 min at a similar temperature. The casks were preconditioned before use by maintaining them full with water for one month. Then, the ageing process was monitored by sampling 100 mL of wine after two months. Because the ratio of the inner surface of the cask to the volume of the cask for a 16 L cask is higher than that for 600 L casks, which are typically used in the ageing of *oloroso* Sherry wines, the ageing process is considered to be faster in 16 L casks than it is in 600 L casks, so only two months were considered (Herrera et al., 2020). The samples were stored in brown glass flasks at room temperature until analyses (2 weeks at most), all of them performed in duplicate. Due to the special nature of this kind of wine (*oloroso* Sherry wine is completely oxidized) it is very stable in time and samples could be kept at room temperature without risk of evolution. A wine without ageing was also considered as initial sample (INI).

2.2. Oenological reference parameters

pH and acidity were measured by means of a PH-matic 23 automatic titrator (Crison Instruments SA, Barcelona, Spain). The alcoholic strength was measured following the official method from the International Organization of Vine and Wine (OIV) in a D.E. 2000 distiller extraction system (Laboratoires Dujardin-Salleron, Arcueil, France). The alcoholic strength was then determined based on the density of the distillate by means of an Anton Paar densimeter DMA 4500 M (Ashland, USA).

2.3. Short chain organic acids

An isocratic HPLC system with conductivity detection was used to determine 6 short chain organic acids contents (Guillén, Barroso, Zorro, Carrascal, & Pérez-Bustamante, 1998). Samples and standards were filtered through 0.45 μm pore size nylon membranes. Each compound was identified by comparing their peak retention times to those previously obtained by the standards and they were quantified by means of specific calibration curves.

2.4. Phenolic and furanic compounds

A number of UHPLC analyses were carried out to quantify 14 phenolic compounds and furfurals (Schwarz, Rodríguez, Guillén, & Barroso, 2009). The samples and the standards were filtered through 0.22 μm pore-size nylon membranes. Each compound was identified by comparing their peaks retention times and UV–Vis spectra to those previously obtained by the standards. The results were expressed in mg of compound per liter of sample.

2.5. Total phenolic index (TPI)

The *TPI* was determined employing the Folin-Ciocalteau method, using a Thermo Helios Gamma UV-VIS Scanning spectrophotometer (Thermo-Fisher Scientific, Waltham, MA, USA) (Singleton & Rossi, 1965). Gallic acid was used for standard calibration, in the range 10 mg/kg to 1000 mg/kg. The *TPI* results are expressed as gallic acid equivalents (*GAE*).

2.6. Chromatic characteristics

A Thermo Helios Gamma UV-VIS Scanning spectrophotometer (Thermo-Fisher Scientific, Waltham, MA, USA), in the range 380 nm–780 nm, was used to measure the chromatic characteristics of the samples according to the method by the Commission Internationale de l'Eclairage (C.I.E. (Commission Internationale de L'Eclairage) (1986). The data sheet Microsoft Excel 2003, Microsoft Corp., Redmond, WA, USA, was used to convert the spectra into CIELab parameters (Delgado-González, Carmona-Jiménez, Rodríguez-Dodero, & García-Moreno, 2018): $a*$ (green to red tones), $b*$ (blue to yellow tones), $L*$ (lightness) and ΔE_{00} (CIEDE2000). CIEDE2000 parameter allows specifying the difference between the stimuli of two colors perceived as belonging to objects that reflects or transmits light, which, in our case, they are *oloroso* wines. This parameter has been calculated following the indications given in the ISO/CIE 11664–6:2014 standard (ISO/CIE 11664-6, 2014).

2.7. Volatile compounds

SBSE-GC-MS was employed for the analysis of the volatile compounds (Guerrero, Marín, Mejías, & Barroso, 2007). A total of 27 volatile compounds were identified and quantified in the samples. The quantitative data were obtained by use of the calibration curve constructed for each studied compound (six levels of concentrations in duplicate) and comparison of the relative base peak area of each volatile compound with that of 4-methyl-2-pentanol (internal standard). In the case of isoamyl alcohols (2-methylbutan-1-ol and 3-methylbutan-1-ol) they coeluted and the mixture was quantified with the calibration curve of 2-methylbutan-1-ol.

2.8. Sensory analysis

All the sensory analyses were carried out in a normalized tasting room (ISO 8589, 2007) at 22 °C. Standardized wine glasses (ISO 3591, 1977) containing 15 mL of samples were used for all the tests. The glasses were identified by three digital numbers and covered to avoid any losses of aroma. Seven judges, 4 women and 3 men between 25 and

48 years of age, all of them members of the research group, formed the tasting panel. All the judges had extensive experience in sensory evaluations of this kind of oenological samples. The reproducibility of their assessments was determined and the homogeneity of the tasting panel was also evaluated by studying the variance, by two-factor ANOVA (judges x samples) of the descriptive data.

Each judge evaluated five glasses per session: i.e. four different samples and one duplicate. The descriptors to be included in the evaluation sheet were selected according to the results from a preliminary test where several samples of *oloroso* wine were evaluated, and they were: for the visual aspects, color intensity and color impression; for the olfactory aspects, oxidative odor, nuts, toasted, woody and aromatic intensity; for the gustatory aspects, alcoholic, bitter, persistence and body. All of these aspects were evaluated according to the following scale: for descriptor intensity: 0, not present; 2, slight; 4, medium; 6, strong; and 8, very strong; for general impression: 0, bad; 2, unsatisfactory; 4, acceptable; 6, good; and 8, very good (ISO 4121, 2003).

2.9. Statistical analysis

The software package Statistica 8.0 by StatSoft, Inc. (Tulsa, OK, USA) was employed for the ANOVA, the principal component analysis (PCA), the linear discriminant analysis (LDA) and the cluster analysis (CA). For other statistical parameters, Microsoft Excel 2016 (Microsoft Corp., Redmond, WA, USA) was employed. For the statistical analysis, 9 different groups were considered: initial wine (INI), and wine aged in American oak, French oak, Spanish oak and chestnut, intensely and medium toasted (AM-INT, AM-MED, FR-INT, FR-MED, SP-INT, SP-ME, CH-INT and CH-MED).

3. Results and discussion

3.1. Oenological reference parameters

Table 1 presents the mean values of the oenological reference parameters, pH, total acidity and alcoholic strength by volume (AS_v), both at the beginning of the process and after 2 months of ageing by means of the different wood types.

