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Kinetics of oxygen consumption, a key factor in the changes of young wines composition

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ABSTRACT

The oxygen that a wine receives during the winemaking process defines its properties. The aim of this work was to evaluate the oxygen consumption capacity of wines and its influence on the modification of their composition. This preliminary work evaluated the changes after 3 months in the chemical composition of twenty-seven Spanish commercial red, white and rosé wines after their air saturation and oxidation process at 35 °C for 7 days. All the wines studied were high oxygen consumers, while the white and rosé wines showed greater variability according to their chemical composition. Wines that consumed a lot of oxygen did so quickly or slowly, while wines that consumed little oxygen did so slowly. All the wines showed a significant decrease in ethyl esters of straight-chain fatty acids (50–58%), ethyl esters of branched-chain fatty acids (48–56%) and alcohol acetates (34–65%) content, and a significant increase in Strecker aldehydes (24%) because of oxygen consumption. This paper presents a preliminary approach to determine the oxidation tendency of different wines showing the importance of controlling the winemaking processes that can increase oxygen availability and of establishing the minimum appropriate level of free sulfur dioxide.

1. Introduction

The oxygen in the air is always present during winemaking and can have a positive or negative influence. This depends mainly on the amount and concentration of dissolved oxygen, the moment of oxygen supply, the time of dissolution, and the characteristics of the wine (i.e. the presence of compounds in the wine that can react with the oxygen and consume it and the free sulfur dioxide content).

It is well-known that controlled oxygen additions can be useful during wine elaboration or aging since they can favor yeast growth, color stabilization, changes in the phenolic and volatile compounds, as well as a reduction in astringency and bitterness (Cano-López et al., 2008; Ortega-Heras, Rivero-Pérez, Pérez-Magariño, González-Huerta, & González-Sanjosé, 2008; Pérez-Magariño, Sánchez-Iglesias, Ortega-Heras, González-Huerta, & González-Sanjosé, 2007). Oxygen is involved in the oxidation, condensation and polymerization reactions with different compounds (mainly phenolic). On the one hand, these reactions can lead to the formation of new polymeric pigments that can stabilize red wine color (Atanasova, Fulcrand, Cheynier, & Moutounet, 2002; Bakker & Timberlake, 1997; Mateus, Silva, Rivas-Gonzalo, Santos-Buelga, & Freitas, 2003; Revilla, Pérez-Magariño, González-Sanjosé, & Beltrán, 1999). On the other hand they can reduce astringency and bitterness due to tannin reactions (Cejudo-Bastante, Hermosín-Gutiérrez, & Pérez-Coello, 2011). The addition of small and controlled amounts of oxygen to wines by microoxygenation can also modulate the aroma and decrease vegetal and green notes (Cejudo-Bastante et al., 2011; Ortega-Heras et al., 2008).

However, an excess of oxygen could produce negative effects, mainly on color and volatile composition (Escudero, Asensio, Cacho, & Ferreira, 2002; Laurie, Salazar, Campos, Cáceres-Mella, & Peña-Neira, 2014; Ugliano, 2013). For example, the oxygen supplied during bottling can play a negative role in wines and affect their final quality, depending on several factors such as the amount of oxygen, temperature, and wine composition (Singleton, 1987). Usually, white, and rosé wines are more

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sensitive to oxidation than reds and an excess of oxygen can produce the formation of brown-yellow pigments due to the oxidation of polyphenols, the formation of volatile compounds linked to aging developing oxidative notes and a decrease in varietal aromas (Carrascón, Fernandez-Zurbano, Bueno, & Ferreira, 2015; Culleré, Cacho, & Ferreira, 2007; Kanavouras, Coutelieris, Karanika, Kotseridis, & Kallithraka, 2020; Mislata, Puxeu, Tomás, Nart, & Ferrer-Gallego, 2020).

Therefore, it is very important to control the oxygen supply in young wines to maintain a high fruit intensity and color and to avoid oxidation flavors. Thus, studying the capacity of the final wines to consume oxygen is an aspect of great interest since it will allow their shelf life to be analyzed. This oxygen consumption capacity has been studied by various authors in different wines, and the results obtained are controversial. On the one hand some authors demonstrated that different types of wines can consume similar amounts of oxygen (Boulton, 2011; Nevares et al., 2017). On the other hand some studies have established that red wines can consume more oxygen than whites (Moutounet & Mazauric, 2001; Singleton, 1987). No research has been found about the speed of oxygen consumption of different wines and its effect on their composition. This knowledge is fundamental for the decisions and actions that winemakers will take with their wines in order to maintain a high fruit intensity and color and to avoid oxidation flavors. Consequently, they will be able to act in controlling the exposure of the wines to oxygen, with precautions in bottling, type of closures, additions of antioxidant compounds, etc. The aim of this preliminary work was to evaluate the kinetics of oxygen consumption and its effect on wine composition, highlighting the relevance of initial wine composition and the level of SO_2.

2. Materials and methods

2.1. Wine and sampling

A total of 27 "young" commercial Spanish wines (not aged in wood) from different Appellations of Origin, varieties, and vintages (between 2016 and 2018) were used: 9 whites, 9 rosés and 9 reds were acquired on the same day in local shops. Table 1 shows the detailed list of samples available in the supermarket, including information related to wine composition and sulfur dioxide content. The wines were made from grapes of different varieties harvested between 2016 and 2018, presenting very different initial characteristics due to both the grape variety, the winery's winemaking process and the time elapsed since they were made. All of them were Bordeaux bottle type with synthetic stoppers in accordance with the specifications of each winery. This is a preliminary study that allows us to observe the behavior of different wines in the kinetics of oxygen consumption with a representative number of samples higher or like that used in this type of experiments Mislata et al. (2020), Bueno et al. (2018), Carrascón et al. (2015, 2017), Escudero (2002). All the wines are "young wines", so according to the Spanish legislation in each area of production, they are wines that do not meet the conditions to be labelled as "crianza", "reserva" or "gran reserva". Furthermore, there is no indication on the bottles that they have been aged in barrels. The objective was to see the effect of oxygen saturation in different wines, which are differentiated into whites, rosés and reds because that is how they are marketed. All the wines were bought at the same time in the supermarket and the analysis started in October 2019 (when they were saturated), finishing the analysis in 2020.

In order to evaluate the effects of oxidation, the wines were evaluated in two different situations: first the newly-opened bottles (Wc) and then, after subjecting those bottles to saturation with air, storing them for 3 months and analyzing them (Ws).

The bottles were opened inside a Jacomex glove chamber (Dagneux, France) in which atmospheric oxygen was maintained at under 0.002% (<3 μg/L) to collect samples for analysis, thus avoiding further oxidation of the initial wines. For each wine, two samples were taken from the same bottle: 100 mL of wine was put into a screw capped bottle, filling to avoid head space, and analyzed (wine control, Wc); another 120 mL was saturated with air (wine saturated, Ws).

2.2. Air saturation of wines

Wine air saturation was performed according to the method established previously by del Alamo-Sanza, Sánchez-Gómez, Martínez-Martínez, Martínez-Gil, and Nevares (2021). Briefly, wine temperature was constantly measured until reaching the set working temperature (35 °C). Then the wines were air saturated for 5 min: 35 °C tempered air was injected, avoiding high-speed air flow (i.e. air flow rates >1 mL/min) and with very small bubbles (Näykki, Jalukse, Helm, & Leito, 2013). Six 500 mL bottles provided with independent porous ceramics were used to saturate 120 mL of wine; this allowed 6 wines to be saturated at the same time until the partial pressure of oxygen in the wine was balanced with that of the atmosphere (100% sat. air). 20 mL of saturated wines was used to measure the dissolved oxygen (DO) and the kinetics of oxygen consumption. The remaining 100 mL of the saturated wine was placed in a thermostatic chamber for 7 days at 35 °C to speed up oxygen consumption. This was subsequently stored for 3 months in the bottle room of the experimental cellar at the University of Valladolid (Palencia, Spain) at a controlled temperature and humidity (15–16 °C and 65–75%) until chemical analyses were carried out (Ws).

2.3. Measurement of DO and kinetics of oxygen consumption

For dissolved oxygen measurements, air-saturated wines were transferred to 2 mL glass SensorVial SV-PSt5 (PreSens Precision Sensing GmbH, Regensburg, Germany), which were airtight. The vials have an optically isolated oxygen sensor integrated at the bottom [Accuracy: 0.02% O_2 at 0% O_2 and 0.02% O_2 at 21% O_2 (37 °C). Resolution: 0.43% O_2 at 0% O_2 and 0.65% O_2 at 21% O_2 (37 °C)]. The SensorVials were placed in a 24-well plate, which was placed on the SDR SensorDish® Reader, ensuring that all the samples were measured under the same conditions and the oxygen consumption kinetics were monitored.

The oxygen consumption kinetics measurement for each wine was carried out in quadruplicate to reduce uncertainty and to ensure that all the samples were measured simultaneously under the same conditions. The SensorVials with the samples placed in the 24-well plate were kept in a high accuracy thermostatic chamber at a constant temperature of 35 ± 0.10 °C (Raypa Trade, Terrassa, Barcelona, Spain) in darkness. The assays were performed in groups of 48 vials. The DO of the samples was measured every hour throughout the consumption process (for 7 days). A total of 108 wine oxygen consumption kinetics (27 wines x 4 replicates) were studied.

