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The impact of ultrasound, micro-oxygenation and oak wood type on the phenolic and volatile composition of a Tempranillo red wine

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ABSTRACT

The accelerated ageing of a Tempranillo red wine has been studied at pilot scale through the combined application of ultrasound, micro-oxygenation and different oak wood type chips (American, French and Spanish). The phenolic and volatile content of the aged samples and their sensory profiles have been determined. The wine samples that had been aged using micro-oxygenation, French or American oak chips, and ultrasound revealed to be similar in terms of polyphenols to those wines that had been aged without implementing this last accelerating technique. Ageing time, with a high extraction kinetics, was the most significant variable with regard to their polyphenol content. In terms of volatiles, Spanish and French oak wood wine samples showed a similar behaviour closely associated to ageing time, while American oak wood achieved a rather low enrichment in volatile constituents, resulting in a poor sensory profile of the final wines, which was particularly poor in the case of ultrasound aged wines.

1. Introduction

Red wine has been traditionally aged in oak barrels usually made of the American or French oak wood varieties. Wine aged under these conditions acquires bouquet and undergoes a series of highly significant changes regarding its flavour and colour, which are all induced by changes in its polyphenolic and volatile compounds content (Apetrei et al., 2012; Rubio-Bretón et al., 2018). Wood origin, stave toasting degree and the general conditions of the barrel do not only have an influence on the wood's ability to release compounds, but also on the oxygenation level of the aged product (Carpena et al., 2020; Chanivet et al., 2020; Del Alamo-Sanza & Nevares, 2018; Herrera et al., 2020). The porosity of this type of wood allows a continuous and gentle micro-oxygenation that facilitates the stabilisation of the wine's colouring matter through the various reactions undergone by the polyphenolic compounds that lead to the modification of the wine's physical-chemical and sensory characteristics (Del Alamo-Sanza & Nevares, 2018).

For the wine's composition changes and developments to take place it is to remain stored for a varying length of time inside very specific containers known as casks, which are generally considered as rather costly. Besides these two factors, i.e. sitting time and manufacturing cost, others are to be considered, such as the limited lifespan of the wood that is used to make the barrels or the menace by undesirable microorganisms of the *Brettanomyces* and *Dekkera* genera which may occasionally appear (Suárez et al., 2007). Evaporation is another phenomenon that takes place during barrel ageing and that may represent a considerable economic loss for wineries (Ruiz de Adana et al., 2005).

All of these factors have driven oenological research to seek alternative ageing methods that emulate traditional ageing while allowing to obtain a quality product in a shorter time with limited risks and lower costs.

In this regard, perhaps the most widespread methodology consists in adding wood shavings. The European Union, by means of Regulation (EC) 1507/2006, authorised the use of oak wood chips for winemaking as long as this is clearly stated on the labelling, thereby trying to prevent any fraud to consumers, as in most cases it is not easy to distinguish between a wine that has produced using wood chips and one that has been aged following more traditional methods. Among other less developed methodologies until present, we can find the use of ultrasound, microwaves, high pressure, electrical pulses or gamma radiation

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(Solar et al., 2021).

The use of chips for wine ageing can be enhanced by combining it with the injection of lesser amounts of oxygen into the vats. A process known as micro-oxygenation and where the amount of injected oxygen is to be strictly controlled. In the case of red wines, doses of between 3 and 9 mL L^{-1} per month favour the polymerisation of blue and red pigments and the condensation of pigments and anthocyanins with flavonoids, thus achieving colour stabilisation. On the other hand, high doses of added oxygen seem to cause oxidation problems. However, the ideal dose seems to depend on the size and the origin of the wood as well as on each particular wine (Del Álamo et al., 2010).

As ultrasound (US) is an economic, non-invasive and environmentally friendly technique, its use has triggered a considerable interest from the food industry, in general, and from the oenological industry, in particular. The use of this energy creates microbubbles inside the oenological product, which finally collapse and cause pressure and temperature to rise, thereby favouring the chemical reactions associated to ageing (Yıldırım & Dündar, 2017). This is particularly noticeable when low frequency (20 kHz) and high energy ultrasound is used (Tao, Garcia-Martín et al., 2014). This technique has been the subject of multiple studies in the oenological field in recent years, where not only the ageing stage but also other extractive stages such as pre-fermentation maceration, which is typical of red wine production, have been addressed (Sánchez-Córdoba et al., 2021).

As for red wine, it has been observed that ultrasound favours the polymerisation of phenols and promotes an increment in pyroanthocyanins and tannins (Ferraretto & Celotti, 2016; Fu et al., 2018; Sun et al., 2019). However, some studies have determined that the colour of ultrasound-treated wine was lighter than that of untreated wine (Del Fresno Flórez, 2019). Regarding volatile compounds, contradictory or unfavourable results have also been observed. Zhang et al. (2020) found that the degradation rate of the higher alcohols in the treated wine was enhanced, while Singleton and Draper (1963) observed a decrease in the wine ester content. Both results were in contradiction with those obtained by Cui et al. (2012) regarding a Riesling white wine.

Jiménez-Sánchez et al. (2020) conducted a series of studies in which Sherry vinegars were aged in an accelerated manner through the combined use of ultrasound, oak wood chips from different origins and micro-oxygenation. The use of ultrasound for a short period of time (4–21 days) allowed to obtain vinegars with similar characteristics to those exhibited by vinegars which had been traditionally aged for several months (2–6 months). Other studies have applied ultrasound also for the ageing of other alcoholic products, such as Balcerek et al. (2017) who studied the effect of ultrasound on plum distillates. The same methodology has been successfully applied at laboratory scale to grape distillates, where the distillates circulated through a capsule packed with American oak wood chips (Schwarz et al., 2014). This technique successfully shortened the ageing time from 18 months to just 30 days while a highly valuable organoleptic product was obtained. All of this has led to considering this technique as an emerging method to improve ageing, since it favours and accelerates the typical chemical reactions that take place over this stage, such as Maillard or esterification reactions (Yu et al., 2021).

In view of this background, in the present study, a number of accelerated ageing tests were carried out on a Tempranillo red wine where micro-oxygenation and ultrasound as well as oak wood chips from different origins were employed. The main objective was to establish if the use of US could accelerate the ageing of this particular red wine, producing a wine with good organoleptic properties and well evaluated from a sensory point of view. Phenolic compound content, volatile profile and a sensory evaluation by a tasting panel were the analytical parameters considered in our study.

2. Materials and methods

2.1. Sample ageing

2018 vintage unaged Tempranillo wine from a winery located at the oenological production area of Jerez de la Frontera in Southwester Spain was used for the present study (pH, 3.4; total acidity, 6 gL^{-1} tartaric acid; 25 mgL^{-1} free SO_2).

The wine was subjected to an accelerated ageing procedure at a pilot scale, using 2 m tall 50 L stainless steel tanks. Different experiments were carried out using 5 g L^{-1} of medium toasted American oak (A; *Quercus alba*), French oak (F; *Quercus petraea*) or Spanish oak (S; *Quercus pyrenaica*) chips. The last ones were supplied by Agrovín, (Alcázar de San Juan, Spain). The chip dosage was established in accordance with the studies carried out by Sánchez-Guillén (2016).

An oxygen supply of 20 mL O_2 $L^{-1}month^{-1}$ (Gómez-Plaza & Bautista-Ortín, 2019) was administered by means of a DosiOx micro-oxygenator (Agrovín).

Ultrasound energy with a power of 1 KW and a frequency of 28 KHz (Tao, García-Martín et al., 2014) was applied to each tank by means of an Ultratechno ultrasound generator (Ultrasonidos Lover, S.A., Valencia, Spain), using successive 5-min operating cycles and 55-min standby periods. The process was continued for 31 days and samples were taken on the 0 (non-aged wine), 4th, 7th, 14th, 21st and 31st day (USMI samples). The samples, in 250 mL bottles, were refrigerated at 4 °C until their subsequent analysis.

Over the whole period, a number of control tanks were used, which contained the same chip dosage and were also subjected to micro-oxygenation, although no ultrasound was applied to them (MI samples). These were also sampled at the same times.

All the processes were carried out in duplicate at 25 °C controlled temperature.

2.2. Determining phenolic compounds content

Each sample's content of low molecular weight polyphenols was determined in duplicate by UPLC (Schwarz et al., 2009). An Acquity UPLC equipment (Waters, Milford, MA, USA) coupled to a photodiode array ultraviolet–visible detector was used. The chromatographic column used was Acquity UPLC BEH C18 of 100 mm × 2.1 mm inner diameter and with a particle size of 1.7 μm - Prior to their analysis, the wines were filtered through 0.22 μm (nylon syringe filters; Millipore, Bedford, MA, USA).