The samples aged in medium toasted barrels presented slightly higher pH values than those aged in intensely toasted ones, with the exception of the samples aged in FR barrels, which presented the same average pH value. The samples' total acidity increased with ageing, what could be derived both from the release of compounds from the wood to the wine (the hydrolysis of acetyl groups of hemicellulose produces acetic acid) and from the oxidation of aldehydes and alcohols into acids during oxidative ageing process, and in general, the samples aged in INT barrels presented slightly higher values. With regards to the type of wood, the samples aged in CH were the ones that showed the highest total acidity levels. Finally, the alcoholic strength of the samples increased slightly with ageing, perhaps due to evaporation phenomena because water molecule is smaller than ethanol molecule and therefore, it could penetrate better through the pores of the wood, and although they were not considered statistically significant (Tukey's HSD test, $p > 0.05$), the samples aged in medium toasted barrels presented slightly higher alcohol values than those aged in intensely toasted barrels.

3.2. Short chain organic acids

Six short chain organic acids were detected and quantified in the studied samples (Table 1). The concentration levels of tartaric acid, lactic acid and acetic acid were significantly higher in the samples aged in chestnut than in those aged in oak barrels. In fact, for the majority of the studied acids, the samples aged in CH presented organic acid levels higher than those of the INI.

Acetic acid was the only acid that increased its concentration in all of the aged samples with respect to the INI. This increment was particularly significant in the samples that had been aged in CH. The cause of this increment in acetic acid contents could be the presence of acetic acid in the chestnut wood itself. The origin of this acid in the wood could be explained by the manufacturing process implemented for this type of barrels, where acetic acid is a secondary product that appears during the thermal degradation process of the wood (Chatonnet, Boidron, & Pons, 1989). Alternatively, this acetic acid higher concentration in wine samples aged in CH could be due to the greater porosity of this type of wood when compared to oak wood (Acuña, Gonzalez, De La Fuente, & Moya, 2014). Such greater porosity would give place to a greater contact between the liquid and the wood, and therefore to a greater extraction and oxidation.

With regards to organic acid concentration levels in relation to wood toasting grades, the samples aged in AM and FR, MED barrels presented slightly higher concentration levels, while SP and CH, MED barrels exhibited the opposite trend. In any case, the differences were only significant for CH barrels (Tukey's HSD test, $p < 0.05$).

3.3. Phenolic and furanic compounds and TPI

Table 1 presents the concentration of low molecular weight phenolic and furanic compounds in the samples. These compounds have also been previously identified in *oloroso* Sherry wine, where a similar concentration growth had been observed as they were aged in the barrels (Fabios, Lopez-Toledano, Mayen, Merida, & Medina, 2000; García-Moreno & García-Barroso, 2002; Ortega, Lopez-Toledano, Mayen, Merida, & Medina, 2003; Ortega, Mayen, & Medina, 2008). This climb was slightly more pronounced in the samples that had been aged in CH or SP in comparison to those aged in AM or FR barrels.

No studies on the oxidative ageing of *oloroso* Sherry wine in wooden barrels, other than AM, has been reported. However, some studies focusing on the effect of different wood types other than AM on the oxidative ageing of red wine, brandy, and other spirits can be found (Alañón et al., 2013; Canas, Silva, & Belchior, 2008; Garcia et al., 2012; García-Moreno et al., 2020). According to such papers, the beverages that were aged in CH barrels generally showed greater phenolic compounds contents than those aged in oak wood barrels. Also, of all the drinks aged in different oak wood types, SP barrels tended to present higher levels of such compounds – particularly gallic acid and other benzoic and cinnamic acids such as protocatechuic, *p*-hydrobenzoic or *p*-coumaric acids. This greater phenolic compounds content in the beverages aged in both CH and SP barrels could be attributed to the greater content levels of such compounds in these two wood types (Alañón et al., 2011; Fernández de Simón, Cadahía, Conde, & García-Vallejo, 1996).

Gallic acid was the major compound of all the quantified phenolic compounds. This acid increased with ageing in all the samples, especially in those wines aged in CH barrels. In our study, the gallic acid concentration levels in the samples aged in AM were similar to those measured by Ortega et al. (2003), and somewhat higher than those reported by Fabios et al. (2000).

p-Hydroxybenzoic acid, caffeic acid and *p*-coumaric acid were some of the other phenolic acids identified in the samples. All of them were present in the INI, *p*-coumaric was not detected in the aged samples, caffeic acid decreased and *p*-hydroxybenzoic acid increased slightly with ageing. These results seem to be consistent with those reported by other authors for *oloroso* Sherry wines subjected to ageing processes, where a decrease in caffeic and *p*-coumaric acids was observed (García-Moreno & García-Barroso, 2002; Ortega et al., 2003), as well as a slight increase in *p*-hydroxybenzoic acid (Fabios et al., 2000). Nevertheless, the data from our study reflected lower levels than those reported in the literature for similar wines.

The concentrations of caftaric acid and *p*-coutaric acid showed minor variations with ageing. Similar behaviors in relation to these acids' content during the ageing of *oloroso* wines were documented by other authors; García-Moreno and García-Barroso (2002), observed that in the early stages of the ageing process the samples did not present any

Table 1

Oenological control parameters, organic acid, phenolic and furanic compounds and Total Polyphenolic Index and chromatic characteristics at the beginning of the process and after 2 months of ageing in American, French and Spanish oaks and Chestnut barrels with different toasting.