Vials for oxygen measurement were calibrated according to the manufacturer's protocol. Calibration was performed at two points: oxygen-free water at a concentration of 0 mg/L (0% air saturation) and saturated air (100% air saturation). The 0% calibration standard was prepared based on a strong reductant (1 g Na_2SO_3 and 50 μL Co $(NO_3)_2$ were dissolved in 100 mL of pure water MilliQ). All calibrations were done at a water temperature of 35 °C.

2.4. Kinetic curve data process

The oxygen consumption kinetic curve data were pre-processed according to del Alamo-Sanza et al. (2021). To obtain the representative curves for each wine, the initial (before the maximum) and final (after the minimum) data were removed. Two kinetics from each wine were resampled in a sampling period of 15 min (mean–std and mean + std). Therefore, a total of 54 curves were obtained from the 104 kinetic curves measured from the 27 samples analyzed (9 for each type of wine).

Table 1
Characteristics of the 27 wine studied samples.

Type	Wine code	Wine	DO/Region	Vintage	Grape variety	Total acidity (g/L)	pH	Acetic acid (g/L)	Free SO_2 (mg/L)	Total SO_2 (mg/L)	Reducing sugars (g/L)
White	1-W	Vega de Barón	Rueda	2018	Verdejo	5.57 ± 0.31	3.23 ± 0.10	0.49 ± 0.08	19 ± 3	96 ± 15	1.5 ± 0.6
	2-W	La bien pintá	Rueda	2018	Verdejo	5.32 ± 0.30	3.28 ± 0.10	0.23 ± 0.04	9 ± 1	84 ± 13	1.4 ± 0.6
	3-W	Vegadelpas	Rueda	2017	Verdejo	5.05 ± 0.28	3.40 ± 0.10	0.24 ± 0.04	23 ± 4	121 ± 19	1.6 ± 0.7
	4-W	Oristan	La Mancha	2018	Verdejo	5.17 ± 0.29	3.34 ± 0.10	0.18 ± 0.03	24 ± 4	72 ± 11	4.8 ± 2.0
	5-W	Nivei	Rioja	2017	n.d.	4.98 ± 0.28	3.31 ± 0.10	0.23 ± 0.04	16 ± 2	80 ± 12	1.5 ± 0.6
	6-W	Mezquíriz	Navarra	2017	Chardonnay	4.51 ± 0.25	3.42 ± 0.10	0.25 ± 0.04	16 ± 2	97 ± 15	2.8 ± 1.1
	7-W	Coto de Ibedo	Ribeiro	2017	Treixadura. Godello and Loureira	4.80 ± 0.27	3.54 ± 0.11	0.47 ± 0.08	33 ± 5	131 ± 20	4.1 ± 1.7
	8-W	Viña pati	Rueda	2017	Verdejo and Viura	5.37 ± 0.30	3.30 ± 0.10	0.22 ± 0.04	50 ± 8	101 ± 16	1.6 ± 0.7
	9-W	Venta morales	n.d.	2017	n.d.	4.51 ± 0.25	3.26 ± 0.10	0.12 ± 0.02	16 ± 2	94 ± 15	3.6 ± 1.5
Rosé	10-W	Cepa Lebrel	Rioja	2017	Tempranillo	4.71 ± 0.26	3.51 ± 0.10	0.38 ± 0.05	22 ± 2	102 ± 12	1.1 ± 0.5
	11-W	Picudo	n.d.	n.d.	n.d.	5.11 ± 0.27	3.22 ± 0.11	0.28 ± 0.08	5 ± 5	97 ± 20	4.1 ± 1.7
	12-W	Valdesalud	Cigales	2018	Tempranillo. Albillo and Verdejo	4.76 ± 0.30	3.53 ± 0.10	0.24 ± 0.04	28 ± 8	74 ± 16	1.6 ± 0.7
	13-W	Peregrino	Tierra de León	2018	Prieto Picudo	5.20 ± 0.25	3.44 ± 0.10	0.35 ± 0.02	13 ± 2	62 ± 15	3.6 ± 1.5
	14-W	Val de Condes	Cigales	2018	Tempranillo. Albillo. Verdejo and Garnacha	4.26 ± 0.24	3.54 ± 0.11	0.38 ± 0.07	10 ± 2	41 ± 6	4.1 ± 1.7
	15-W	Flavium	Bierzo	2018	Mencia	5.18 ± 0.29	3.41 ± 0.10	0.30 ± 0.05	6 ± 1	51 ± 8	1.3 ± 0.5
	16-W	Catedral de Léon	León	2018	n.d.	4.94 ± 0.28	3.28 ± 0.10	0.38 ± 0.07	7 ± 1	95 ± 15	1.4 ± 0.6
	17-W	Viña Serea	n.d.	n.d.	n.d.	4.97 ± 0.28	3.44 ± 0.11	0.38 ± 0.07	30 ± 5	141 ± 22	2.4 ± 1.0
	18-W	Conde Ansurez	Cigales	2018	Verdejo. Garnacha. Tinto Fino and Albillo Mayor	5.43 ± 0.30	3.25 ± 0.10	0.23 ± 0.04	24 ± 4	94 ± 12	1.3 ± 0.5
Red	19-W	Fidencio	La Mancha	2018	Tempranillo	4.17 ± 0.23	3.65 ± 0.11	0.19 ± 0.03	20 ± 3	53 ± 8	1.5 ± 0.6
	20-W	Arteso	Rioja	2018	n.d.	4.58 ± 0.26	3.67 ± 0.11	0.38 ± 0.07	21 ± 3	59 ± 9	1.8 ± 0.7
	21-W	Comportillo	Rioja	2018	n.d.	4.67 ± 0.26	3.59 ± 0.11	0.29 ± 0.05	24 ± 4	53 ± 8	1.5 ± 0.6
	22-W	Castillo de Soldepeñas	Valdepeñas	n.d.	Tempranillo and Garnacha	4.71 ± 0.26	3.56 ± 0.11	0.31 ± 0.05	17 ± 3	64 ± 10	2.0 ± 0.8
	23-W	Señorio de Nava	Toro	2016	Tinta de Toro	4.51 ± 0.25	3.63 ± 0.11	0.40 ± 0.07	3 ± 0	48 ± 7	1.6 ± 0.7
	24-W	Condado de Teón	Ribera del Duero	2016	n.d.	4.88 ± 0.27	3.67 ± 0.11	0.55 ± 0.09	14 ± 2	64 ± 10	1.4 ± 0.6
	25-W	Sierra Salinas	Utiel-Requena	2017	Bobal and Tempranillo	4.94 ± 0.28	3.52 ± 0.11	0.51 ± 0.09	20 ± 3	135 ± 21	4.2 ± 1.7
	26-W	Fin del Rio	Castilla y León	2018	Tempranillo	4.82 ± 0.27	3.79 ± 0.12	0.66 ± 0.11	10 ± 2	62 ± 10	10.4 ± 4.3
	27-W	Conquero	Toro	2018	Tempranillo	4.91 ± 0.27	3.63 ± 0.11	0.40 ± 0.07	22 ± 3	48 ± 7	5.9 ± 2.4

n.d. no data. **W**:Wine.

2.5. Wine analyses

A total of 27 wines were analyzed in duplicate (54 wine samples were studied).

2.5.1. Analysis of oenological and color parameters and total phenolic composition

Standard oenological parameters in wines were determined according to official analysis methods (OIV, 2016): pH, titratable acidity (as g/L tartaric acid), volatile acidity (as g/L acetic acid) free and total sulfur dioxide (as mg/L sulfur dioxide). The methods used to evaluate these parameters are accredited by ISO 17025 Norm and uncertainty was also calculated according to this Norm.

Color parameters, color intensity and tonality were evaluated using the Glories methodology (Glories, 1984). Phenolic composition was evaluated by the quantification of total phenols by reaction to Folin–Ciocalteu in mg/L of gallic acid and total anthocyanins, and by pH changes in mg/L of malvidin-3-glucoside (Paronetto, 1977). Polymeric anthocyanins were determined according to the method described by Levengood and Boulton (2004) and expressed as a percentage.

2.5.2. Analysis of low molecular weight phenolic compounds

Hydroxybenzoic acids (HBA), hydroxycinnamic acids (HCA), ellagic acid, phenolic alcohols, flavanols, flavonols and stilbenes were analyzed by direct injection of wine samples (diluted 1:2) in an Agilent liquid chromatograph series 1100 (Agilent Technologies Inc., United States) equipped with a photodiode array detector (DAD). The chromatographic conditions and the quantification of phenolic compounds were established by Pérez-Magariño, Ortega-Heras, and Cano-Mozo (2008) and the validation information is summarized in Supplementary Table S2.

2.5.3. Analysis of volatile compounds

Major volatile compounds were quantified by direct injection of 1 μL of wine, using an Agilent 7890A gas chromatograph with a flame ionization detector (FID). Samples were injected in split mode (25:1), with the chromatographic conditions indicated in Pérez-;Magariño et al. (2019). Higher alcohols (methanol, 1-propanol, isobutanol, 1-butanol, 2-phenylethanol and isoamylalcohols), acetaldehyde and ethyl acetate were quantified (Supplementary Table S3).