On the one hand, for identification purposes, the retention time and ultraviolet visible spectra of the low molecular weight polyphenolic compounds were compared against those of the commercial available standards. On the other hand, for their quantification, individual calibration curves were generated for each compound using the closest wavelength to its maximum absorption (280 or 320 nm).

Each sample's total anthocyanin content was determined according to its absorbance at 520 nm in an acid medium. Thus, 0.2 mL of each sample was placed into 10 mL tubes, to which 3.8 mL of 1M HCL solution (Panreac, Barcelona, Spain) was added. The tubes were then shaken and let to settle for 1 h. Then, absorbance at 520 was measured against a blank of 1M HCl (Iland et al., 2004). The results were expressed as mgL^{-1} malvidin-3-glucoside equivalents (molar extinction coefficient $28000M^{-1}cm^{-1}$; molecular weight 529 $gmol^{-1}$).

As for total tannins, they were quantified using the methylcellulose tannin precipitation method (MCP) (Mercurio & Smith, 2008; Sarneckis et al., 2006). The results were expressed in mgL^{-1} epicatechin equivalents.

Measurements were performed in triplicate and using an UV-VIS spectrophotometer (Spectronic Helios, Thermo Electron Corporation, Waltham, USA).

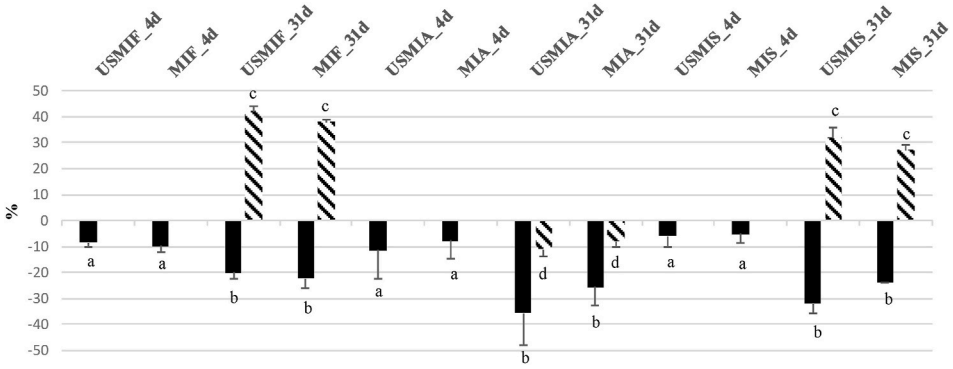

Fig. 1. Changes in total anthocyanins and tannins for the wines accelerately aged with Spanish (S), American (A), and French (F) chips, microoxygenation (MI) and with (US) and without ultrasound respect to the initial wine (non-aged wine); 4d: 4 days; 31d: 31 days. Different letters indicate significant differences (Tukey's test at $p < 0.05$).

2.3. Determining volatile compounds

The volatile compounds were determined in duplicate by means of a previously optimized SBSE-GC-MS methodology (Guerrero et al., 2006, 2007), which was specifically validated for its application to wines (Herrera et al., 2020). 25 mL of sample together with 5.86 g NaCl and 84 μL of 4-methyl-2-pentanol (2.27 gL^{-1} in Milli-Q water, 15% (vv^{-1}) ethanol content) were used. PDMS-coated stir bars (Gerstel, Mülheim a/d Ruhr, Germany) were used for the extraction at 1250 rpm for 120 min at a temperature of 25 °C. After completing the extraction process, the stir bars were thermally desorbed in an Agilent 6890 GC-5973N MS chromatographic system (Agilent Technologies, Little Falls, DE, USA) equipped with a DBWax (J&W Scientific, Folsom, CA, USA.) 0.25 μm thick coated 60 m × 0.25 mm inner diameter capillary column. The peaks identification was carried out by comparison with the Wiley 7N Edition Library (Wiley Registry of Mass Spectral Data, 7th Edition, 2000) at 90% minimum match and confirmed by Linear Retention Indices (LRI). The Linear Retention Indices were first determined and then compared with those reported by the literature. The different volatile compounds identified were quantified by using the individual calibration curves developed for each of them from commercial standards (Sigma-Aldrich (St. Louis, MO, USA)).

2.4. Sensory evaluation

The sessions were carried out in a standardized tasting room which intends to minimize any external influence on the panel assessments. The room temperature was set at 22 °C. The evaluations were carried out by 9 panel members with medium to high experience level in the sensory assessment of oenological products (ISO 8589-2007, International Organization for Standardization (ISO), 2007)).

Forty mL of each sample was poured into black glass wine glasses (International Organization for Standardization (ISO), 1977) and covered with a glass lid to stabilise the headspace for at least 10 min prior to tasting. Nose and mouth perceptions were recorded.

Descriptive as well as ranking tests were run (ISO 8587:2006, International Organization for Standardization (ISO), 2006). The descriptors were selected according to the literature, and 9-point interval numerical rating scales were used (International Organization for Standardization (ISO), 2003).

Table 1
Mean concentrations of phenolic compounds (mg/L) obtained for the red wines submitted to accelerated ageing with and without US for 31 days employing different oak types. ANOVA results for the variables "Oak", "Time", and "US".

Polyphenols	Non-aged wine	American oak	Spanish oak	French oak	ANOVA "time"		ANOVA "oak"		ANOVA "US"	
					F	*p*-value	F	*p*-value	F	*p*-value
Gallic acid	2.92 ± 0.34	4.12 ± 2.90^{a}	5.91 ± 1.01^{b}	4.34 ± 0.75^{a}	7.48	0.0000*	12.36	0.0000*	0.53	0,5896
Protocatechuic acid	1.81 ± 0.35	1.96 ± 0.27	2.41 ± 1.71	2.55 ± 0.47	5.03	0.0004*	2.77	0.0680	0.03	0.9739
Caftaric acid	7.04 ± 0.62	7.19 ± 0.43^{a}	6.18 ± 0.58^{b}	6.73 ± 0.54^{c}	6.36	0.0000*	54.42	0.0000*	3.83	0.0256
Protocatechualdehyde	2.87 ± 1.01	3.10 ± 0.71^{a}	2.44 ± 0.87^{b}	1.46 ± 0.20^{c}	31.43	0.0000*	105.86	0.0000*	0.71	0.4944
p-Hydroxybenzoic acid	1.16 ± 0.25	1.25 ± 0.22	1.21 ± 0.12	1.28 ± 0.08	4.72	0.0007*	0.56	0.5759	4.02	0.0213*
Tyrosol	18.24 ± 2.35	21.36 ± 4.79^{a}	17.75 ± 4.10^{b}	18.22 ± 2.73^{b}	0.40	0.8505	7.68	0.0090*	0.59	0.5571
Catechin	5.42 ± 0.48	5.49 ± 2.63^{a}	1.49 ± 2.69^{b}	2.22 ± 0.43^{b}	1.51	0.1943	35.30	0.0000*	0.78	0.4596
Caffeic acid	12.51 ± 2.38	12.22 ± 1.76^{a}	8.50 ± 1.90^{b}	10.85 ± 1.14^{c}	11.73	0.0000*	80.49	0.0000*	4.97	0.0091
Vanillic acid	15.38 ± 1.62	19.19 ± 2.84^{a}	17.38 ± 2.12^{b}	16.64 ± 1.71^{b}	15.74	0.0000*	23.31	0.0000*	1.25	0.2911
p-Hydroxybenzaldehyde	0.71 ± 0.32	1.12 ± 0.27^{a}	0.77 ± 0.29^{a,b}	0.76 ± 0.18^{b}	2.04	0.0805	22.44	0.0000*	1.21	0.3023
Syringic acid	2.78 ± 0.97	3.34 ± 1.00^{a}	3.04 ± 0.90^{a,b}	2.62 ± 0.40^{b}	1.97	0.0908	4.08	0.0203*	0.63	0.5374
Epicatechin	27.11 ± 2.96	31.90 ± 9.76	27.11 ± 17.97	28.28 ± 10.96	25.58	0.0000*	2.03	0.1377	1.56	0.2150
Vanillin	6.89 ± 1.66	7.76 ± 1.13^{a}	5.58 ± 1.53^{b}	4.51 ± 0.73^{c}	20.03	0.0000*	122.99	0.0000*	1.33	0.2688
cis-p-Coumaric acid	19.14 ± 1.76	18.13 ± 1.33^{a}	14.43 ± 2.94^{b}	18.75 ± 1.85^{a}	5.02	0.0004*	41.54	0.0000*	8.84	0.0003*
Ethyl gallate	10.15 ± 1.42	14.18 ± 2.46^{a}	8.42 ± 2.03^{b}	5.70 ± 0.92^{c}	7.65	0.0000*	173.44	0.0000*	3.68	0.0293*
Syringaldehyde	2.08 ± 0.31	2.55 ± 0.56^{a}	4.80 ± 1.02^{b}	3.35 ± 1.23^{c}	7.14	0.0000*	90.49	0.0000*	5.84	0.0042*
Ferulic acid	1.86 ± 0.26	1.37 ± 0.52	1.40 ± 0.41	1.48 ± 0.22	2.89	0.0186*	0.26	0.7721	1.89	0.1566
m-Coumaric acid	1.82 ± 0.90	1.93 ± 0.56	1.81 ± 0.58	1.86 ± 0.47	0.90	0.4850	0.57	0.5684	0.10	0.9045
Coniferyl aldehyde	1.05 ± 0.67	1.22 ± 0.30^{a}	0.95 ± 0.35^{b}	0.59 ± 0.22^{c}	2.67	0.0272*	39.79	0.0000*	5.45	0.0059*
Sinapaldehyde	0.89 ± 0.38	1.17 ± 0.35^{a}	0.65 ± 0.34^{b}	0.65 ± 0.23^{b}	0.68	0.6378	27.49	0.0000*	1.01	0.3669
Kampherol-3-glucoside	14.49 ± 1.29	21.87 ± 1.77^{a}	19.25 ± 2.58^{b}	17.06 ± 7.15^{c}	2.85	0.0199*	16.87	0.0000*	7.05	0.0015*
Ethyl caffeate	4.32 ± 1.14	3.90 ± 0.52^{a}	3.30 ± 1.17^{b}	4.10 ± 0.97^{a,b}	1.61	0.1666	3.80	0.0262	3.36	0.0395*
Ethyl *p*-coumarate	5.30 ± 0.31	4.92 ± 0.41^{a}	3.83 ± 0.96^{b}	5.00 ± 0.60^{a}	5.55	0.0002*	31.08	0.0000*	6.53	0.0023*