	Initial	*American Oak*		*French Oak*		*Spanish Oak*		*Chestnut*	
		Intense toasting	*Medium toasting*	*Intense toasting*	*Medium toasting*	*Intense toasting*	*Medium toasting*	*Intense toasting*	*Medium toasting*
Oenological control parameters									
pH	3.20 ± 0.01[a]	3.35 ± 0.00[bc]	3.38 ± 0.01[cd]	3.38 ± 0.00[cd]	3.38 ± 0.00[cd]	3.36 ± 0.00[bcd]	3.39 ± 0.01[d]	3.34 ± 0.01[b]	3.37 ± 0.00[cd]
Total acidity	4.16 ± 0.01[a]	4.32 ± 0.02[b]	4.42 ± 0.01[de]	4.42 ± 0.01[de]	4.44 ± 0.01[e]	4.39 ± 0.01[cd]	4.36 ± 0.01[bc]	4.70 ± 0.01[g]	4.64 ± 0.00[f]
AS_v (%)	17.25 ± 0.13[a]	17.39 ± 0.19[ab]	17.61 ± 0.37[ab]	17.52 ± 0.34[ab]	18.18 ± 0.16[b]	17.50 ± 0.17[ab]	18.25 ± 0.13[b]	17.77 ± 0.24[ab]	18.25 ± 0.15[b]
Organic Acids (mg/L)									
Citric	100.3 ± 1.3[a]	103.2 ± 4.1[a]	102.2 ± 5.6[a]	114.9 ± 7.8[a]	126.8 ± 4.5[a]	122.4 ± 4.7[a]	128.8 ± 2.3[a]	129.2 ± 21.4[a]	118.5 ± 6.5[a]
Tartaric	3167.6 ± 36.7[a]	2765.0 ± 109.2[a]	3223.0 ± 344.7[a]	2994.0 ± 25.5[a]	3278.0 ± 50.5[a]	3090.0 ± 195.7[a]	3036.0 ± 122.5[a]	4621.0 ± 44.3[b]	4114.0 ± 24.8[b]
Malic	450.4 ± 22.0[a]	390.0 ± 20.8[a]	466.0 ± 73.7[a]	428.0 ± 5.4[a]	485.0 ± 5.3[a]	472.0 ± 31.9[a]	466.0 ± 11.6[a]	464.0 ± 14.3[a]	438.0 ± 5.8[a]
Succinic	609.7 ± 18.5[a]	540.0 ± 1.2[a]	655.0 ± 109.1[a]	560.0 ± 9.2[a]	618.0 ± 22.1[a]	594.0 ± 41.7[a]	598.0 ± 38.1[a]	591.0 ± 32.1[a]	544.0 ± 8.0[a]
Lactic	772.0 ± 39.8[abc]	618.0 ± 20.8[a]	728.0 ± 111.8[abc]	659.0 ± 6.1[ab]	734.0 ± 32.3[abc]	714.0 ± 48.4[ab]	725.0 ± 28.2[ab]	914.0 ± 38.8[c]	820.0 ± 11.8[bc]
Acetic	256.1 ± 16.2[a]	288.0 ± 15.0[ab]	372.0 ± 68.8[bc]	268.0 ± 11.1[ab]	286.0 ± 33.0[ab]	363.0 ± 27.4[abc]	335.0 ± 9.8[abc]	506.0 ± 2.8[d]	415.0 ± 7.5[cd]
Phenolic and furanic compounds and TPI (GAE)									
Gallic acid	7.27 ± 0.04[a]	11.20 ± 0.01[b]	12.15 ± 0.07[d]	11.95 ± 0.02[c]	13.36 ± 0.02[e]	18.10 ± 0.03[g]	17.36 ± 0.06[f]	99.67 ± 0.01[h]	106.07 ± 0.02[i]
p-Hydroxybenxoic acid	0.23 ± 0.00[a]	0.42 ± 0.01[e]	0.46 ± 0.00[f]	0.40 ± 0.00[d]	0.33 ± 0.00[c]	0.65 ± 0.00[h]	0.58 ± 0.00[g]	0.28 ± 0.00[b]	0.28 ± 0.00[b]
Caffeic acid	1.80 ± 0.00[d]	1.50 ± 0.00[b]	1.54 ± 0.01[c]	1.53 ± 0.00[c]	1.51 ± 0.00[b]	2.04 ± 0.00[f]	1.83 ± 0.00[e]	1.45 ± 0.00[a]	1.45 ± 0.01[a]
p-Coumaric acid	0.64 ± 0.01[b]	n.d.	n.d.	n.d.	n.d.	0.6 ± 0.00[a]	n.d.	n.d.	n.d.
Caftaric acid	27.51 ± 0.49[e]	26.78 ± 0.04[d]	25.84 ± 0.03[c]	24.99 ± 0.01[a]	25.16 ± 0.01[ab]	24.98 ± 0.02[a]	24.98 ± 0.01[a]	25.77 ± 0.01[bc]	25.82 ± 0.02[c]
Coutaric acid	16.49 ± 0.06[g]	15.18 ± 0.03[b]	14.85 ± 0.04[a]	15.21 ± 0.01[b]	15.39 ± 0.01[c]	15.71 ± 0.02[d]	15.80 ± 0.03[d]	16.24 ± 0.03[f]	16.09 ± 0.04[e]
Vanillin	1.08 ± 0.01[d]	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.71 ± 0.00[c]
Sinapaldehyde	1.80 ± 0.01[e]	1.35 ± 0.01[b]	1.25 ± 0.00[a]	1.45 ± 0.03[c]	1.50 ± 0.01[cd]	1.25 ± 0.01[a]	1.31 ± 0.01[ab]	1.54 ± 0.04[d]	1.45 ± 0.00[c]
Syringaldehyde	0.63 ± 0.02[a]	3.37 ± 0.01[g]	3.03 ± 0.04[f]	2.89 ± 0.00[e]	2.70 ± 0.00[d]	3.34 ± 0.02[g]	3.10 ± 0.03[f]	1.92 ± 0.02[b]	2.48 ± 0.02[c]
Furfural	0.34 ± 0.01[a]	8.02 ± 0.01[b]	8.35 ± 0.01[c]	9.29 ± 0.01[d]	9.54 ± 0.01[e]	14.80 ± 0.01[f]	15.07 ± 0.02[g]	14.76 ± 0.01[f]	16.36 ± 0.01[h]
5-Methylfurfural	0.13 ± 0.01[b]	0.25 ± 0.00[e]	0.24 ± 0.01[de]	0.22 ± 0.00[d]	0.23 ± 0.00[d]	0.09 ± 0.00[a]	0.13 ± 0.00[b]	0.18 ± 0.00[c]	0.14 ± 0.00[b]
5-Hydroxymethylfurfural	1.72 ± 0.06[a]	4.55 ± 0.01[b]	4.73 ± 0.01[c]	4.46 ± 0.04[b]	4.91 ± 0.01[d]	7.68 ± 0.02[f]	7.01 ± 0.01[e]	7.79 ± 0.04[f]	8.67 ± 0.02[g]
TPI	522.80 ± 0.01[a]	512.21 ± 16.58[a]	515.49 ± 10.61[a]	570.37 ± 1.99[b]	536.60 ± 3.32[ab]	645.56 ± 3.29[c]	634.00 ± 17.43[c]	845.21 ± 3.32[d]	861.15 ± 0.66[d]
Chromatic characteristics									
L* (%)	86.0 ± 0.6[e]	84.1 ± 0.1[de]	82.4 ± 0.4[cd]	77.7 ± 0.4[b]	85.9 ± 0.0[de]	78.0 ± 0.0[b]	79.0 ± 0.5[bc]	70.1 ± 2.2[a]	71.6 ± 1.2[a]
a*	4.9 ± 0.1[a]	6.3 ± 0.4[bc]	6.7 ± 0.1[c]	5.8 ± 0.1[b]	6.5 ± 0.0[bc]	9.8 ± 0.2[d]	9.6 ± 0.0[d]	12.9 ± 0.1[e]	13.9 ± 0.0[f]
b*	37.4 ± 0.9[b]	38.0 ± 0.3[b]	38.1 ± 0.1[b]	35.6 ± 0.2[a]	37.2 ± 0.0[b]	48.7 ± 0.0[c]	48.7 ± 0.1[c]	55.4 ± 0.3[d]	57.6 ± 0.4[e]
ΔE_{00}	–	1.6 ± 0.1[a]	2.6 ± 0.3[a]	5.7 ± 0.3[b]	0.8 ± 0.0[a]	6.9 ± 0.0[b]	6.3 ± 0.2[b]	13.1 ± 1.4[c]	12.5 ± 0.7[c]