Minor volatile compounds were analyzed by headspace solid-phase micro-extraction (HS-SPME) and gas chromatography with mass detector (GC-MS). A volume of 10 mL of diluted wine (1:3 with a hydroalcoholic solution and the addition of four internal standards (IS): methyl 2-methylbutyrate, methyl octanoate, heptanoic acid and 3,4-dimethylphenol) was placed in a 20-mL glass vial with 3.5 g/L of sodium chloride. The samples were incubated at 40 °C for 5 min and the volatiles in the headspace of the vial were then extracted with a 1-cm 50/30-μm DVB/Carboxen/PDMS SPME fiber (Supelco) at the same temperature and an agitation speed of 500 rpm for 60 min. After extraction, the fiber was desorbed for 3 min in the injector at 250 °C using the splitless mode.

Chromatographic analyses were performed with an Agilent 78902B CG coupled to a 5977B MSD and an autosampler PAL RSI 120 equipped with a DB-WAX Ultra Inert capillary column (60 m in length, 0.25 mm i.d., and 0.50 mm film thickness, Agilent), following the chromatographic conditions established by Rodríguez-Bencomo, Ortega-Heras, and Pérez-Magariño (2010). The volatile compounds were identified using the retention times and mass spectra of the standard compounds and the NIST library. Quantification followed the internal standard quantification method, using selected quantification ions and IS chosen for each compound (Supplementary Table S4).

Thirty-nine minor volatile compounds were quantified in the studied wines and grouped as ethyl esters of straight-chain fatty acids (ethyl butyrate, ethyl hexanoate, ethyl octanoate and ethyl decanoate), ethyl esters of branched-chain fatty acids (ethyl 2-methylbutyrate and ethyl isovalerate), alcohol acetates (propyl acetate, butyl acetate, isobutyl acetate, phenylethyl acetate, hexyl acetate and isoamyl acetate), terpenes (linalool, α-terpineol, geraniol and β-citronellol), fatty acids (isobutyric, butyric, isovaleric, hexanoic, octanoic and decanoic acid), C6 alcohols (1-hexanol, *cis*-3-hexenol and *trans*-3-hexenol), vanillin derivates (vanillin, methyl vanillate, ethyl vanillate and acetovanillone), furanic aldehydes (furfural, 5-methylfurfural and 5-hydroxymethylfurfural), volatile phenols (guaiacol, 4-methylguaiacol, 4-vinylguaiacol, 4-ethylguaiacol, 4-vinylphenol, 4-ethylphenol and eugenol) and Strecker aldehydes (2-methylbutanal, 3-methylbutanal, isobutyraldehyde, phenylacetaldehyde and methional).

2.6. Statistical analyses

A one-way analysis of variance (ANOVA) and the least significant difference (LSD) test were carried out to identify significant differences by wine type (white, rosé or red), oxygen consumption capacity (high or low) and oxygen consumption speed (fast, medium or slow) of all the variables. A level of $p < 0.05$ was considered as statistically significant for all tests. Factor analyses were carried out with the significant chemical variables to determine the variables that most contribute to the air saturation effect by wine type. In addition, principal component analysis was performed with the initial chemical variables. The wines were differentiated by oxygen consumption to evaluate the relationship between the oxygen consumed and the variation in the content of the different compounds analyzed by wine type. All the statistical analyses were carried out using the Statgraphics Centurion statistical program (version 18.1.12; StatPoint, Inc., VA, USA).

3. Results and discussion

3.1. Wine oxygen consumption

The average kinetics of the oxygen consumption of each wine showed that in almost one week (160 h) the DO of all red wines was at the bottom of the graph, indicating that they were large consumers of oxygen (Fig. 1). In addition, 4 of the white wines studied and 3 of the rosés also consumed a large part of the available oxygen. This result indicates that different types of wines can consume similar amounts of oxygen, as previously indicated (Boulton, 2011; Nevares et al., 2017). However, other studies have established that red wines can consume more oxygen than whites (Moutounet & Mazauric, 2001; Singleton, 1987). It has been shown that the dose of free SO_2 is related to the oxygen consumption capacity of the wines, this being particularly relevant in the case of white and rosé wines, which consumed more oxygen at higher levels of free SO_2. This result agrees with Fracassetti, Coetzee, Vanzo, Ballabio, and Toit (2013) who studied Sauvignon Blanc wines with different levels of sulfur dioxide which were saturated with air. Their results showed that oxygen consumption was strongly influenced by the presence of sulfur dioxide. They observed a large variation in the oxygen consumption of the wines, with the rate of oxygen consumption rising with an increasing SO_2 concentration. This indicates that the protection of this type of wine relies on adequate free SO_2 levels, which depend on wine properties such as pH and alcohol content. However, this relationship was not found in reds; all the wines studied showed a high capacity to consume oxygen and different levels of free SO_2. This indicates the great influence of phenolic compounds in the protection of red wines against oxidation. Many of the white and rosé wines have also been found to present an excessive level of molecular SO_2 since, according to their pH, the levels of molecular SO_2 are above those necessary to ensure protection against microorganisms and unwanted bacteria. This excessive SO_2 dosage could be avoided in cases such as that of white wine 4-W, which had a molecular SO_2 of 1 mg/L with a pH and free SO_2 level of 24 mg/L (Table 1) (Henderson-Hasselbalch equation). This wine had sufficient SO2 to consume the dosed oxygen but did not consume all that available. It was one of the white wines studied with the lowest level of oxygen consumed ($DO_{max\text{-}min}$) (Table 2). The

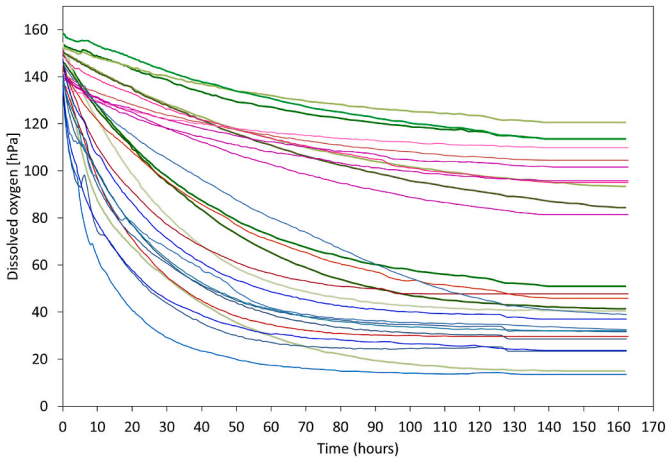

Fig. 1. Oxygen kinetics mean of the 27 studied wines. White wines: green color; rosé wines: red-pink color; red wines: blue color. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

same was found in the case of rosé wine 18-W. Therefore, the study of the oxygen consumption capacity of the wines provides information on the type of protection they need and lower levels of SO_2 could be used.

As regards consumption kinetics, the parameters that characterize it in each wine were obtained according to the method established by del Alamo-Sanza et al. (2021). Table 2 shows the average result of the 12 parameters of the consumption kinetics obtained for the white, rosé and red wines according to their capacity to consume DO (high or low) and maximum speed of consumption (fast, medium or slow). The parameters are related to the DO present at the different moments of the consumption kinetics (O_{max}, O_{int}, O_{10} and ΔO_{max_min}), the time spent in consuming those amounts of oxygen ($t_{O\text{-}90}$, $t_{O\text{-}int}$, $t_{O\text{-}10}$ and $t_{O\text{-}min}$), the maximum speed of DO consumption (R_{max}) and the area under the curve (A_{max_min} and $A_{90\text{-}10}$).

The maximum DO that a wine can admit (O_{max}) after saturation with air was higher in the white wines than in the reds or rosés. Eight of the white wines had a partial pressure of oxygen above 147 hPa while only one rosé and one red presented similar levels (11-W and 22-W). Depending on the type of wine and despite being saturated according to the same protocol, not all of them were able to reach the same oxygen partial pressure, either since the wine did not follow Henry's law or due to oxygen solubility depending on their ethanol, sugar and phenolic compound content. Overall, the red wines presented the lowest DO values at the end of the consumption kinetics (O_{10} and O_{min}), and therefore a higher consumption capacity (ΔO_{max_min}). No significant differences were found in the whites and rosés in O_{10}, O_{int}, O_{min} and ΔO_{max_min}. The deviation found in these parameters was very high, more than twice that found in the reds, indicating the great variability within white and rosé wines. Table 2 also shows the results differentiating low or high oxygen consuming wines, establishing those with a $\Delta O_{max\_min} >$ 70 hPa and an $O_{min} <$80 hPa as high. High oxygen consuming wines can absorb twice as much oxygen ($\Delta O_{max_min} >$102 hPa) as low consumers ($\Delta O_{max_min} <$ 48 hPa). They exhibited a residual unconsumed oxygen O_{min} around 100 hPa in comparison with high consuming wines that left an oxygen level below 41 hPa unconsumed. Red wines showed a significantly higher consumption rate (R_{max}) than whites and rosés, between which there were no significant differences. Moreover, no relationship was found between the amount of oxygen the reds were capable of consuming and their speed of consumption.