Different letters in the same row indicate significant differences according to Tukey's test ($p < 0.05$) for different oak woods (American, French and Spanish oak).
* Significant values ($p < 0.05$).

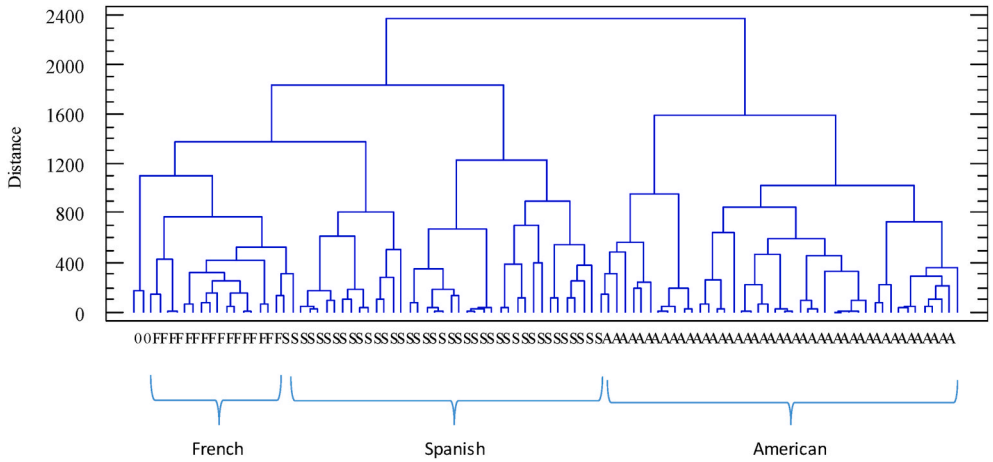

Fig. 2. Clusters Analysis on phenolic content for all wines A: American chips; S: Spanish chips; F: French chips.

The restrictions imposed by the Covid 19 health crisis only allowed the sensory study of the final samples corresponding to the trials with American oak wood chips.

2.5. Statistical analysis

The data obtained were subjected to Analysis of Variance (ANOVA), Cluster Analysis (CA), and Principal Component Analysis (PCA). The statistical program Statgraphics Centurion, version 19.0 (Statpoint Technologies Inc., Virginia, USA) was used for these purposes.

3. Results and discussion

3.1. Phenolic compounds

Fig. 1 shows the percentage of anthocyanin and tannin loss/gain observed in each sample with respect to the initial content in the wine together with each sample's standard deviation. As we can see, anthocyanin contents experienced clear losses in all of the samples considered. Such losses were significantly higher with longer treatment times (31d, ANOVA, $p = 0.0050$), while no significant differences could be noticed when the wine samples subjected to just micro-oxygenation and chips were compared against those wine samples that had also been subjected to ultrasound for the same length of time (ANOVA, $p = 0.1365$). Nor, in those cases, were any significant differences observed between the wines aged with different origin oak wood chips (ANOVA, $p = 0.1699$), all of which registered similar total anthocyanins losses when treated for the same length of time. No significant double interactions among the considered factors were observed for these losses.

In terms of tannin content, wines aged with French and Spanish oak experienced significant gains after 31 days of treatment in relation to wines aged in American oak (ANOVA, $p = 0.0123$), which presented losses in this polyphenolic fraction. In relation to the use of US, the changes observed for the different types of oak wood were only somewhat higher, without statistical difference (ANOVA, $p = 0.9334$) in those wines aged with US.

It seems, therefore, that the anthocyanins present in the wine, and to which it owes its colour, experienced significant losses during the accelerated ageing process. This fact that could be explained by the scarce presence of tannins that would enable their stabilisation based on the favourable environmental conditions created by micro-oxygenation, or by an excess of oxygen that would cause their oxidation and precipitation. The bibliographic studies indicate that the ideal dose of oxygen to be applied in micro-oxygenation procedures depends on the application length of time and on the capacity of the sample to combine its polyphenolic content with the oxygen supplied. Thus, doses up to 90 mg oxygen $L^{-1}month^{-1}$ have been considered as acceptable (Gómez-Plaza & Bautista-Ortín, 2019), although it has also been observed that high oxygen doses can give rise to high molecular weight molecules with low stability in the solution, which would lead to their precipitation and consequently to a faded wine colour (Cano-López et al., 2006).

As for the individualized analysis of the low molecular weight polyphenol content, Table 1 shows the mean content together with its standard deviation corresponding to the initial wine (non-aged wine) and to the accelerately aged wines with chips from different origin. It also includes the results derived from the analysis of variance to which these data were subjected, where time, treatment with and without ultrasound, and the origin of the oak wood chips used were the independent variables. As can be seen, time and oak type were the most significant variables. Thus, the ultrasound variable (US) was only significant for *cis-p*-coumaric acid, ethyl gallate, syringaldehyde, coniferyl aldehyde, kampherol-3-glucoside, ethyl caffeate, and ethyl *p*-coumarate, with higher values recorded for all of them, with the exception of the ethyl gallate content in the samples subjected to ultrasound (data not shown). It should be noted that all of these polyphenolic compounds, along with others, have been demonstrated to be clear markers of ageing in oak for a variety of oenological products (Chanivet et al., 2020; Del Álamo et al., 2010; Jimenez-Sánchez et al., 2020). Several studies have shown the effectiveness of ultrasound to accelerate the extraction of polyphenols from maritime pine wood (Meullemiestre et al., 2016) or spruce bark (Ghitescu et al., 2015). In the case of oak wood, some authors have also observed a substantial increment in the release of the polyphenols from oak into the wine when ultrasound is applied, however, this was dependent on factors such as acoustic energy density (W L^{-1} of wine) and temperature, with better results when both factors were enhanced (Tao, Zhang, et al., 2014).

In terms of oak wood origin and time, both variables were found to be significant for a large number of polyphenolic compounds, with greater concentrations being generally observed as the ageing time was increased (Table 1). However, the greatest differences were detected after only 4 days of ageing, which highlights the rapid extraction kinetics. This fact had already been noted in a previous study on accelerated Sherry vinegar ageing under similar conditions (Jiménez-Sánchez et al., 2020). In the case of the present work, this high extraction kinetics could be favoured by the alcohol content in the samples (12.5% vv-1) together with their low pH (3.4) (Gómez-Plaza & Bautista-Ortín, 2019) and even by the agitation created within the liquid as a result of the micro-oxygenation procedure.

Previous studies had already revealed that a decrease in pH did favour polyphenol extraction when using ultrasound (Fernandes et al., 2020).