Data are mean values ± standard deviation (n = 4); for the same row, different letters in different columns indicate significant differences, according to Tukey test ($p < 0.05$).

AS_v: Alcoholic strength by volume; GAE: Gallic acid equivalents; n.d.: not detected.

variations in content, while Ortega et al. (2003) observed some content increase over the first ageing year. The concentration values were similar to those found by the same and other authors (Fabios et al., 2000) in *oloroso* wines that was aged for several years.

The aldehydes, syringaldehyde, sinapaldehyde and vanillin content levels, presented a marked correlation with the degradation of the wood lignin that takes place during the ageing. Of the three aldehydes detected, syringaldehyde, in particular, increased with ageing, while sinapaldehyde and vanillin presented a slight decrease over the ageing process. The detected concentrations were similar to those of an *oloroso* wine aged for 3–4 years (Ortega et al., 2003). The samples aged in AM and SP showed higher values for syringaldehyde contents. According to Cadahía et al. (2001), AM (*Quercus alba*) and FR (*Quercus robur*) are the oaks that contain the largest concentrations of this compound; However, other authors (Alañón et al., 2011) consider that FR (*Quercus robur*) and SP (*Quercus pyrenaica*) are the oaks with the greatest syringaldehyde contents. However, this content largely depends on charring level.

Furfural and 5-HMF increased considerably with ageing, while 5-MF did so by a lesser degree. The samples aged in CH and SP were the ones where the highest levels of the three compounds were detected in the *oloroso* wine, just like in aged brandies (García-Moreno et al., 2020). These woods are the most porous of the four types in the study and also the highest cellulose content level, which favors the formation of these compounds during the toasting of the wood and their subsequent extraction during the ageing process (Acuña et al., 2014; Canas et al., 1999; Chatonnet et al., 1989).

The *TPI* data that have been included in Table 1 confirmed the results already reported for phenolic and furanic compounds: the samples aged in CH showed the highest *TPI* values, followed by SP, FR, and finally, those aged in AM, which presented values even lower than the initial sample. Finally, the dendrogram obtained from the cluster analysis (Fig. 1) showed a clear differentiation between the samples, both by type of wood and by toasting grade based on the standardized concentration of phenolic and furanic compounds and *TPI*. Based on these results, it is evident that wood type was the main differentiating factor in the ageing process, followed by the toasting grade.

3.4. Chromatic characteristics

The CIELab coordinates of the samples, $a*$ (green to red tones), $b*$ (blue to yellow tones), $L*$ (lightness) and CIEDE2000 parameter (ΔE_{00}), are shown in Table 1.

In relation to the ΔE_{00}, according to the Tukey's HSD test ($p < 0.05$),
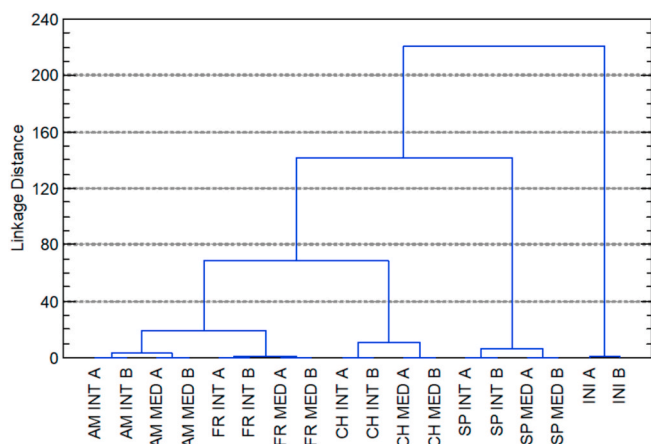

Fig. 1. Cluster analysis obtained using the square Euclidean distance as metrics and the Ward method as clustering rule for the analyzed samples. The phenolic and furanic compounds and the TPI were used as the variables (INI: Initial wine; AM: American oak; FR: French oak; SP: Spanish oak; CH: Chestnut; INT: Intense toasting level; MED: Medium toasting level).

three groups were clearly observed. The first group would be formed by the samples aged in AM (AM-INT and AM-MED) and in FR-MED, these samples had the lowest ΔE_{00} values, all of them lower than 3. In these cases, the color differences between the samples and the initial sample were not easily detected by an observer (Martínez, Melgosa, Pérez, Hita, & Negueruela, 2001). Therefore, it can be said that no color difference could be noticed by the tasters (Gómez-Míguez, Gómez-Míguez, Vicario, & Heredia, 2007). The samples in second group formed by FR-MED and SP (SP-INT and SP-MED) presented intermediate values of ΔE_{00}, between 5.5 and 7.0, which allowed the tasters a certain differentiation between the initial and the final samples. The last group formed by the samples aged in CH (CH-INT and CH-MED) registered the highest ΔE_{00} values, between 12 and 14. These samples presented the widest color difference when compared to the initial wine.