A fast-consuming wine reached a consumption rate >10 hPa/h, a medium between 5 and 10 hPa/h and a slow <5 hPa/h. Therefore, 7 of the 9 red wines studied were fast and the other 2 (23-W and 25-W) were medium. Regarding the 4 white wines previously considered high oxygen consumers, 2 were fast (6-W and 7-W) and 2 were slow (3-W and 8-W). The rest of the white wines studied consumed oxygen slowly (5 hPa/h). The rosé wines presented medium and slow oxygen consumption rates, regardless of whether their capacity was low or high (Fig. 1). In this study all the red wines presented higher oxygen avidity, reflected in the higher value of ΔO_{max_min} and a R_{max} more than twice that of the other wines (Table 2).

Table 2 also represents the average of the consumption kinetic parameters differentiating the wines by their capacity to consume oxygen (ΔO_{max_min}) and by the maximum consumption rate (R_{max}). Therefore, the wines with a lower maximum oxygen consumption rate maintained higher oxygen contents both in the middle of the kinetics and at the end resulting in lower ΔO_{max_min}.

Table 2
Mean value of the parameters from the oxygen consumption wine kinetic curves.

	Type of wines			Oxygen consumption capacity					Oxygen consumption rate					
				High			Low		Fast		Medium		Slow	
	White	Rosé	Red	White	Rosé	Red	White	Rosé	White	Red	Rosé	Red	White	Rosé
O_{max}	151.9 b	142.4 a	140.0 a	149.8 b α	143.0 ab α	140.0 a	153.6 b α	142.0 a α	151.3 b	139.0 a	144.2	143.3	152.1 b	140.1 a
O_{int}	113.4 b	110.7 b	84.5 a	93.4 b α	92.1 ab α	84.5 a	129.4 b β	120.0 a β	89.5 α	83.1	104.1 α	89.4	120.2 β	119.0 β
O_{10}	82.6 b	85.5 b	40.2 a	48.3 α	51.4 α	40.2	110.0 β	102.5 β	40.2 α	38.5	72.1 α	46.3	94.7 β	102.2 β
O_{min}	74.8 b	79.1 b	29.0 a	37.0 α	41.1 α	29.0	105.1 β	98.1 β	27.8 α	27.2	64.0 α	35.5	88.3 β	97.9 β
ΔO_{max_min}	77.1 a	63.3 a	110.9 b	112.8 β	101.9 β	110.9	48.4 α	43.9 α	123.5 b β	11.8 a	80.1 β	107.9	63.8 α	42.2 α
t_{O_90}	5.4 c	3.3 b	1.8 a	3.6 b α	2.9 ab α	1.8 a	6.8 b β	3.4 a α	2.6 b α	1.1 a α	2.8	4.1 β	6.2 b β	3.8 a
t_{O_int}	33.6 b	28.8 b	16.6 a	22.7 α	20.7 α	16.6	42.2 b β	32.9 a β	16.3 α	12.8 α	26.1	30.0 β	38.5 β	32.3
t_{O_10}	96.6 b	87.6 b	58.0 a	74.0 α	66.2 α	58.0	114.7 b β	98.4 a β	62.3 α	50.9 α	78.9	83.0 β	106.4 β	98.6
t_{O_min}	147.9 b	133.0 a	143.2 b	148.2 b α	119.8 a α	143.2 b	147.7 b α	139.6 a β	146.6	141.2	128.2 a	150.1 b	148.3 b	139.1 a
R_{max}	6.2 a	5.4 a	15.1 b	9.9 ab α	6.9 a α	15.1 b	3.2 α	4.6 α	15.6 β	17.2 β	7.3 β	7.7 α	3.5 α	3.0 α
A_{90_10}	9975 b	9049 b	4076 a	5605 α	5177 α	4076	13472 b β	10985 a β	4239 α	3439 α	7606 α	6306 β	11614 β	10853 β
A_{max_min}	13977 b	13034 b	6945 a	9030 α	7805 α	6945	17936 b β	15648 a β	7255 α	6237 α	11073 α	9423 β	15898 β	15484 β

For each parameter, different letters indicate significant differences among different wines according to the Fisher's LSD test ($\alpha < 0.05$). For each type of wine, different Greek letters indicate differences between wines with high or low oxygen consumption capacity and between fast. medium or slow oxygen consumption. There is no letter if there were not significant differences.

O_{max}: *Maximum/Initial oxygen value (hPa);* ***O_{int}*** *: Average oxygen value between the maximum and the minimum oxygen values (hPa);* ***O_{10}*** *: Oxygen value that represents* 10% *of the range between the maximum and minimum values (hPa);* ***O_{min}*** *: Minimum/Final value (hPa);* ***ΔO_{max_min}***: *Total oxygen consumed (hPa);* ***t_{O_90}*** *: Time when oxygen* 90 *is reached (h);* ***t_{O_int}***: *Fall time to 50% air saturation (h);* ***t_{O_10}***: *Time when oxygen 10 is reached (h);* ***t_{O_min}*** *: Total consumption time (h);* ***R_{max}*** *: Maximum value of the oxygen consumption/rate curve (hPa /h);* ***A_{90_10}*** *: Area under the oxygen consumption curve and between t*90 *and t*10 *(hPa·h);* ***A_{max_min}***: *Area under the oxygen consumption curve (hPa·h).*

Table 3
Mean values of the different phenolic compounds (mg/L), color parameters and volatile composition (mg/L) of the Wc and the difference between Ws-Wc wines, depending on the type of wine, the capacity to consume oxygen and the maximum speed of that consumption.