As for the type of oak, in general, the red wine samples that had been

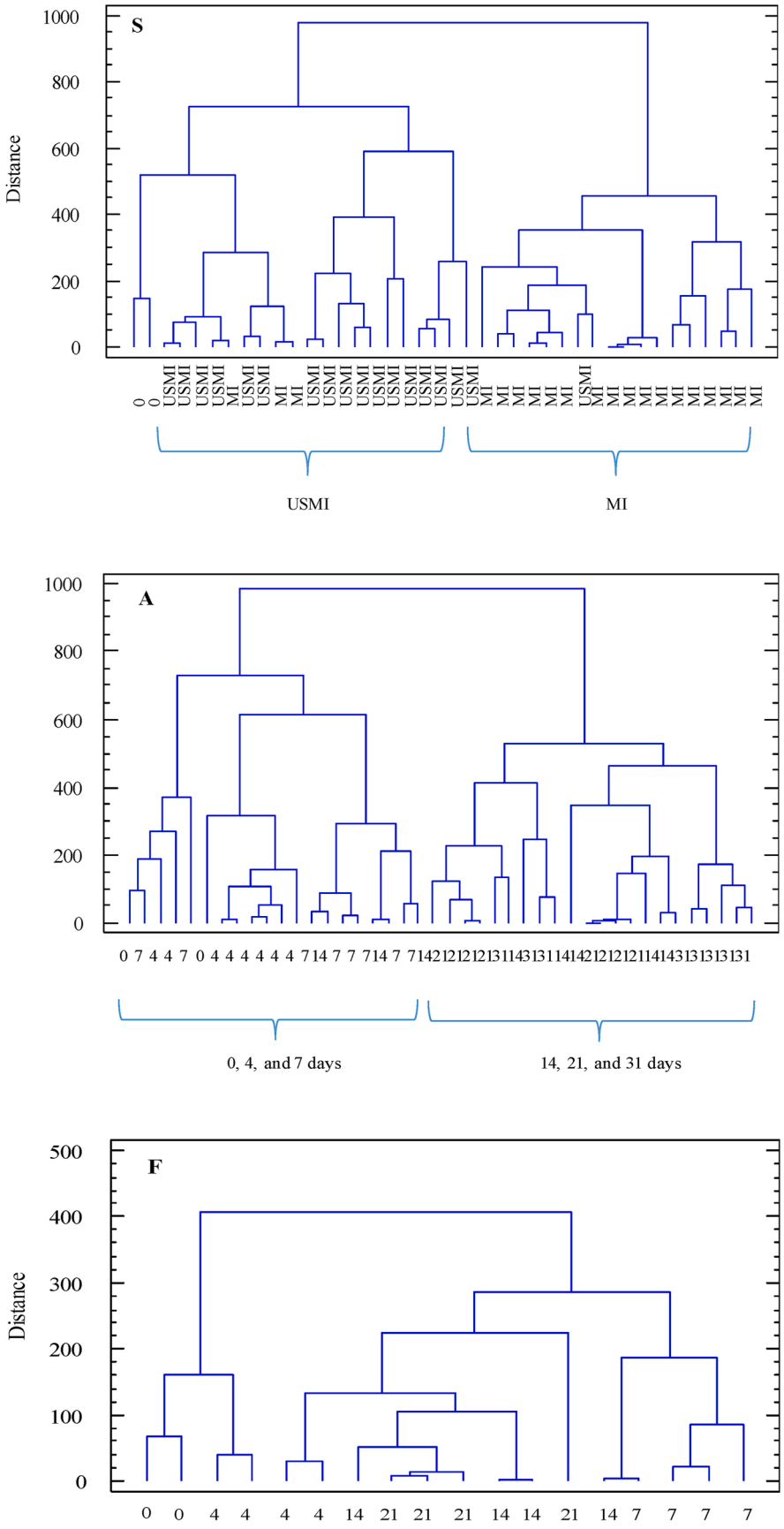

Fig. 3. Clusters Analysis on phenolic content for each wine according to type of chip. A: American chips; S: Spanish chips; F: French chips.

Table 2
Mean Concentrations of volatile compounds (mg/L) obtained for the red wine samples submitted to accelerated ageing for 31 days employing different oak types, microoxygenation and with and without ultrasound. ANOVA results for the variables "Oak origin" and "Time" and "US".

Volatile compounds	Non-aged wine	American oak		French oak		Spanish oak		ANOVA "time"		ANOVA "oak"		ANOVA "US"	
								F	*p*-value	F	*p*-value	F	*p*-value
Isobutyl acetate	0.0287 ± 0.0153	0.0286	±0.0153	0.0339	±0.0097	0.0287	±0.0241	4.82	0.0017*	0.00	0.9984	4.54	0.0367*
Ethyl butyrate	0.2249 ± 0.1942	0.0810	±0.0334^{a}	0.1535	±0.0350^{b}	0.1131	±0.0949^{a,b}	7.36	0.0001*	5.59	0.0056*	0.21	0.6482
Isobutanol	34.24 ± 3.49	28.72	±15.76^{a}	41.97	±25.34^{a,b}	44.31	±14.85^{b}	4.76	0.0019*	8.26	0.0006*	1.21	0.2756
Isoamyl acetate	0.3031 ± 0.0069	0.0285	±0.0243	0.0476	±0.0031	0.0488	±0.0050	5.68	0.0005*	0.06	0.9464	2.18	0.1441
Ethyl valerate	0.0514 ± 0.0122	0.0285	±0.0243^{a}	0.0476	±0.0031^{b}	0.0488	±0.0050^{b}	10.83	0.0000*	22.78	0.0000*	0.67	0.4169
Ethyl caproate	0.2242 ± 0.0024	0.0822	±0.0347	0.1432	±0.0345	0.0907	±0.1049	7.46	0.0000*	1.94	0.1513	0.17	0.6812
Hexyl acetate	0.0049 ± 0.0009	0.0044	±0.0002	0.0044	±0.0004	0.0045	±0.0004	4.36	0.0033*	1.27	0.2870	0.15	0.6962
cis-3-hexenyl acetate	0.0207 ± 0.0081	0.0128	±0.0042	0.0139	±0.0045	0.0129	±0.0083	3.01	0.0236*	0.20	0.8202	0.58	0.4496
1-Hexanol	2.01 ± 0.29	0.7497	±0.0958	0.7766	±0.1725	0.8100	±0.4575	3.62	0.0096*	0.48	0.6186	0.02	0.8951
Ethyl octanoate	0.1676 ± 0.0536	0.0875	±0.0336	0.0925	±0.0269	0.0853	±0.0733	5.56	0.0006*	0.08	0.9216	0.11	0.7392
Acetic acid	2734.9 ± 362.5	362.2	±238.2	235.6	±250.4	359.6	±269.3	5.42	0.0007*	0.06	0.9446	0.24	0.6252
2-Furaldehyde	0.7620 ± 0.1037	0.6023	±0.0144^{a}	0.7489	±0.1047^{b}	0.6673	±0.1327^{c}	2.35	0.0626	11.26	0.0001*	0.13	0.7207
Benzaldehyde	0.0096 ± 0.0041	0.0129	±0.0048^{a}	0.0184	±0.0071^{b}	0.0127	±0.0033^{a}	2.68	0.0387*	6.86	0.0019*	0.05	0.8188
Isobutyric acid	9.44 ± 1.27	5.39	±1.12^{a}	7.78	±3.64^{b}	5.49	±1.16^{a}	5.91	0.0004*	10.19	0.0001*	1.20	0.2764
γ-Butyrolactone	11.79 ± 2.76	13.26	±9.39	19.51	±8.24	13.47	±14.49	3.44	0.0126*	0.76	0.4698	0.03	0.8626
Isovaleric acid	2.37 ± 0.47	1.13	±0.39^{a}	1.87	±0.52^{b}	1.28	±0.68^{a}	5.51	0.0006*	5.83	0.0045*	0.03	0.8522
Diethyl succinate	3.71 ± 1.01	2.09	±0.18	2.64	±0.70	2.46	±1.40	4.26	0.0038*	1.58	0.2129	0.45	0.5036
Benzyl alcohol	0.7714 ± 0.0415	0.7833	±0.1066	0.8066	±0.0773	0.8057	±0.1065	3.27	0.0161*	0.19	0.8279	0.02	0.8862
trans-Whiskeylactone	0.0166 ± 0.0081	0.0206	±0.0081^{a}	0.0296	±0.0245^{a,b}	0.0385	±0.0204^{b}	7.06	0.0001*	12.25	0.0000*	3.78	0.0557
Benzenethanol	14.79 ± 2.28	9.15	±1.65	10.71	±2.32	9.58	±4.96	3.81	0.0073*	0.39	0.6781	0.03	0.8533
4-Ethylguaiacol	0.0233 ± 0.0076	0.0303	±0.0008^{a}	0.0305	±0.0007^{a}	0.4787	±0.2585^{b}	6.39	0.0000*	93.26	0.0000*	0.67	0.4142
Octanoic acid	1.56 ± 0.37	0.9161	±0.2252	0.8497	±0.1621	1.01	±0.6289	4.01	0.0055*	1.51	0.2274	0.09	0.7595
Nonanoic acid	0.0164 ± 0.0055	0.0212	±0.0191	0.0153	±0.0046	0.0150	±0.0058	1.32	0.2719	2.28	0.1102	0.00	0.9494
Eugenol	0.0024 ± 0.0011	0.0028	±0.0014^{a}	0.0028	±0.0021^{a}	0.0062	±0.0014^{b}	11.24	0.0000*	44.82	0.0000*	6.79	0.0112*
4-Ethylphenol	0.0247 ± 0.0087	0.0103	±0.0068^{a}	0.0174	±0.0153^{a,b}	0.0115	±0.0071^{a}	2.61	0.0428*	3.59	0.0326*	2.13	0.1492
Decanoic acid	0.2892 ± 0.0622	0.1506	±0.0508	0.1509	±0.0382	0.1677	±0.1243	5.19	0.0010*	0.66	0.5179	0.06	0.7998