Regarding the parameters $a*$, $b*$ and $L*$ (Table 1), the color differences between the initial sample and the aged samples were due to a decrease in lightness (decrease in $L*$) and an increase in the red and brown colors (increase in $a*$ and decrease in $b*$). These changes correspond to a typical ageing process of *oloroso* Sherry wine (Ortega et al., 2003). During the oxidative ageing typical of these wines, some brown compounds appear as the result of the condensation of the phenolic compounds in the presence of acetaldehyde and phenolic aldehydes (Fabios et al., 2000; Ortega et al., 2003). In addition, similarly to other aged beverages subjected to oxidative processes, they could also be originated from the oxidation of the ellagitannins in the barrel's own wood (Fujieda, Tanaka, Suwa, Koshimizu, & Kouno, 2008).

The decrease in lightness was less pronounced in the samples aged in oak barrels than in those aged in CH. Lightness values grew as follows: AM < FR < SP < CH, also in line with the ΔE_{00} parameter values. The wines aged in AM and FR presented values of $a*$ and $b*$ very similar to those of the initial sample. The hue values of these aged wines were, therefore, very similar to those of the initial unaged *oloroso* Sherry wine. Wine aged in SP and CH presented values of $a*$ and $b*$ higher than those of the initial samples, the *oloroso* wine samples aged in CH were the ones that presented the highest values. Therefore, these samples had more intense red (higher $a*$ values) and yellow tones (higher $b*$ values), which in turn results in a more intense brown tone. Other authors have reported similar results for brandy samples aged in different oak wood types and CH: i.e. the samples aged in AM and in FR showed a slighter color increment than the samples aged in SP, and these in turn showed less color increase than those aged by means of CH (García-Moreno et al., 2020). No differences in the color parameters of the wines aged in the same type of wood and different toasting grades could be detected.

3.5. Volatile compounds

Table 2 presents the mean concentrations of the volatile compounds studied. As can be seen, FR and CH were the woods that produced greater modifications of the wine's aroma during the ageing period. These wines presented significant differences between the initial and final samples for a higher number of volatile compounds. Others authors reported similar results when they studied CH as an alternative for the ageing of red wines (Alañón, Schumacher, Castro-Vázquez, Díaz-Maroto, Díaz-Maroto, et al., 2013) and sweet wines (Herrera et al., 2020). On the other hand, AM and SP modified the volatile profile of wines to a lesser extent. Volatile compounds derived from wood ageing increased significantly their concentrations during the process and in general terms, their concentrations were higher when medium toast was employed. Similar results have been reported by other authors with regards to fortified and sweet wines aged in wood (Herrera et al., 2020; Hevia et al., 2016).

The same behavior presented eugenol and guaiacol — compounds derived from the degradation of lignin. Although their content increased in all of the aged wines regardless of the wood type used, guaiacol did not present significant differences when aged in AM. On the other hand, β-methyl-γ-octalactone was only detected in the wines aged in the

Table 2
Mean concentrations of volatile compounds at the beginning of the process and after 2 months of ageing in American, French and Spanish oaks and Chestnut barrels with different toasting.