		Type of wines			Oxygen consumption capacity					Oxygen consumption rate					
					High			Low		Fast		Medium		Slow	
		White	Rosé	Red	White	Rosé	Red	White	Rosé	White	Red	Rosé	Red	White	Rosé
Total polyphenols	Wc	183 a	299 b	1861 c	209 a β	332 a β	1861 b	162 a α	283 b α	197 a	1835 b	315 a	1951 b	179 a	280 b
	Ws-Wc	−14.4	−14.4	−20.3	−22.1 α	−21.4 α	−20.3	−8.16 β	−10.9 β	−17.1	−26.4	−16.7 a	1.18 b	−13.6	−11.6
Total anthocyanins	Wc		25.6 a	149 b		22.2 a	149 b		27.3		175 β	24.9 a	59.4 b α		26.4
	Ws-Wc		−7.86 b	−40.2 a		−9.20 b	−40.2 a		−7.19		−46.6 α	−8.31 b	−17.6 a β		−7.30
Polymeric anthocyanins (%)	Wc		53.5	54.3		60.0	54.3		50.2		50.0 α	54.8	69.5 β		51.9
	Ws-Wc		10.2	7.13		13.3	7.13		8.64		8.14 β	12.27	3.57 α		7.60
HBA[a]	Wc	9.0 a	15.1 a	60.5 b	10.0 a	15.5 a	60.5 b	8.2	15.0	9.2 a	60.7 b	13.7 a	59.9 b	8.9	17.0
	Ws-Wc	0.11 b	−0.68 a	1.69 c	0.24 b	−1.00 a	1.69 c	0.00 b	−0.52 a	0.30 a	1.74 b	−1.03 a α	1.52 b	0.05	−0.24 β
HCA[a]	Wc	21.5 a	20.2 a	49.5 b	20.5 a	24.6 a β	49.5 b	22.3	17.9 α	21.9 a	48.6 b	23.1 a β	52.5 b	21.4	16.6 α
	Ws-Wc	−0.15 a	0.01 a	0.47 b	−0.16 a	−0.17 a β	0.47 b	−0.13 a	0.10 b α	−0.26 a	0.61 b	−0.03	−0.02	−0.11 a	0.07 b
Ellagic acid	Wc			2.59			2.59				2.91 β		1.47 α		
	Ws-Wc			−0.14			−0.14				−0.18 α		−0.01 β		
Phenolic alcohols	Wc	24.2 a	34.0 b	85.3 c	20.6 a	32.1 a	85.3 b	27.0	35.0	19.8 a	88.2 b	33.6 a	75.4 b	25.4 a	34.5 b
	Ws-Wc	−0.69 c	−2.25 b	−7.70 a	−1.14 b α	−2.37 b	−7.70 a	−0.33 b β	−2.20 a	−0.83 a	−8.55 b α	−1.38 b	−4.76 a β	−0.65 b	−3.35 a
Flavanols	Wc	4.11 a	2.20 a	32.2 b	5.10 a	2.00 a	32.2 b	3.32	2.30	6.87 a β	34.4 b β	2.07 a	24.5 b α	3.32 α	2.36
	Ws-Wc	0.03 b	−0.17 b	−2.66 a	−0.07 b	−0.33 b α	−2.66 a	0.12 b	−0.09 a β	−0.16 a	−3.39 b α	−0.19	−0.11 β	0.09	−0.15
Flavonols	Wc	0.36 a	1.97 a	31.2 b		2.81 a	31.2 b	0.64	1.55		31.6	2.29 a	29.8 b	0.46 a	1.58 b
	Ws-Wc	−0.01 b	−0.14 b	−2.39 a		−0.37 b α	−2.39 a	−0.01	−0.03 β		−2.29	−0.26 b α	−2.71 a	−0.01	0.01 β
Stilbenes	Wc	-	0.28 a	2.36 b		0.26 a	2.36 b		0.29		1.94 α	0.28 a	3.82 b β		0.29
	Ws-Wc		0.00 b	−0.31 a		0.00 b	−0.31 a		0.01		−0.31	0.00 b	−0.31 a		0.01
Color intensity	Wc	0.11 a	0.95 a	8.46 b	0.13 a β	0.86 a	8.46 b	0.10 a α	0.99 b	0.12 a	8.39 b	0.95 a	8.71 b	0.11 a	0.94 b
	Ws-Wc	0.03 a	0.21 b	1.38 c	0.05 a α	0.24 a	1.38 b	0.02 a β	0.19 b	0.04 a	1.55 b β	0.21 a	0.78 b α	0.03 a	0.20 b
Tonality	Wc		1.08 b	0.81 a		1.16 b	0.81 a		1.03		0.78 α	1.08	0.90 β		1.07
	Ws-Wc		0.09 b	0.03 a		0.18 b β	0.03 a		0.04 α		0.03	0.14 b β	0.03 a		0.03 α
Yellow %	Wc		47.6 b	39.4 a		49.0 b	39.4 a		46.9		38.6 α	47.4	42.1 β		47.8
	Ws-Wc		1.72	0.62		3.44 b β	0.62 a		0.86 α		0.62	2.80 β	0.62		0.37 α
Blue%	Wc		7.57 a	11.5 b		7.88 a	11.5 b		7.42		11.73	7.89 a	10.9 b		7.18
	Ws-Wc		0.15 a	0.47 b		0.35	0.47		0.05		0.57	0.15	0.13		0.15
Red %	Wc		44.9 a	49.0 b		43.2 a	49.0 b		45.7		49.6 β	44.8	47.0 α		45.0
	Ws-Wc		−1.87	−1.09		−3.79 a α	−1.09 b		−0.91 β		−1.19	−2.95 α	−0.75		−0.52 β
Higher alcohol	Wc	308 a	299 a	536 b	311	289 a	536 b	305	304	290 a	544 b	293 a	509 b	313	307
	Ws-Wc	−4.60 ab	4.64 b	−12.9 a	2.68 β	−1.50	−12.9	−10.4 a α	7.71 b	1.30	−20.3 α	0.53	13.0 β	−6.28 a	9.79 b
Acetaldehyde	Wc	7.80 a	67.3 b	19.1 a	9.7 β	85.5 b	19.1 a	6.2 a α	58.3 b	10.6	15.0 α	87.0 a β	33.4 b β	7.0 a	42.8 b α
	Ws-Wc	4.96 b	−4.58 a	−6.25 a	6.71 b	−11.3 a	−6.25 a	3.57	−1.21	9.33 b β	−6.42 a	−15.2 α	−5.66	3.72 β α	8.72 β
Ethyl acetate	Wc	60	59	67	61	57	67	59	60	65	64	58 a	75 b	59	60
	Ws-Wc	−19.2 ab	−15.1 b	−21.7 a	−17.3 ab	−13.9 b	−21.7 a	−20.6	−15.7	−18.3	−21.5	−13.6 b	−22.4 a	−19.4	−16.9
EE-SCFA[b]	Wc	1.303 b	1.162 b	0.582 a	1.301 b	1.129 b	0.582 a	1.305	1.178	1.265 b	0.628 a β	1.073 b	0.421 a α	1.314	1.272
	Ws-Wc	−0.75 a	−0.61 b	−0.29 c	−0.73 a	−0.53 b	−0.29 c	−0.78	−0.64	−0.71 a	−0.33 b α	−0.52 a	−0.18 b β	−0.77	−0.71
EE-BCFA[b]	Wc	0.039 a	0.032 a	0.054 b	0.039 ab	0.031 a	0.054 b	0.040	0.033	0.033	0.045 α	0.032 a	0.088 b β	0.041	0.032
	Ws-Wc	−0.02 b	−0.02 b	−0.03 a	−0.02 b	−0.01 c	−0.03 a	−0.02	−0.02	−0.02	−0.02 β	−0.01 b	−0.05 a α	−0.02	−0.02
Alcohol acetates	Wc	1.029 b	1.017 b	0.546 a	0.895	1.030	0.546	1.136	1.010	0.748	0.610	1.126	0.321	1.109	0.880
	Ws-Wc	−0.66 a	−0.56 a	−0.19 b	−0.57 a	−0.60 a	−0.19 b	−0.74	−0.54	−0.47	−0.21 α	−0.64	−0.08 β	−0.72	−0.47
Fatty acids	Wc	10.2 c	8.55 b	6.32 a	10.7 c	8.67 b	6.32 a	9.88 b	8.49 a	11.4 b	6.39 a	8.31 b	6.08 a	9.89 b	8.84 a
	Ws-Wc	0.69 b	0.32 a	0.44 ab	0.46	0.48	0.44	0.87 b	0.25 a	0.43	0.42	0.43	0.51	0.76	0.19
C6 alcohols	Wc	2.01 a	2.74 b	3.02 b	2.25 a β	3.11 b	3.02 b	1.81 a α	2.56 b	2.42 β	3.08	2.74	2.84	1.89 a α	2.74 b
	Ws-Wc	0.31 b	−0.17 a	−0.16 a	0.50 b	−0.17 a	−0.16 a	0.16	−0.16	0.42 b	−0.14 a	−0.14	−0.23	0.28 b	−0.20 a
Terpenes	Wc	0.040 ab	0.034 a	0.049 b	0.036 a	0.034 a	0.049 b	0.043	0.034	0.047	0.048	0.032 a	0.053 b	0.038	0.036
	Ws-Wc	0.01 b	0.01 b	0.00 a	0.01 b	0.01 b	0.00 a	0.01	0.01	0.01	0.00	0.01 b	0.00 a	0.01	0.01

(*continued on next page*)

Table 3 (*continued*)

		Type of wines			Oxygen consumption capacity				Oxygen consumption rate					
					High			Low	Fast		Medium		Slow	
		White	Rosé	Red	White	Rosé	Red	White	Rosé	White	Red	Rosé	Red	White	Rosé
Vanillin derivates	Wc	0.122 a	0.105 a	0.289 b	0.124 a	0.096 a	0.289 b	0.120	0.110	0.103 a α	0.290 b	0.091 a	0.287 b	0.127 β	0.122
	Ws-Wc	0.003	0.002	0.004	0.00	0.00	0.00	0.00	0.00	0.00 a	0.01 b	0.00	0.00	0.00	0.00
Furanic aldehydes	Wc	0.252 b	0.120 a	0.088 a	0.317 c	0.182 b β	0.088 a	0.199 b	0.090 a α	0.318 b	0.086 a	0.141	0.093	0.233 b	0.094 a
	Ws-Wc	0.13 b	0.11 b	0.04 a	0.19 b β	0.19 b β	0.04 a	0.08 α	0.07 α	0.16 b	0.03 a	0.14 b β	0.04 a	0.13	0.06 α
Volatile phenols	Wc	0.167 c	0.019 a	0.078 b	0.194 b	0.034 a β	0.078 a	0.145 b	0.011 a α	0.119	0.074	0.023 a	0.092 b	0.180 b	0.014 a
	Ws-Wc	−0.01 a	0.00 ab	0.00 b	−0.01 a	−0.01 a α	0.00 b	−0.01	0.00 β	−0.01 a	0.00 b	−0.01	0.00	−0.01	0.00
Strecker aldehydes	Wc	0.169 a	0.189 b	0.175 ab	0.156	0.169	0.175	0.179	0.198	0.167	0.169 α	0.179	0.199 β	0.169 a	0.201 b
	Ws-Wc	0.03 a	0.05 b	0.04 ab	0.04	0.06	0.04	0.02 a	0.05 b	0.04	0.04	0.05	0.05	0.03	0.05

For each parameter, different letters indicate significant differences among different wines according to the Fisher's LSD test ($\alpha < 0.05$). For each type of wine, different Greek letters indicate differences between wines with high or low oxygen consumption capacity and between fast. medium or slow oxygen consumption. There is no letter if there were not significant differences.

[a] HBA: hydroxybenzoic acids, HCA: hydroxycinnamic acids.

[b] EE-SCFA: ethyl esters of straight-chain fatty acids, EE-BCFA: ethyl esters of branched-chain fatty acids.

3.2. Effect of oxygen on wine composition

Table 3 shows the mean values of the Wc and the differences between Ws and Wc of the different phenolic compounds, color parameters and volatile composition evaluated in order to study the effect of air saturation on the white, rosé and red wines, within the type of wine, the capacity to consume oxygen and the maximum speed of consumption.