Different letters in the same row indicate significant differences according to Tukey's test ($p < 0.05$) for different oak woods (American, French and Spanish oak).
* Significant values ($p < 0.05$).

aged with American oak wood chips showed higher concentrations of most of the polyphenolic compounds considered, while the red wine samples aged with Spanish oak wood chips presented an intermediate polyphenolic content level between those of American and French oak wine samples. This fact was corroborated by the significant double interactions obtained from the ANOVA study. The interaction 'time-type of oak' was significant for several polyphenols, such as *p*-hydroxybenzoic acid, tyrosol, catechin, *p*-hydroxybenzaldehyde, syringaldehyde, and *m*-coumaric acid. Some decreases were observed, in general, for those wines aged in Spanish and French oak and some increases for those ones aged in American oak as the ageing time increased.

These results are in line with those reported by other authors on the traditional ageing of red wines in American, French and Spanish oak casks (Fernández de Simón et al., 2003).

All of these findings were also supported by the cluster analysis to which the polyphenolic data were subjected (Fig. 2). In the dendogram obtained (Ward's method and squared Euclidean distance) three clear groups can be observed, each according to the type of oak wood chips, with greater similarity between Spanish and French oak. On the other hand, the wine aged with American oak wood chips was clearly different from the other two. In view of these results, each wine sample was studied separately by cluster analysis according to the origin of the oak wood used and attending to its polyphenolic content. Fig. 3 shows the dendograms obtained for each of the oak types in the study. As can be seen, Spanish oak exhibited a grouping that distinguishes it from the other two types of oak. In the case of Spanish oak (S), this was explained by the treatment or non-treatment with ultrasound, while American (A) or French (F) oak samples would group according to their ageing time, which evidences a lesser influence from the US treatment on the samples that had been aged with these two oak types. This led us to affirm that the use of ultrasound during red wine ageing with oak wood chips seems to be conditioned by the origin of the oak wood.

Other authors have found that the release of polyphenolic compounds from wood chips into wine during accelerated ageing trials would depend on the origin of the wood, with larger releases of ellagitannins from French versus American oak (Jourdes et al., 2011).

3.2. Volatile compounds

Concerning the volatile constituents, a total of 26 compounds were

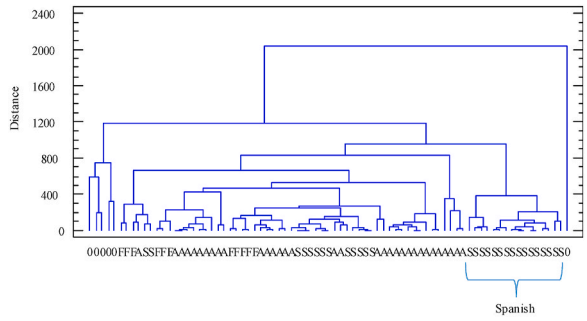

Fig. 4. Clusters Analysis on volatile content for all wines A: American chips; S: Spanish chips; F: French chips.

successfully identified and quantified. The same as with the polyphenolic content, the volatile compounds data obtained were subjected to an ANOVA to determine the influence of the three factors studied: oak type, time and ultrasound. As can be seen in Table 2, time length was the most influential variable, followed by oak wood type. Ultrasound (US) was only significant with respect to isobutyl acetate and eugenol ($p < 0.05$). In relation to double interactions among the factors studied, the most significant interaction was again 'time-type of oak' for several compounds such as *cis*-3-hexenyl acetate, 1-hexanol, isovaleric acid, benzyl alcohol, 4-ethylguaiacol, octanoic acid, and decanoic acid, among others, but a clear behavior was not observed for the different types of oak considered in this study.

These facts reinforced the results observed for polyphenolic content, where the US factor was also reported as the least significant. The pairwise comparison study, Tukey's test, showed the rapid extraction kinetics of the accelerated ageing method, since no significant volatile compound extraction differences were registered between the samples aged for a period of 4–31 days. That is, the largest significant differences appeared when comparing the concentrations after only 4 days of ageing with respect to unaged wine. These differences remained stable as time was increased, so that no significant increments were observed with respect to most of the volatile compounds considered (data not shown). Other authors have already demonstrated the acceleration of the wood volatile compounds extraction kinetics when using ultrasound-assisted methods (Jiménez-Sánchez et al., 2020).

Table 2 presents the results of mean concentrations together with their standard deviations corresponding to the initial wine (non-aged wine) and to the different samples of accelerated-ageing red wines obtained using the three types of oak wood. It can be seen that French oak, together with Spanish oak, provided the highest concentration of several of the volatile compounds derived from wood (2-furaldehyde, benzaldehyde, 4-ethylguaiacol, *trans*-whiskeylactone or eugenol) (Herrera et al., 2020). In a previous study on traditional and accelerated ageing of vinegars, higher concentrations of these wood-derived compounds were obtained when samples had been aged using French oak wood. It was also observed that in the case of Spanish oak, the vinegars would more closely resemble those aged in French oak as the ageing time was increased (Jiménez-Sánchez et al., 2020). In that study, the extraction kinetics of the volatile compounds from Spanish oak seemed to be more similar to that of French oak than to the kinetics observed when American oak wood was used under similar average concentrations.

The lower concentrations of many volatile compounds observed in American oak (Table 2) are in agreement with those reported by previous studies, both regarding either samples of American oak vinegars (Chanivet et al., 2020) or sweet wines (Herrera et al., 2020).

Similarly to the analysis of the polyphenolic compounds, the volatile compounds were subjected to cluster examination in order to determine any similarities and disparities between the different red wine samples.

The resulting dendogram (Fig. 4), which was obtained using Ward's method and the squared Euclidean distance, allowed us to identify a small group of samples corresponding to the wine samples aged with Spanish oak, which rather noticeably differed from the rest of the red wine samples while keeping a rather close similarity between them.

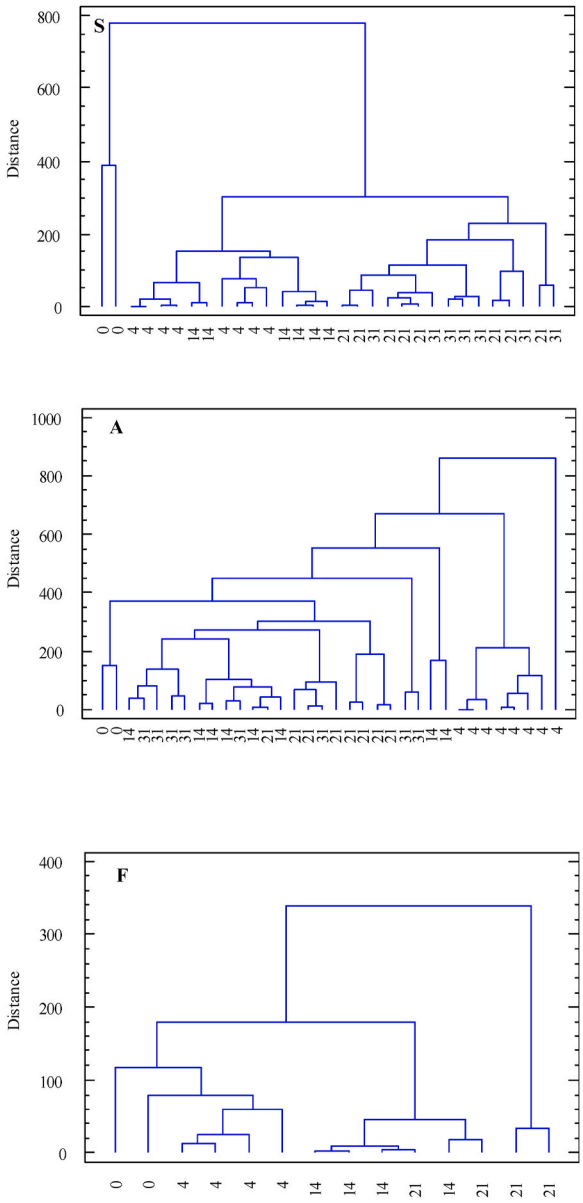

Fig. 5. Clusters Analysis on volatile content for each wine according to type of chip. A: American chips; S: Spanish chips; F: French chips.