Volatile compounds (μg/L)	Initial	American Oak		French Oak		Spanish Oak		Chestnut	
		Intense toasting	Medium toasting	Intense toasting	Medium toasting	Intense toasting	Medium toasting	Intense toasting	Medium toasting
Isobutyl acetate	44.61 ± 17.31[ab]	25.93 ± 3.21[a]	28.81 ± 2.41[a]	78.21 ± 9.71[b]	137.11 ± 3.31[c]	44.81 ± 1.61[ab]	42.31 ± 1.71[ab]	29.14 ± 2.57[a]	34.74 ± 5.10[ab]
Ethyl butyrate	114.72 ± 40.43[a]	110.75 ± 6.65[a]	129.31 ± 17.47[a]	103.72 ± 24.61[a]	98.00 ± 23.91[a]	80.01 ± 6.01[a]	82.22 ± 4.41[a]	108.32 ± 23.01[a]	115.74 ± 23.40[a]
Butyl acetate	125.79 ± 40.35[a]	122.01 ± 6.70[a]	140.53 ± 17.48[a]	114.88 ± 24.60[a]	109.20 ± 23.90[a]	91.09 ± 6.01[a]	93.33 ± 4.40[a]	119.49 ± 23.01[a]	126.83 ± 23.42[a]
Ethyl isovalerate	3.00 ± 1.38[a]	2.04 ± 0.21[a]	2.46 ± 0.11[a]	9.18 ± 0.60[b]	2.51 ± 0.92[a]	2.35 ± 0.40[a]	1.14 ± 0.21[a]	2.79 ± 0.63[a]	3.52 ± 0.81[a]
Isoamyl acetate	207.08 ± 38.89[a]	172.01 ± 13.91[a]	211.15 ± 5.41[a]	398.88 ± 51.12[b]	503.06 ± 47.74[b]	398.53 ± 9.34[b]	377.45 ± 54.90[b]	104.79 ± 44.11[a]	141.00 ± 65.00[a]
Ethyl valerate	6.43 ± 1.53[bc]	2.21 ± 0.31[ab]	2.64 ± 1.91[ab]	10.01 ± 1.13[a]	9.56 ± 2.91[c]	7.72 ± 0.71[bc]	7.67 ± 3.01[bc]	1.59 ± 0.58[a]	4.70 ± 3.01[abc]
Butan-1-ol[a]	1.51 ± 0.50[a]	1.58 ± 0.04[a]	1.75 ± 0.16[a]	1.00 ± 0.19[a]	0.97 ± 0.04[a]	1.23 ± 0.06[a]	1.21 ± 0.07[a]	1.34 ± 0.10[a]	1.39 ± 0.10[a]
Isoamyl alcohols[a,b]	21.99 ± 3.79[a]	25.35 ± 0.29[a]	27.96 ± 1.53[a]	20.30 ± 1.94[a]	21.06 ± 1.39[a]	20.22 ± 0.74[a]	20.51 ± 0.48[a]	20.96 ± 3.27[a]	24.17 ± 0.28[a]
Ethyl caproate	123.31 ± 7.19[a]	78.29 ± 2.31[a]	120.94 ± 12.22[a]	112.54 ± 10.63[a]	134.45 ± 19.45[a]	97.48 ± 17.88[a]	113.80 ± 0.50[a]	99.80 ± 15.17[a]	131.91 ± 12.64[a]
Hexyl acetate	11.45 ± 2.52[c]	0.32 ± 0.10[a]	3.00 ± 0.71[ab]	6.92 ± 1.73[abc]	8.27 ± 0.24[cb]	7.00 ± 0.68[abc]	7.30 ± 0.80[abc]	0.31 ± 0.17[a]	1.41 ± 0.33[ab]
Hexan-1-ol[a]	1.27 ± 0.11[a]	1.15 ± 60.46[a]	1.29 ± 42.54[a]	1.15 ± 0.04[a]	1.24 ± 0.05[a]	1.18 ± 0.02[a]	1.12 ± 0.03[a]	1.22 ± 0.20[a]	1.37 ± 0.03[a]
Ethyl octanoate	83.94 ± 34.00[b]	8.36 ± 1.84[a]	42.60 ± 7.20[ab]	88.21 ± 28.33[b]	117.77 ± 30.70[b]	71.79 ± 3.18[ab]	93.05 ± 0.01[b]	23.74 ± 7.88[ab]	61.31 ± 21.00[ab]
Benzaldehyde	18.61 ± 9.61[b]	24.03 ± 1.01[bc]	23.80 ± 4.80[bc]	16.01 ± 0.33[ab]	24.77 ± 17.81[bc]	13.21 ± 1.48[ab]	16.00 ± 3.77[ab]	76.44 ± 38.66[d]	51.63 ± 3.50[cd]
Isobutyric acid[a]	n.d.	n.d.	n.d.	2.24 ± 0.65[a]	3.77 ± 1.21[a]	n.d.	n.d.	n.d.	n.d.
γ-Butyrolactone[a]	5.88 ± 1.10[ab]	10.50 ± 3.21[c]	6.45 ± 0.62[abc]	5.76 ± 0.65[ab]	9.22 ± 1.85[bc]	5.62 ± 1.04[ab]	4.98 ± 0.84[ab]	3.97 ± 0.69[a]	7.11 ± 3.05[abc]
Diethyl succinate[a]	2.07 ± 0.64[a]	2.27 ± 0.14[a]	2.46 ± 0.03[a]	1.81 ± 0.05[a]	2.09 ± 0.06[a]	1.54 ± 0.12[a]	1.69 ± 0.01[a]	4.33 ± 0.40[b]	4.71 ± 0.07[b]
α-Terpineol	12.27 ± 5.41[a]	7.04 ± 2.83[a]	6.15 ± 0.80[a]	9.94 ± 0.42[a]	11.31 ± 0.61[a]	10.41 ± 0.49[a]	8.75 ± 1.26[a]	8.01 ± 1.22[a]	10.21 ± 0.22[a]
Guaiacol	181.10 ± 54.38[a]	280.86 ± 67.33[ab]	281.45 ± 33.21[ab]	236.21 ± 89.24[ab]	429.21 ± 161.44[b]	289.71 ± 94.39[ab]	346.25 ± 4.00[b]	349.51 ± 24.81[b]	433.54 ± 17.55[b]
β-Methyl-γ-octalactone	n.d.	66.18 ± 12.32[ab]	140.22 ± 44.41[bc]	209.46 ± 68.51[cd]	315.33 ± 173.91[d]	9.43 ± 0.60[ab]	110.45 ± 20.71[abc]	n.d.	n.d.
Phenylethanol[a]	3.53 ± 0.45[a]	3.43 ± 0.20[a]	3.92 ± 0.05[a]	8.84 ± 0.15[b]	9.77 ± 0.33[b]	9.23 ± 0.15[b]	8.99 ± 0.15[b]	9.05 ± 0.84[b]	10.21 ± 0.07[b]
4-Ethylguaiacol	2.34 ± 0.36[a]	2.66 ± 0.21[a]	3.14 ± 0.20[a]	6.04 ± 0.61[b]	7.76 ± 1.00[cd]	7.53 ± 1.40[bcd]	7.90 ± 0.93[cd]	6.42 ± 0.33[bc]	8.64 ± 0.22[d]
Octanoic acid[a]	0.57 ± 0.07[b]	0.40 ± 0.02[a]	0.49 ± 0.04[ab]	1.20 ± 0.12[d]	1.35 ± 0.05[d]	1.30 ± 0.00[d]	1.42 ± 0.04[d]	0.67 ± 0.07[b]	0.95 ± 0.00[c]
Nonanoic acid	12.12 ± 5.07[a]	8.01 ± 1.21[a]	11.63 ± 0.43[a]	8.71 ± 1.81[a]	9.66 ± 0.60[a]	8.53 ± 0.10[a]	10.51 ± 0.45[a]	3.11 ± 0.56[a]	3.24 ± 0.56[a]
Eugenol	n.d.[a]	2.84 ± 0.09[ab]	9.43 ± 2.60[ab]	18.82 ± 4.94[ab]	34.92 ± 12.52[b]	13.12 ± 9.00[ab]	21.80 ± 3.33[ab]	32.09 ± 0.81[b]	84.74 ± 50.22[c]
4-Ethylphenol	6.22 ± 2.11[a]	4.04 ± 0.47[a]	4.23 ± 0.50[a]	7.19 ± 0.88[ab]	7.81 ± 1.64[ab]	7.14 ± 0.91[ab]	9.25 ± 0.43[ab]	6.17 ± 1.94[ab]	12.81 ± 4.33[b]
Decanoic acid	184.50 ± 57.85[bc]	87.24 ± 22.84[ab]	128.52 ± 19.91[abc]	172.37 ± 48.68[abc]	231.60 ± 17.43[bc]	180.60 ± 30.11[abc]	285.53 ± 11.11[c]	39.17 ± 1.54[a]	78.61 ± 1.22[ab]

Data are mean values ± standard deviation (n = 4); for the same row, different letters in different columns indicate significant differences, according to Tukey test ($p < 0.05$). n.d.: not detected.

[a] mg/L.