3.2.1. Effect of oxygen on phenolic compounds

All red wines were high oxygen consumers; however, within the whites and rosés there were wines with high and low oxygen consumption capacity. Those with a high capacity initially presented a higher content of total polyphenols, confirming their implication in oxygen consumption. It is known that phenolic compounds react rapidly with oxygen, a reaction mediated mainly by the metals iron and copper (Danilewicz, 2007; Singleton, 1987). A slight decrease was observed in all the total polyphenols, but they did not show any statistically significant differences by wine type. The average decrease was 16.4 mg/L, this being higher than 26 mg/L in the case of the red wines (Table 3). However, the loss of these compounds in the whites and rosés varied according to their consumption capacity, with the highest losses in the most oxygen-consuming wines. The loss of total polyphenols showed a significant correlation with different studied parameters of oxygen consumption kinetics. Thus, the higher polyphenol loss (Ws-Wc) was related to higher O_{max}, O_{int}, O_{10}, O_{min}, $t_{O\text{-}90}$, t_{Oint} and to lower oxygen consumption $\Delta O_{max\text{-}min}$ and a lower oxygen consumption rate R_{max}. (Supplementary Table S1).

Anthocyanins were the phenolic compounds most affected by oxygen consumption, with differences between Wc and Ws exceeding 40 mg/L in the reds and 8 mg/L in the rosés (Table 3). Those reds with a higher total anthocyanin content and less polymerized anthocyanins were found to consume oxygen faster (Table 3). The total anthocyanin content decreased significantly with oxygen consumption: 28% in the reds and 36% in the rosés. Oxygen favors the oxidation of certain phenolics and their reaction with anthocyanins or compounds such as acetaldehyde, pyruvic acid and glyoxylic acid (Bakker & Timberlake, 1997; Es-Safi, Le Guerneve, Cheynier, & Moutounet, 2000; Fulcrand, Benabdeljalil, Rigaud, Cheynier, & Moutounet., 1998; Mateus et al., 2003), which can also be formed by oxidation of ethanol, malic, lactic and tartaric acids (Palacios & Chatonnet, 2018). The loss of total anthocyanins is also related to oxygen consumption, since these compounds undergo polymerization and condensation with other compounds, processes accelerated by oxygen. The loss of total anthocyanins was greater in wines with a higher initial content, so it was greater in the reds that consumed oxygen more quickly. Rosé wines with a high oxygen consumption capacity lost 40% of their anthocyanins by oxygen consumption compared to 26% of rosé wines with a low oxygen consumption capacity. These results agree with the formation of polymeric anthocyanins (Table 3), increasing between 17% and 22% in all rosé wines. That indicated that the rosé wines with a higher anthocyanin content suffered a significant oxidative loss without participating in the formation of polymeric anthocyanins.

As observed with total polyphenols, the difference in anthocyanins correlated negatively with the kinetic parameters $\Delta O_{max\text{-}min}$ and R_{max}, indicating that the loss of anthocyanins due to oxidation was greater when more oxygen was consumed and at a higher rate. However, the increase in polymerization only showed significant correlations with O_{max}: the higher the polymerization, the more oxygen the wines were able to assimilate (Supplementary Table S1) because oxygen favors polymerization reactions.

There was no significant relationship between the initial content of hydroxybenzoic acids (HBA), phenolic alcohols and the capacity and speed of oxygen consumption of the white, rosé and red wines. In general, HBA content increased after oxygen consumption except for the rosé wines, which showed a higher loss of HBA the faster these wines consumed oxygen. HBA increased with the capacity and speed of oxygen

consumption and a positive correlation was found between the increase in these compounds and $\Delta O_{max\text{-}min}$ and R_{max} (Supplementary Table S1). The greatest decrease in phenolic alcohol concentrations occurred in whites with a greater capacity to consume oxygen, while in the reds this loss was greater the faster the rate of oxygen consumption. The rosé wines with higher initial hydroxycinnamic acid (HCA) content were able to consume more oxygen and more rapidly, showing a greater loss of these compounds (Table 3). The effect of oxygen consumption in HCA of the red wines was different, increasing in the fast-consuming ones and decreasing in the slow consuming ones. The whites consuming oxygen rapidly showed an initial flavonol content more than double that of the slow consumers. This same trend was observed in the red wines, so those that initially presented a higher content of flavonols or ellagic acid and a lower one in stilbenes consumed more oxygen and more rapidly, suffering greater losses of these compounds in the oxidation process.

3.2.2. Effect of oxygen on color

The color intensity (CI) of the wines increased after consuming oxygen as described by other authors (Cano-López et al., 2008; Carrascón et al., 2015; Pérez-Magariño et al., 2007). The white wines that consumed more oxygen presented the greatest increase in CI and the greatest loss of polyphenols and phenolic alcohols. This is probably due to the increase in yellow-brown tones produced by the oxidation process that favors the formation of brown compounds by polymerization of phenolic compounds (Kanavouras et al., 2020). In the rosé and red wines, an increase in yellow and blue tones and a decrease in red tones were observed, a phenomenon due to the formation of more stable pigments attributed to the condensation of anthocyanins and flavanols mediated by acetaldehyde and/or by ethyl and vinyl bridges (Atanasova et al., 2002; Bakker & Timberlake, 1997; Mateus et al., 2003; Revilla et al., 1999). This was corroborated by the polymeric anthocyanins data (Table 3). The red wines that consumed oxygen more rapidly showed the greatest increases in CI, which was related to the greater losses of anthocyanins, ellagic acid and phenolic alcohols and to the greater increase in polymeric anthocyanins. The rosé wines presented the greatest tonality and a greater increase in tonality due to oxygen consumption. Although no significant differences were found in the CI of the rosés due to oxygen consumption and the speed of oxygen consumption, those wines that consumed more oxygen and faster showed more browning. This process could be due to the greater decrease in total polyphenols, HCA, flavanols and flavonols in the rosé wines that consumed more oxygen, and to the greater loss of HBA and flavonols in the case of those that consumed oxygen more rapidly.

3.2.3. Effect of oxygen on volatile compounds

Free acetaldehyde content presented a different trend depending on the wine type. In whites, a significant increase of around 73% was observed, which could be due to ethanol oxidation (Palacios & Chatonnet, 2018; Singleton, 1987), and which increased with the maximum oxygen consumption rate. However, the concentration values were below 20 mg/L and did not indicate any defect in the wines. Free acetaldehyde decreased in the rosés and reds, probably due to the reaction between acetaldehyde and the phenolic compounds, mainly anthocyanins, as previously stated, and especially if oxygen consumption was fast. With slow oxygen consumption both white and rosé wines (7 whites and 4 rosés) showed a slight increase in acetaldehyde. It should be noted that acetaldehyde may also be released from its form bound to SO_2 during a decrease in free SO_2. Carrascón et al. (2015 and 2017) observed an increase in acetaldehyde in all types of wine, while Mislata et al. (2020) found a decrease in some reds. Therefore, the different results in acetaldehyde evolution during air saturation could be due to the initial characteristics of wines, since Bueno et al. (2018) indicated that the presence of aldehyde-reactive polyphenols (mainly anthocyanins and small tannins) avoided acetaldehyde accumulation. The initial ethyl acetate content was similar in all the wines: 60–80 mg/L. This is above the odor threshold (12.3 mg/L), but at low levels it can contribute to the fruity aroma and impact positively on wine aroma. The oxidation process that all the wines underwent caused a reduction in their content (average 18.6 mg/L), which was significantly greater in the reds than in the whites and rosés and was independent of the capacity and rate of oxygen consumption (Table 3). The initial content of fatty acids was higher in the white wines followed by the rosés and reds and increased around 0.48 mg/L after saturation with air and 3 months bottle storage. Although this increase was not significant, it was greater in the white wines. The vanillin derivates content did not change significantly with the oxygen supply, although a higher initial content was observed in the whites that consumed oxygen more slowly.

A significant decrease in the content of ethyl esters of straight-chain fatty acids (EE-SCFA, ≈50–58%), ethyl esters of branched-chain fatty acids (EE-BCFA, ≈48–56%) and alcohol acetates was observed (≈34–65%) in all the wines. This indicated a loss of their fruity and floral aromas (Escudero et al., 2007; Ferreira et al., 2000). Carrascón et al. (2015) and Mislata et al. (2020) also found a significant decrease of these compounds, although this was generally lower than in our study. Carrascón et al. (2015) also stated that these changes cannot be only associated with the oxidation process, but also with hydrolysis/esterification equilibria and with the hydrolysis of precursors. Many works indicated that the evolution of these volatile compounds during wine aging can be related to their different hydrolysis/esterification equilibria (Makhotkina & Kilmartin, 2012; Ramey & Ough, 1980). In general, a decrease in the concentration of ethyl esters and alcohol acetates was observed depending on aging time and conditions (Ferreira, Escudero, Fernandez, & Cacho, 1997; González-Centeno, Chira, & Teissedre, 2016; Patrianakou & Roussis, 2013) but to a lesser extent than that observed in this study. The decrease in EE-SCFA and alcohol acetates was lower in the red wines and higher in the whites, while the decrease in EE-BCFA was higher in the reds. The initial content of EE-SCFA was higher and that of EE-BCFA was lower in the reds that showed fast oxygen consumption. Thus, the decrease in EE-SCFA, EE-BCFA and alcohol acetates by exposure to oxygen was greater in the wines with a high initial content and with the lower rate of oxygen consumption (Table 3). However, the wines that consumed most oxygen and fastest were those with the highest initial EE-BCFA content. They presented the greatest loss of these compounds.