When the wine samples were examined separately according to the type of oak wood (Fig. 5), a clear grouping could be observed that seemed to depend on the ageing time variable. This was particularly noticeable in Spanish and French oak wood samples. On the other hand, American oak wood aged wines presented a grouping that seemed to indicate either possible losses or degradation of certain volatile compounds. Such volatile compounds would have been extracted in the first stages of the accelerated ageing and the subsequent loss or degradation of the volatile compounds would result in a closer volatile content similarity between red wine samples aged for 14–31 days, while a clear differentiation could be noted when compared against the samples that had been aged for only 4 days.

3.3. Data set multivariate analysis

In order to monitor the influence as a whole from the different factors

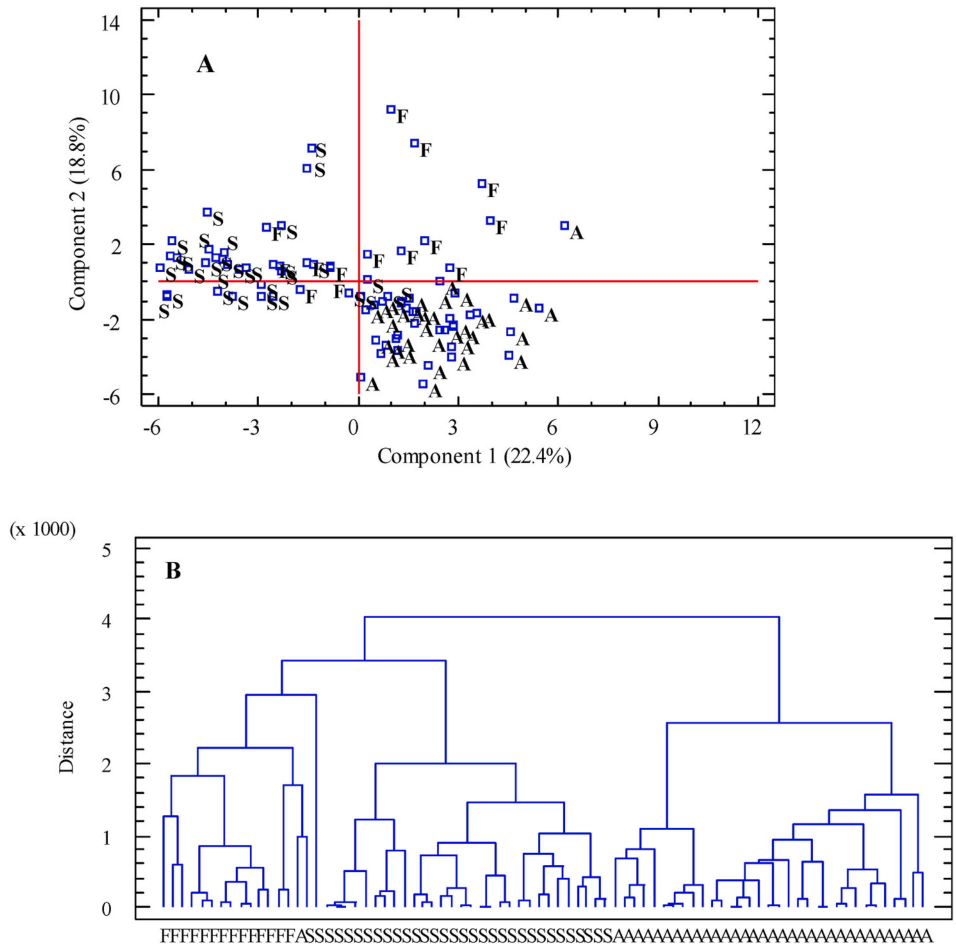

Fig. 6. Principal component analysis (A) and clusters analysis (B) on phenolic and volatile contents for all wines.

considered on the analytical parameters, a principal component analysis (PCA) was conducted based on the data obtained for polyphenolic compounds as well as for volatile compounds. The PCA indicated 11 principal components (PCs) with an eigenvalue >1 that explained 82.95% of the variability. Fig. 6A shows the distribution of the samples within the space formed by the two first PCs. These two first PCs explained 41.18% of the variability. It should be noted that PC1 (22.4% total variability) allowed to separate the samples that had been aged using Spanish oak wood, which was located in the negative area of this PC, from those aged with American or French oak wood chips. The PC2, on the other hand, allowed to separate the American oak aged samples — with negative values — from the French and Spanish oak aged samples. Regarding the PC1, the compounds that exhibited a higher weight were mostly polyphenols, such as caffeic acid, caftaric acid, protocatechualdehyde, *p*-hydroxybenzaldehyde, ethyl *p*-coumarate, vanillin and *p*-coumaric acid, all of them with positive signs, together with syringaldehyde, with negative sign. In the case of the PC2 (18.8% total variability), most of the higher weight compounds were volatiles such as 1-hexanol, furfuraldehyde, γ-butyrolactone, isovaleric acid, ethyl succinate, benzyl alcohol, 2-phenylethanol, and decanoic acid, all of which had positive signs. The combined study of phenols and volatile compounds allowed to corroborate what had already been observed in their previous separate studies: the wine aged using French oak was similar to that aged with American oak in terms of polyphenolic content (mostly related to the PC1). However, in terms of volatiles, American oak wood samples, in the negative area of the PC2 because of a greater influence from volatile compounds in this PC, exhibited a different behaviour from those registered for French or Spanish oak wood aged samples. The subsequent cluster analysis to which all the phenolic and volatile data were subjected revealed two clear groups attending to the type of oak wood (Fig. 6B): the first one would correspond to the red wine samples aged using American oak and the other group would be formed by the wines aged with Spanish or French oak wood chips. All of which, further confirms the results that had been previously obtained by the PCA.

3.4. Sensory analysis

With respect to the descriptive tests, in order to ensure the homogeneity of the panel, the evaluations that were confirmed as discrepant data (Q-Dixon) were discarded. Then, the final data were subjected to an analysis of variance.

For the trials that were carried out using American oak chips, as can be concluded from the analysis of Table 3, that contains the mean olfactory and taste scores that had been granted to the samples submitted for study, no significant differences were perceived regarding any of the descriptors considered ($p > 0.05$). However, given the dispersion of the tasting panel's judgments (somewhat greater than in the physical-chemical analysis), the differences with a low p_{anova} would not be discarded, even if they were greater than the threshold that confirms statistical significance. Red and black fruits, and floral descriptors showed slight differences, with higher values in the case of the starting wine (not aged) and in the wines corresponding to the accelerated ageing tests without ultrasound. It should also be noted that the perceived intensity of oak odour was greater in the case of the accelerated-aged samples.

To evaluate the global olfactory and taste impressions, ranking tests were conducted (ISO 8587; 2006, International Organization for Standardization (ISO), 2006) According to Friedman's criterion, for 9 judges

Table 3
Mean values for the descriptors considered in the sensory study on the wines aged with US, microoxygenation and American chips for 31 days.

Descriptor	Wines					ANOVA
	USMIAa	USMIAb	MIAa	MIAb	Non-aged wine	P
Aromatic intensity	4.1 ± 0.8	3.5 ± 1.3	3.8 ± 1.5	4.6 ± 1.4	4.3 ± 1.5	0.2876
White fruit	0.8 ± 1.0	0.7 ± 1.3	1.8 ± 2.6	1.7 ± 2.2	1.0 ± 1.3	0.9138
Red and black Fruit	2.9 ± 0.8	2.9 ± 0.9	2.8 ± 1.2	3.6 ± 0.5	3.3 ± 1.3	0.0611
Ripe fruit	2.1 ± 1.2	2.4 ± 0.5	3.8 ± 1.3	2.9 ± 2.0	2.3 ± 1.4	0.5036
Sweet. raisin	0.9 ± 1.1	0.9 ± 0.9	1.8 ± 1.6	1.0 ± 1.7	1.0 ± 0.8	0.4409
Dairy	1.1 ± 0.9	1.7 ± 1.1	1.1 ± 1.4	1.1 ± 1.1	2.0 ± 1.2	0.5066
Winy	2.8 ± 1.3	2.3 ± 1.3	3.1 ± 0.7	2.0 ± 1.3	3.1 ± 0.7	0.8393
Herbaceous	2.6 ± 1.8	2.4 ± 1.3	1.9 ± 1.7	1.9 ± 1.5	2.0 ± 1.6	0.8714
Floral	1.3 ± 1.1	0.7 ± 0.5	1.8 ± 1.2	1.5 ± 1.2	2.1 ± 1.8	0.0991
Fresh, balsamic	1.3 ± 1.2	0.9 ± 0.9	1.9 ± 1.3	1.1 ± 0.9	1.9 ± 1.6	0.1203
Defect	1.0 ± 1.4	2.9 ± 2.2	0.6 ± 0.5	0.2 ± 0.4	1.1 ± 1.3	0.1449
Oak	2.3 ± 1.1	2.3 ± 1.7	1.7 ± 1.6	2.4 ± 1.1	0.5 ± 0.5	0.2707
Olfactory impression	3.4 ± 2.1	1.8 ± 1.2	3.9 ± 1.8	4.0 ± 1.4	3.5 ± 1.4	0.1269
Acidity	5.0 ± 1.3	3.4 ± 1.6	2.9 ± 0.8	3.8 ± 1.0	3.4 ± 1.5	0.1270
Bitterness	2.4 ± 1.3	2.3 ± 2.1	2.1 ± 1.8	2.3 ± 1.0	2.0 ± 1.2	0.9583
Astringency	2.1 ± 1.6	2.3 ± 0.9	2.8 ± 1.4	2.7 ± 1.1	1.8 ± 1.6	0.6608
Body	1.9 ± 0.6	1.8 ± 0.5	2.1 ± 1.1	2.6 ± 1.2	2.4 ± 1.5	0.3001
Persistence	2.4 ± 1.3	2.4 ± 1.1	2.9 ± 1.4	3.4 ± 1.6	3.1 ± 1.6	0.6195
Gustatory impression	1.9 ± 0.8	2.0 ± 1.4	3.3 ± 1.6	4.6 ± 1.5	3.7 ± 1.4	0.2316