[b] mixture of isomers (2-methylbutan-1-ol + 3-methylbutan-1-ol).

different oak woods. This was to be expected on the basis of previous studies (Fernández de Simón, Esteruelas, Muñoz, Cadahía, & Sanz, 2009; Masson, Guichard, Fournier, & Puech, 1995). Very high concentrations of γ-butyrolactone (in the range of mg/L) were determined in all the samples. Similar results were obtained by other authors (Hevia et al., 2016), who reported similar high concentrations of this compound in aged *oloroso* wines. Other compounds such as ethyl valerate, hexyl acetate or ethyl octanoate, with floral and fruity notes, decreased with wood-aged wines (De Rosso et al., 2009) except for those wines aged in FR. In this case, the above mentioned compound and others such as isobutyl acetate, ethyl valerate or isoamyl acetate increased their concentrations in a significant way after wood ageing. Some compounds that could produce off-flavors in wine such as 4-ethylphenol or 4-ethyl-guaiacol (Martorell, Martí, Mestres, Busto, & Guasch, 2002) were also determined in the samples. As can be seen, these compounds increased

their concentrations after wood ageing and similar results were obtained by other authors in previous studies (Alañón, Schumacher, Castro-Vázquez, Díaz-Maroto, Díaz-Maroto, et al., 2013). However, their concentrations were below their olfactory perception thresholds (Csikor, Pusztai, & Barátossy, 2018).

In order to study the similarities between the volatile composition of the samples, the set of data obtained was submitted to PCA. According to Kaiser's criterion (eigenvalues > 1) 6 PC were obtained that explained 90.04% of the total variability, whereas the first two PCs explained the 56.03%. As can be seen in Fig. 2a, AM and CH samples presented positive values for PC1, whereas the FR and SP samples presented negative values for this PC. On the contrary, PC2 was able to separate samples aged in CH, from those aged in oak wood, although the samples from FR and SP were not clearly separated (Fig. 2a). On the one hand, the volatile compounds that contributed the most to PC1 were isoamyl acetate, ethyl
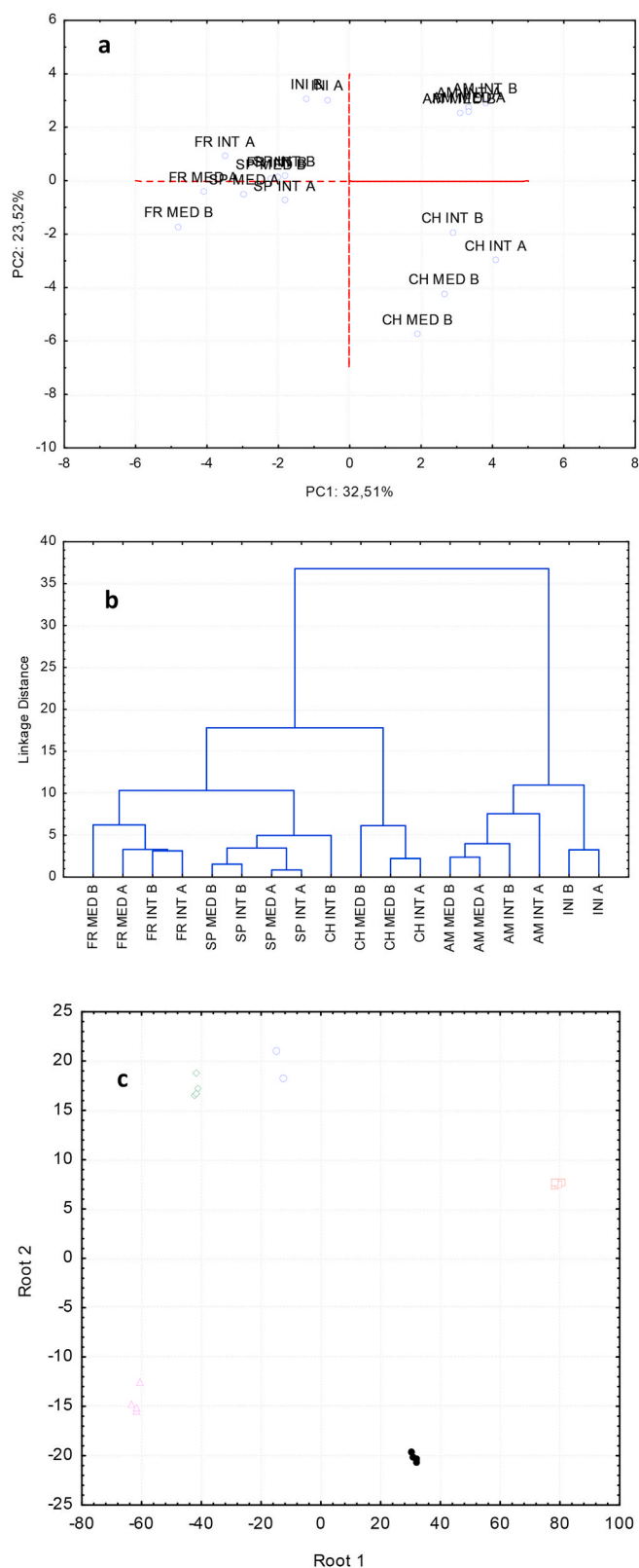
valerate, ethyl octanoate, butan-1-ol, decanoic acid and octanoic acid, among others. On the other hand, the compounds related to wood ageing were those that contributed the most to PC2: γ-butyrolactone, eugenol, β-methyl-γ-octalactone, guaiacol, 4-ethylguaicol, among others.

In order to corroborate the similarities observed in the PCA, the samples' volatile composition was subjected to a cluster analysis of cases (CA). In the resulting dendogram (Fig. 2b) the aged samples were grouped according to the wood employed for the process. As can be seen, the initial unaged samples were more similar to those aged in AM barrels. This fact corroborated the results from the ANOVA and the PCA, i. e.: the wines aged with AM suffered less modifications in comparison to the initial unaged wine. Moreover, the rest of the samples formed another group, in which the FR and SP samples presented closer similarities between them when compared to the CH samples.

So, on the basis of the results obtained from the CA, and in order to obtain valid classification rules for the samples according to their volatile composition, a forward stepwise linear discriminant analysis (LDA) was applied to the data set. The classification criterion employed was the type of wood, with four categories: CH, AM, FR and SP. 100% of the samples were successfully classified (Fig. 2c). A total of 9 significant variables were obtained from the model ($p < 0.05$), being diethyl succinate, ethyl isovalerate, isoamyl alcohols (2-methylbutan-1-ol + 3-methylbutan-1-ol), or β-methyl-γ-octalactone content the most relevant variables for the wine samples classification.