After undergoing oxidation, the content of higher alcohols in the wines in relation to their initial content, differed according to the type of wine. They decreased in the whites and reds but increased in the rosés without any relation to the capacity or speed of oxygen consumption. On the other hand, furanic aldehydes and Strecker aldehydes showed a statistically significant increase (averages of 75% and 24%, respectively) due to contact with oxygen. In general, the Strecker aldehydes were significantly increased by the effect of consumed oxygen. These compounds can provoke the appearance of negative notes, such as honey, malty aromas and/or ripe fruit (Culleré et al., 2007). The increase in Strecker aldehydes can be due to the oxidation of higher alcohols, their formation from amino acid precursors and/or by their release from bound forms once SO_2 is consumed (Bueno, Carrascón, & Ferreira, 2016 and, 2018). The first two mechanisms were concluded to be less relevant and to occur mainly when free SO_2 concentrations were less than 5 mg/L. Most of the Strecker aldehydes can be formed during fermentation and the bound forms are released during the oxidation of wines as SO_2 decreases. Different behavior was also observed depending on both the aldehyde and the wine. The concentrations of the Strecker aldehydes in the studied wines were above their odor thresholds (Culleré et al., 2007), except for 2-methylbutanal, so could have a negative sensory effect. A greater increase was found in the rosé wines, independent of their oxygen consumption capacity or speed (Table 3). However, furanic aldehydes increased similarly in the white and rosé wines, especially in those that consumed more oxygen, and at a faster rate in the case of the rosés.

The initial content of C6 alcohols was lower in the white wines than in the rosés and reds. The whites that consumed more oxygen and more

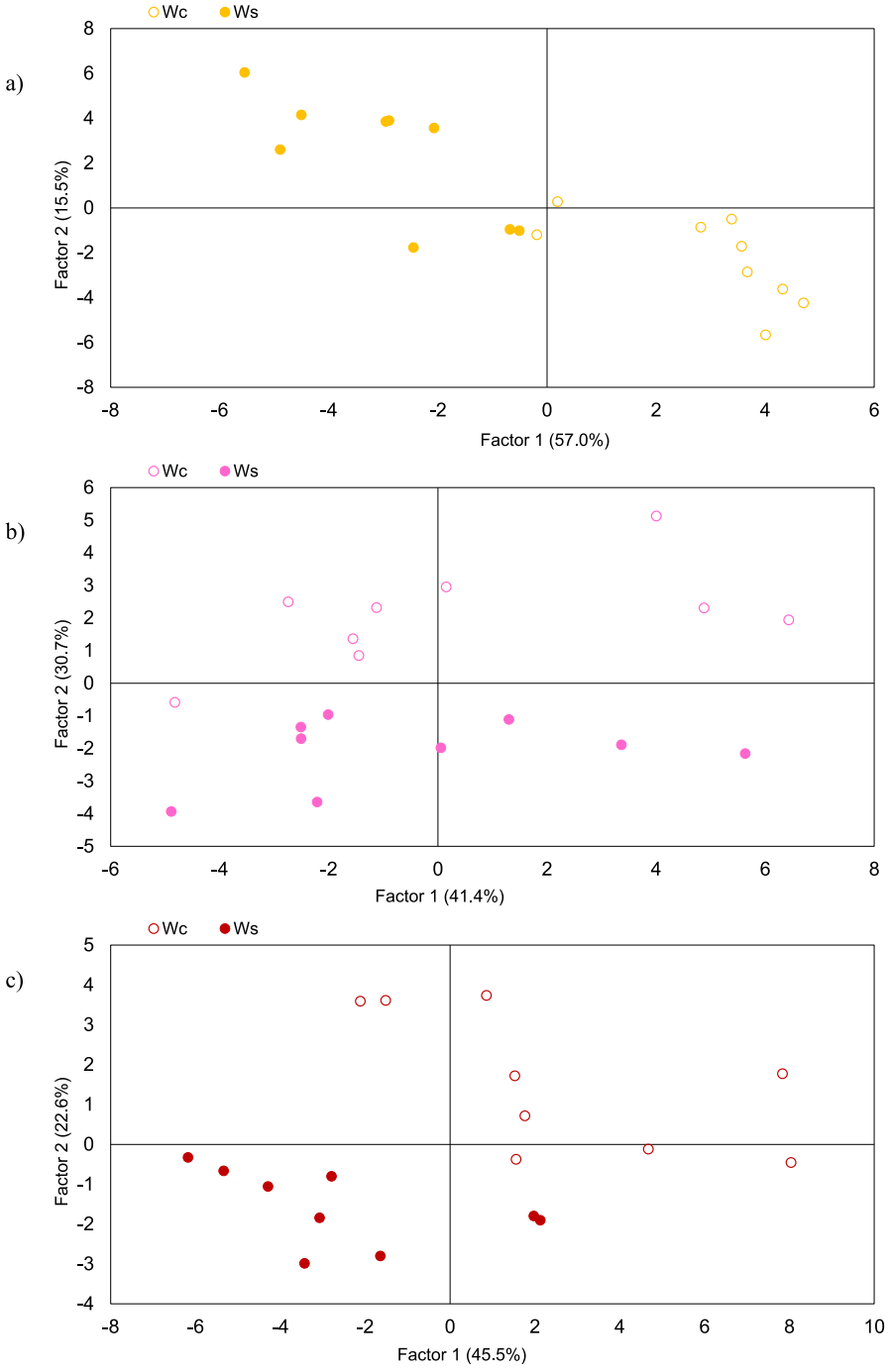

Fig. 2. Distribution of a) white wines, b) rosé wines and c) red wines at Wc (wine control) and Ws (wine saturated) in the plot defined by two first factors. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

quickly had the highest initial content of these alcohols. The white Ws wines showed a higher content of these compounds, while a decrease was observed in the reds and rosés.

The terpene content of red Ws wines was lower than that of Wc wines, while it increased in the whites and rosés, especially in those that consumed little oxygen and slowly. Although the terpene concentrations showed statistically significant differences, the quantitative values were low and the slight differences may be due to their release and/or equilibrium and rearrangement (Marais, 1983; Slaghenaufi & Ugliano, 2018; Williams, Strauss, Wilson, & Massy-Westropp, 1982). This increase in terpenes was corroborated by showing a significant and positive sign correlation with most of the kinetics parameters, except with $\Delta O_{max\text{-}min}$ and R_{max} which were negative. The volatile phenol content after three months decreased more in the white wines which had the highest initial content. The rosé wines with the highest initial content consumed the most oxygen and showed greater losses than those with low consumption.

3.3. Multivariate analyses

Factorial analyses were performed with the chemical variables that presented statistically significant differences in the ANOVA to determine those contributing most to the air saturation effect in each type of wine. Fig. 2a shows the distribution of the white wines studied before (Wc)

Table 4
Factor loadings after varimax rotation of the studied wines.

Compounds	White wines		Rosé wines			Red wines		
	Factor 1	Factor 2	Factor 1	Factor 2	Factor 3	Factor 1	Factor 2	Factor 3
EE-SCFA[a]	**0.903**	−0.283		**0.773**	0.404	**0.883**	0.346	
EE-BCFA[a]	**0.829**		0.360		**0.866**		**0.879**	
Alcohol acetates	**0.679**	−0.501	**0.618**	**0.658**		**0.768**		
Furanic aldehydes	−0.291	**0.853**	−0.383	**−0.689**		**−0.740**	−0.402	
Strecker aldehydes	−0.580	0.423	0.541	**−0.706**		**−0.860**		0.345
Acetaldehyde		**0.869**						
Ethyl acetate	**0.727**		−0.254	0.368	**0.793**		**0.904**	
Total anthocyanins			**0.956**			**0.859**		
Polymeric anthocyanins			**−0.889**	−0.252	−0.278	**−0.810**	0.389	0.304
Color intensity	−0.260	**0.875**	**0.835**		−0.445			**0.973**
Eigenvalue	4.56	1.24	3.7	2.8	1.0	4.1	2.0	1.3
Cumulative variance (%)	57.0	72.5	41.4	72.1	83.5	45.5	68.2	83.0

Loadings lower than absolute values of 0.250 are not shown. Values in bold indicate the highest weight of each compound in each factor.
[a] EE-SCFA: ethyl esters of straight-chain fatty acids, EE-BCFA: ethyl esters of branched-chain fatty acids.

and after (Ws) oxygen consumption in the plane defined by the first two factors with an eigenvalue >1, which explained 72.5% of the total variance. The variables associated with the two factors allowed the whites to be differentiated by oxygen consumption effect. The wines saturated with air and three months bottling (Ws) were located in the upper left zone of the plane, while the control unsaturated wines (Wc) were in the lower right zone. The Ws wines were characterized by higher furanic aldehydes, acetaldehyde, and color intensity and lower EE-SCFA, EE-BCFA and alcohol acetates than the Wc wines (Table 4).