and 5 samples, the tabulated F value was 9.22 (95% certainty level). The values obtained for the samples evaluated were 13.96 for olfactory impression and 17.07 for gustatory impression, which means that there are differences in the perceived quality between some of the samples. Fisher's least significant difference test was applied to specifically identify which of the samples presented a differing quality. Their descending ranking is presented below according to the sums of the scores (in parentheses) and their differences (different superscripts indicate significant differences at $p < 0.05$): MIAb (34)^{a}> Control (non-aged wine) (33)^{a}> MIAa (29)^{a}> USMIAa (27)^{a}> USMIAb (12)^{b} were registered for olfactory impression, while MIAb (39)^{a}> MIAa (33)^{a}> Control (29)^{a,b}> USMIAa (17)^{b} = USMIAb (17)^{b} were recorded for gustatory impression.

It can be concluded from these results that in the case of American oak wood aged wine, the best sensory scores were granted to those samples that had been aged at an accelerated rate using micro-oxygenation and chips. On the other hand, the worst scores were obtained by those samples that had been subjected to the combined action of these two techniques plus ultrasound.

4. Conclusions

The research that has been carried out has highlighted the high extraction kinetics of both volatile and polyphenolic constituents from oak wood. This is so, even without the use of ultrasound, possibly because of the low pH and high alcoholic content of the starting wine used in this study and perhaps favoured by the agitation generated within the liquid by the micro-oxygenation procedure.

In the case of polyphenols, the effect of ultrasound on their concentration was proven to be dependent on the type of oak wood, so that it hardly had any effect on the wine polyphenol content when the wood used for ageing was either American or French oak.

Again, regarding volatile constituents, the ultrasound factor proved to be the least significant. In fact, noticeable concentration increments were registered after only 4 days of accelerated ageing, even in those wines that had not been subjected to ultrasound.

The type of wood, therefore, was a determining factor that conditioned the volatile and polyphenolic profile of the final wines, also in those cases where the wine samples had been aged at an accelerated rate using US.

The wine aged using Spanish oak wood presented a similar polyphenolic content to that of the wine aged with French oak wood. In terms of volatiles, it was the American oak wood aged wine, with a lower concentration of wood volatiles, the one that showed a different behaviour, with higher concentrations after longer ageing periods.

With regard to the sensory characteristics of the wine aged with American oak wood, it was observed that the use of ultrasound reduced the scores granted by the panel members with respect to those wines that had been obtained just by micro-oxygenation and chips. This fact corroborates the data that had been registered when analysing the samples' volatile profile.

From a sensory point of view, future studies should be conducted to confirm the behaviour of wine when subjected to accelerated ageing using Spanish or French oak wood.

Industrial relevance text

Nowadays, the employment of US in the oenological industry has an attractive scientific interest. However, the consequences of its use on wine's composition and organoleptic characteristics are not still totally solved. Therefore, this study examines the feasibility of this novel extraction technology, ultrasound, and their combination with better known technologies such as microoxygenation and wood chips, as potential method for the ageing of a red wine, with special dedication to phenolic and volatile compounds. The effect of using US to accelerate the ageing of a Tempranillo wine has been proven to mostly depend on the type of wood used, having this variable a remarkable effect on their phenolic content levels, indicating that the oenological relevance of this technique is depending on the wood origin. This fact should be taken into account in order to optimize a possible ageing process using US.

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Author contributions

M.J.S. conducted the experiments and carried out the statistical analysis. R.C. and E.D.G. supervised the experiments. M.C.R.D. carried out the sensory study and the sensory data treatment. R.C. wrote and edited the manuscript. All authors have read, reviewed and approved the final manuscript.

Author statement

I can confirm that this work is original, all authors have consented to its submission and have contributed to it.

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.

References

Apetrei, I. M., Rodríguez-Méndez, M. L., Apetrei, C., Nevares, I., Del Alamo, M., & De Saja, J. A. (2012). Monitoring of evolution during red wine aging in oak barrels and alternative method by means of an electronic panel test. *Food Research International, 45*(1), 244–249. https://doi.org/10.1016/J.FOODRES.2011.10.034

Balcerek, M., Pielech-Przybylska, K., Dziekońska-Kubczak, U., Patelski, P., & Strak, E. (2017). Changes in the chemical composition of plum distillate during maturation with oak chips under different conditions. *Food Technology and Biotechnology, 55*(3), 333–359. https://doi.org/10.17113/ftb.55.03.17.5145

Cano-López, M., Pardo-Minguez, F., López-Roca, & Jose María Gómez-Plaza, E. (2006). Effect of microoxygenation on anthocyanin and derived pigment content and chromatic characteristics of red wines. *American Journal of Enology and Viticulture, 57*(3), 325–331.

Carpena, M., Pereira, A. G., Prieto, M. A., & Simal-Gandara, J. (2020). Wine aging technology: Fundamental role of wood barrels. *Foods, 9*(9), 1–25. https://doi.org/10.3390/foods9091160

Chanivet, M., Durán-Guerrero, E., Barroso, C. G., & Castro, R. (2020). Suitability of alternative wood types other than American oak wood for the ageing of Sherry vinegar. *Food Chemistry, 316*, Article 126386. https://doi.org/10.1016/j.foodchem.2020.126386

Cui, Y., Lv, W., Liu, J. F., & Wang, B. J. (2012). Effect of different ending fermentation technologies on microbial-stability of Italian Riesling low alcohol sweet white wine. *Advanced Materials Research, 363–365*, 1165–1168.

Del Alamo-Sanza, M., & Nevares, I. (2018). Oak wine barrel as an active vessel: A critical review of past and current knowledge. *Critical Reviews in Food Science and Nutrition, 58*(16), 2711–2726. https://doi.org/10.1080/10408398.2017.1330250

Del Álamo, M., Nevares, I., Gallego, L., Fernández de Simón, B., & Cadahía, E. (2010). Micro-oxygenation strategy depends on origin and size of oak chips or staves during accelerated red wine aging. *Analytica Chimica Acta, 660*(1–2), 92–101. https://doi.org/10.1016/J.ACA.2009.11.044

Del Fresno Flórez, J. M. (2019). *Aplicación de ultrasonidos en la crianza sobre lías y envejecimiento de vinos tintos* [Universidad Politécnica de Madrid] http://oa.upm.es/54176/.

Fernandes, F. A. N., Fonteles, T. V., Rodrigues, S., de Brito, E. S., & Tiwari, B. K. (2020). Ultrasound-assisted extraction of anthocyanins and phenolics from jabuticaba (myrciaria cauliflora) peel: Kinetics and mathematical modeling. *Journal of Food Science & Technology, 57*, 2321–2328. https://doi.org/10.1007/s13197-020-04270-3

Fernández de Simón, B., Hernández, T., Cadahía, E., Dueñas, M., & Estrella, I. (2003). Phenolic compounds in a Spanish red wine aged in barrels made of Spanish, French and American oak wood. *European Food Research and Technology, 216*(2), 150–156. https://doi.org/10.1007/s00217-002-0637-4

Ferraretto, P., & Celotti, E. (2016). Preliminary study of the effects of ultrasound on red wine polyphenols. *CyTA - Journal of Food, 14*(4), 529–535. https://doi.org/10.1080/19476337.2016.1149520

Fu, X. Z., Zhang, Q. A., Zhang, B. S., & Liu, P. (2018). Effect of ultrasound on the production of xanthylium cation pigments in a model wine. *Food Chemistry, 268*, 431–440. https://doi.org/10.1016/J.FOODCHEM.2018.06.120

Ghitescu, R. E., Volf, I., Carausu, C., Bühlmann, A. M., Gilca, I. A., & Popa, V. I. (2015). Optimization of ultrasound-assisted extraction of polyphenols from spruce wood bark. *Ultrasonics Sonochemistry, 22*, 535–541. https://doi.org/10.1016/j.ultsonch.2014.07.013

Gómez-Plaza, E., & Bautista-Ortín, A. B. (2019). Emerging technologies for aging wines: Use of chips and micro-oxygenation. In A. Morata (Ed.), *Red wine technology* (pp. 149–162). Academic Press. Elsevier Inc. https://doi.org/10.1016/B978-0-12-814399-5.00010-4.