3.6. Sensory analysis

The sensory analysis of the samples comprised three phases: visual, olfactory and gustatory. During the first phase, the judges evaluated color intensity and color impression. In the second phase, the following descriptors were evaluated: oxidative odor, nuts, toasted, woody, aromatic intensity and odor impression, and finally, during the gustatory phase, alcoholic sensation, bitter, persistence, body and gustative impression were the descriptors to be evaluated.

The ANOVA test applied to differentiate wood types showed that there were few descriptors that could make a definite distinction between the four wood types. In fact, 'Color intensity' and 'color impression' showed similar values from a statistical point of view ($p_{ANOVA} > 0.05$) in relation to both wood type and level of toasting. However, the *oloroso* wine samples aged in CH barrels obtained slightly higher medium values for the descriptor 'color impression' (Fig. 3a) and 'color intensity' (data not shown). This in concordance with the results obtained from the CIELab coordinates analysis, in which CH samples presented the most intense brown tone (Table 1). Likewise, no significant differences were detected between 'aromatic intensity', 'oxidative odor', 'toast', 'alcoholic', 'body' and 'gustative impression', in the olfactory and gustative evaluation phases.

The greatest differences between wood types found by the judges were associated to the intensity of the 'nuts' descriptor ($p_{ANOVA} = 0.035$) and these were ranked as follows: FR > CH > SP > AM. This descriptor was regarded as a positive olfactory feature based on the highly appreciated FR samples, which were greatly valued for 'odor impression'. In addition, the FR wine samples were also granted the highest scores for 'gustative persistence'.

When the ANOVA test was applied to discriminate between MED and INT level of toasting (Fig. 3b), the only apparently relevant descriptor was 'woody'. All of the MED samples received higher scores for this descriptor, with the exception of the FR-MED ones, where the perception of its woody character was granted with lower scores. The judges showed a trend to consider a high woody character as a negative feature, which could be the reason why the FR-MED samples were very highly valued both in the olfactory and gustatory phases (data not shown). This fact could also be explained by the increase of several volatile compounds in FR wines such as hexyl acetate, isoamyl acetate, or ethyl octanoate, among others (Table 2), that contributed with positive fruity
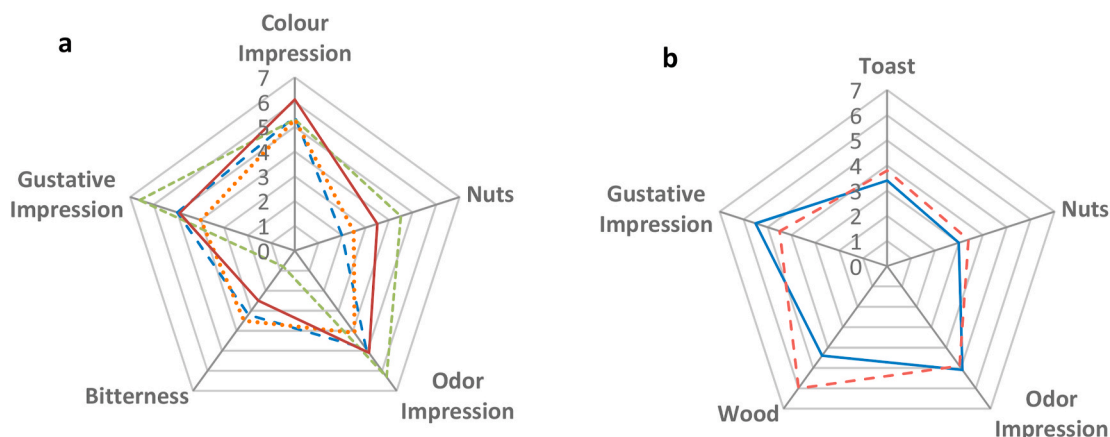
Fig. 2. Multivariate analysis for volatile composition: a) Principal Component Analysis. (INI: Initial wine; AM: American oak; FR: French oak; SP: Spanish oak; CH: Chestnut; INT: Intense toasting level; MED: Medium toasting level). b) Cluster Analysis. (INI: Initial wine; AM: American oak; FR: French oak; SP: Spanish oak; CH: Chestnut; INT: Intense toasting level; MED: Medium toasting level). c) Linear Discriminant Analysis. (◯ Initial; ☐ Chestnut; ◇ American oak; △ Spanish oak; ● French oak).

Fig. 3. Radar chart of the descriptors that showed significant differences during the sensory analyses: a) by wood type (– – – American oak; ----- French oak; .... Spanish oak; —— Chestnut). b) by toasting grade (—— Intense toasting; – – Medium toasting).

and floral descriptors. Surprisingly, no score differences associated to the toasting grades could be noticed. This result is opposite to that reported by other authors for wood aged spirits such as brandy (García-Moreno et al., 2020). However, in our case, the volatile composition analysis provided similar results and samples were better discriminated on the basis of the type of wood, rather than the toasting level (Fig. 2).

4. Conclusions

The results confirmed that the use of alternative wood types to age *oloroso* Sherry wine has some potential to provide the final product with significantly different features other that those obtained through traditional ageing procedures in AM. These new features are consistent with the characteristics that consumers seek for: sweetness, nutty character, softness, no woody character, etc. The combination of more than one wood type would lead to an optimization that would provide them with the most highly appreciate features from each wood species: e.g. softness from AM, dark color from CH, aroma from FR or body from SP. Wood toasting grade has also proven to be a relevant factor to be taken into account. In this sense, according to the results from our sensory evaluation, the wine samples aged in intensely toasted wood barrels were more appreciated. This could be due to their lesser woody character.

CRediT authorship contribution statement

M. Valme García-Moreno: Software, Visualization, Writing - original draft. **Manuel M. Sánchez-Guillén:** Investigation, Data curation. **Manuel J. Delgado-González:** Software, Data curation. **Enrique Durán-Guerrero:** Validation, Software, Writing - review & editing. **M. Carmen Rodríguez-Dodero:** Methodology, Software, Formal analysis. **Carmelo García-Barroso:** Resources, Funding acquisition. **Dominico A. Guillén-Sánchez:** Conceptualization, Project administration, Supervision.

Declaration of competing interest

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

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Acknowledgements

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