Guerrero, E. D., Marín, R. N., Mejías, R. C., & Barroso, C. G. (2006). Optimisation of stir bar sorptive extraction applied to the determination of volatile compounds in vinegars. *Journal of Chromatography A, 1104*(1–2), 47–53. https://doi.org/10.1016/j.chroma.2005.12.006

Guerrero, E. D., Marín, R. N., Mejías, R. C., & Barroso, C. G. (2007). Stir bar sorptive extraction of volatile compounds in vinegar: Validation study and comparison with solid phase microextraction. *Journal of Chromatography A, 1167*(1), 18–26. https://doi.org/10.1016/j.chroma.2007.08.039

Herrera, P., Durán-Guerrero, E., Sánchez-Guillén, M. M., García-Moreno, M. V., Guillén, D. A., Barroso, C. G., & Castro, R. (2020). Effect of the type of wood used for ageing on the volatile composition of Pedro Ximénez sweet wine. *Journal of the Science of Food and Agriculture, 100*(6), 2512–2521. https://doi.org/10.1002/jsfa.10276

Iland, P., Bruer, N., Wilkes, E., & Edward, G. (2004). Anthocyanins (colour) and total phenolics of grape berries. In S. A. Campbelltown (Ed.), *Chemical Analysis of Grapes and wine: Techniques and concepts (first edit). Patrick iland wine promotions*.

(ISO), International Organization for Standardization. (1977). *Sensory analysis. Apparatus*. Wine-tasting glass (ISO 3591:1977).

(ISO), International Organization for Standardization. (2003). *Sensory analysis. Guidelines for the use of quantitative response scales*. ISO 4121:2003).

(ISO), International Organization for Standardization. (2006). *Sensory analysis. Methodology. Ranking* (ISO 8587:2006).

(ISO), International Organization for Standardization. (2007). *Sensory analysis. General guidance for the design of test rooms*. ISO 8589:2007).

Jiménez-Sánchez, M., Durán-Guerrero, E., Rodríguez-Dodero, M. C., Barroso, C. G., & Castro, R. (2020). Use of ultrasound at a pilot scale to accelerate the ageing of sherry vinegar. *Ultrasonics Sonochemistry, 69*(June), Article 105244. https://doi.org/10.1016/j.ultsonch.2020.105244

Jourdes, M., Michel, J., Saucier, C., Quideau, S., & Teissedre, P. L. (2011). Identification, amounts, and kinetics of extraction of C-glucosidic ellagitannins during wine aging in oak barrels or in stainless steel tanks with oak chips. *Analytical and Bioanalytical Chemistry, 401*(5), 1535–1543. https://doi.org/10.1007/s00216-011-4949-8

Mercurio, M. D., & Smith, P. A. (2008). Tannin quantification in red grapes and wine: Comparison of polysaccharide- and protein-based tannin precipitation techniques and their ability to model wine astringency. *Journal of Agricultural and Food Chemistry, 56*(14), 5528–5537. https://doi.org/10.1021/jf8008266

Meullemiestre, A., Petitcolas, E., Maache-Rezzoug, Z., Chemat, F., & Rezzoug, S. A. (2016). Impact of ultrasound on solid-liquid extraction of phenolic compounds from maritime pine sawdust waste. Kinetics, optimization and large scale experiments. *Ultrasonics Sonochemistry, 28*, 230–239. https://doi.org/10.1016/j.ultsonch.2015.07.022

Rubio-Bretón, P., Garde-Cerdán, T., & Martínez, J. (2018). Use of oak fragments during the aging of red wines. Effect on the phenolic, aromatic, and sensory composition of wines as a function of the contact time with the wood. *Beverages, 4*(4), 102. https://doi.org/10.3390/beverages4040102

Ruiz de Adana, M., López, L. M., & Sala, J. M. (2005). A Fickian model for calculating wine losses from oak casks depending on conditions in ageing facilities. *Applied Thermal Engineering, 25*(5), 709–718. https://doi.org/10.1016/j.applthermaleng.2004.07.021

Sánchez Guillén, M. M. (2016). *Estudio del comportamiento de "holandas" de diversos orígenes vitivinícolas durante su envejecimiento por diferentes metodologías tradicionales y aceleradas*. http://hdl.handle.net/10498/18455.

Sánchez-Córdoba, C., Durán-Guerrero, E., & Castro, R. (2021). Olfactometric and sensory evaluation of red wines subjected to ultrasound or microwaves during their maceration or ageing stages. *Lebensmittel-Wissenschaft & Technologie, 144*(March). https://doi.org/10.1016/j.lwt.2021.111228

Sarneckis, C. J., Dambergs, R. G., Jones, P., Mercurio, M., Herderich, M. J., & Smith, P. A. (2006). Quantification of condensed tannins by precipitation with methyl cellulose: Development and validation of an optimised tool for grape and wine analysis. *Australian Journal of Grape and Wine Research, 12*(1), 39–49. https://doi.org/10.1111/j.1755-0238.2006.tb00042.x

Schwarz, M., Rodríguez-Dodero, M. C., Sánchez, M., Guillén, D. A., & Barroso, C. G. (2014). Development of an accelerated aging method for Brandy. *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology, 59*(1), 108–114. https://doi.org/10.1016/J.LWT.2014.05.060

Schwarz, M., Rodríguez, M. C., Guillén, D. A., & Barroso, C. G. (2009). Development and validation of UPLC for the determination of phenolic compounds and furanic derivatives in Brandy de Jerez. *Journal of Separation Science, 32*(11), 1782–1790. https://doi.org/10.1002/jssc.200800706

Singleton, V. L., & Draper, D. E. (1963). Ultrasonic treatment with gas purging as a quick aging treatment for wine. *American Journal of Enology and Viticulture, 14*(1), 23–35. http://www.ajevonline.org/content/14/1/23.abstract.

Solar, S., Castro, R., & Guerrero, E. D. (2021). New accelerating techniques applied to the ageing of oenological products. *Food Reviews International*, 1–21. https://doi.org/10.1080/87559129.2021.1934009, 00(00).

Suárez, R., Suárez-Lepe, J. A., Morata, A., & Calderón, F. (2007). The production of ethylphenols in wine by yeasts of the genera Brettanomyces and Dekkera: A review. *Food Chemistry, 102*(1), 10–21. https://doi.org/10.1016/j.foodchem.2006.03.030

Sun, J., Luo, H., Li, X., Li, X., Lu, Y., & Bai, W. (2019). Effects of low power ultrasonic treatment on the transformation of cyanidin-3-O-glucoside to methylpyranocyanidin-3-O-glucoside and its stability evaluation. *Food Chemistry, 276*, 240–246. https://doi.org/10.1016/J.FOODCHEM.2018.10.038

Tao, Y., García Martín, J., & Sun, D.-W. (2014). Advances in wine aging technologies for enhancing wine quality and accelerating wine aging process. *Critical Reviews in Food Science and Nutrition, 54*, 817–835. https://doi.org/10.1080/10408398.2011.609949

Tao, Y., Zhang, Z., & Sun, D.-W. (2014). Experimental and modeling studies of ultrasound-assisted release of phenolics from oak chips into model wine. *Ultrasonics Sonochemistry, 21*(5), 1839–1848. https://doi.org/10.1016/J.ULTSONCH.2014.03.016

Yıldırım, H. K., & Dündar, E. (2017). New techniques for wine aging. *BIO Web of Conferences, 9*, Article 02012. https://doi.org/10.1051/bioconf/20170902012

Yu, Z., Su, Y., Zhang, Y., Zhu, P., Mei, Z., Zhou, X., & Yu, H. (2021). Potential use of ultrasound to promote fermentation, maturation, and properties of fermented foods: A review. *Food Chemistry, 357*(March), Article 129805. https://doi.org/10.1016/j.foodchem.2021.129805

Zhang, Q. A., Xu, B. W., Chen, B. Y., Zhao, W. Q., & Xue, C. H. (2020). Ultrasound as an effective technique to reduce higher alcohols of wines and its influencing mechanism investigation by employing a model wine. *Ultrasonics Sonochemistry, 61*, Article 104813. https://doi.org/10.1016/j.ultsonch.2019.104813