In the rosé wines factor analysis selected three with an eigenvalue >1, which explained 83.5% of the total variance, and the first two explained most of the variability (72.1%). The variables associated with factor 2 were the most significant when differentiating the wines (Fig. 2b), with the Ws wines at the bottom of the plane. This was due to the increase in furanic aldehydes and Strecker aldehydes and to the decrease in EE-SCFA and alcohol acetates, compounds associated negatively and positively with factor 2, respectively (Table 4).

Factor analysis of the red wines selected three with an eigenvalue >1, which explained 83.0% of the total variance. The scores of the red wines in the plane defined by the first two factors, which explained 68.2% of the total variance, are shown in Fig. 2c. The variables associated with factors 1 and 2 were those most important when differentiating between the Wc and Ws wines. EE-SCFA, alcohol acetates, and total anthocyanins were positively associated with factor 1, while polymeric anthocyanins, furanic aldehydes and Strecker aldehydes were negatively associated with factor 1 (Table 4). This indicated that an increase in polymeric anthocyanins, furanic aldehydes and Strecker aldehydes and a decrease in EE-SCFA, alcohol acetates, and total anthocyanins occurred in the wines after air saturation and three months of storage.

As mentioned previously, oxygen consumption involves modification of the chemical compounds in wine, depending on its type. Therefore, a principal component analysis was performed to evaluate the relationship between the oxygen consumed ($DO_{max\text{-}min}$) by wine type and the variation in the content of the different compounds analyzed in the Wc and the Ws wines. The analysis used the initial chemical variables that were significant in the differentiation of wines by oxygen consumption. Analysis of the white wines selected one component with an eigenvalue greater than 1, which explained 63.5% of the total variance. This component is associated positively with phenolic alcohols (Fig. 3a) and negatively with oxygen consumption producing a greater increase in furanic aldehydes, in acetaldehyde and CI caused by greater browning of the wines. The wines located in the negative axis consumed more oxygen (3-W, 6-W, 7-W, 8-W) and more rapidly, so 6-W and 7-W showed a higher rate and were those with the lowest total acidity (Fig. 3b), showing no differences with respect to SO_2 content (Table 1). However, the whites located in the positive axis consumed less oxygen and at a slower rate. They were defined by a lower phenolic alcohol loss (Table 3). In the case of the rosé wines, three components with an eigenvalue greater than 1 were obtained, which explained 84.3% of the total variance. The first component, which explained 48.4% of the variance (Fig. 3c), was associated positively with the increase in furanic aldehydes and oxygen consumption. The rosé wines 10-W, 12-W and 17-W positively correlated with this component and consumed more oxygen at a medium rate (Fig. 3d). The other rosés consumed less oxygen (except 17-W) and more slowly and were located in the negative axis of this component. They were defined by a lower total polyphenol loss. All the reds were major consumers of oxygen and the analysis differentiated between those that consumed oxygen rapidly (more than 10 hPa/h) and those that consumed it at a medium rate (between 5 and 10 hPa/h). In this case, the analysis selected three components with an eigenvalue greater than 1, which explained 83.4% of the total variance (Fig. 3e). The red wines with a fast oxygen consumption rate were located in the positive axis and associated with a lower loss of EE-BCFA and increased polymeric ACY. Those with a mid-oxygen consumption rate were in the negative axis and defined by a lower loss of anthocyanins, flavanols, phenolic alcohols, EE-SCFA and ethyl acetate (Fig. 3f).

4. Conclusions

This preliminary study indicates that all wines studied can be high oxygen consumers and the rate of oxygen consumption is independent of the oxygen consumption capacity. There is greater variability in oxygen consumption parameters in white and rosé wines than in reds. The whites with higher initial contents of total polyphenols, color intensity, acetaldehyde and C6 alcohols showed a higher oxygen consumption capacity, leading to a loss of total polyphenols, phenolic alcohols and increased browning. The higher oxygen consumption capacity of rosé wines was also related to a higher initial content of total polyphenols, hydroxycinnamic acids, furanic aldehydes and volatile phenols. All the reds behaved as high oxygen consumers, but those with a higher initial content of anthocyanins, ellagic acid, flavonols and straight-chain fatty acids showed a higher oxygen avidity and consumed oxygen faster. These red wines showed a higher loss of anthocyanins, ellagic acid and phenolic alcohols. This was reflected in a higher color stabilization due to a greater increase in polymeric anthocyanins and color intensity. They also showed a higher loss of volatile compounds such as ethyl esters of straight-chain fatty acids and alcohol acetates related to fruity aromas. There are still commercial wines that have SO_2 levels well above what is needed. Therefore, the oxygen consumption kinetics of more types of wines need to be evaluated to determine their oxidation tendency. It is important to control winemaking processes which may increase oxygen uptake. The level of free sulfur dioxide should also be adjusted to reduce its dosage to the minimum required by current

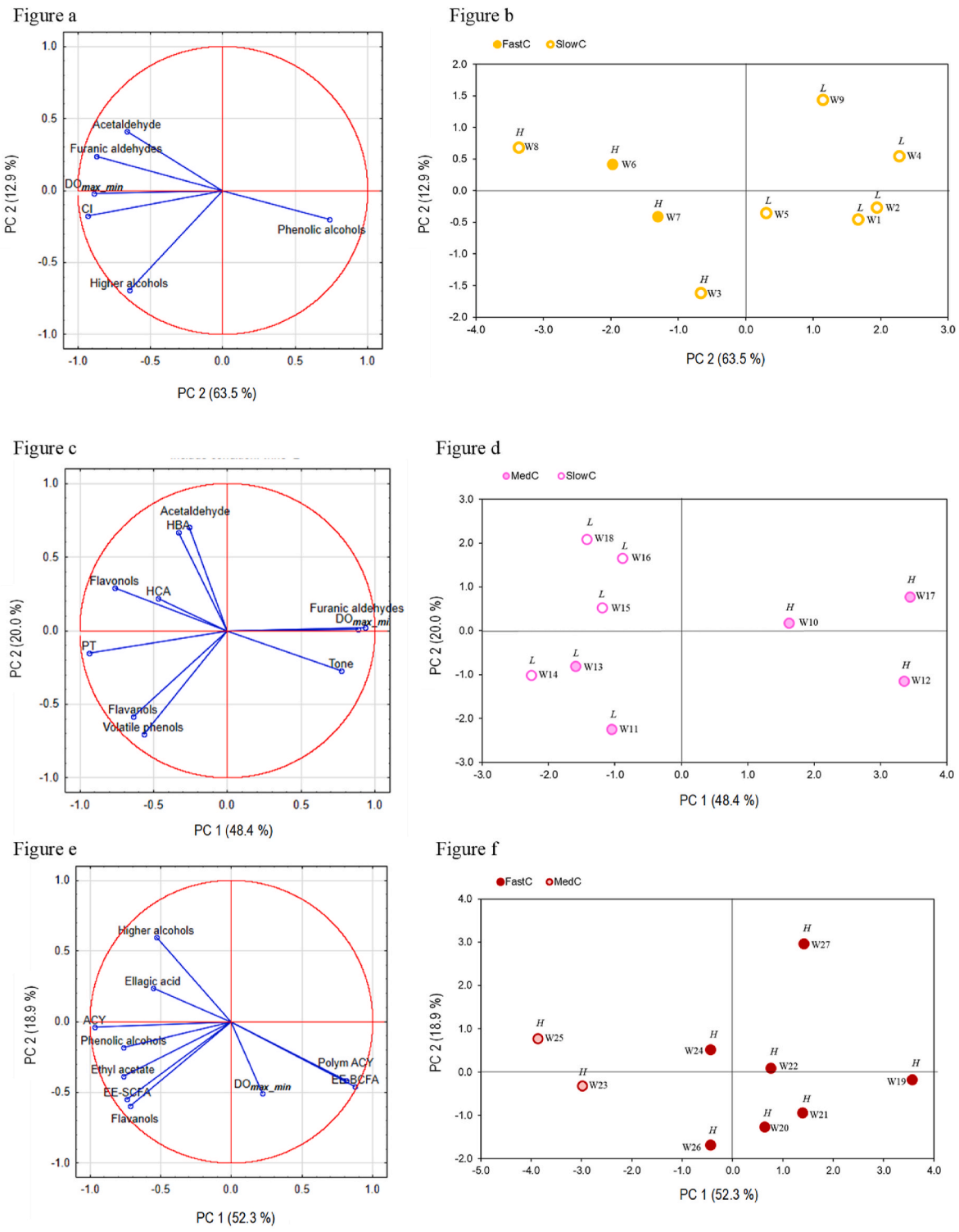

Fig. 3. Distribution of variables (left column) and wines (right column) for a-b) white, c-d) rosé and e-f) red wines with oxygen consumption rate slow (SlowC), medium (MedC) and fast (FastC) using principal component 1 and 2. Each wine is labelled with its number according to Table 1 and with the oxygen consumption capacity high (H) or low (L). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

demands.

CRediT authorship contribution statement

Silvia Pérez-Magariño: Methodology, Supervision, Writing – original draft, Writing – review & editing, Project administration, Funding acquisition. **Ana Martínez-Gil:** Investigation, Writing – original draft, Writing – review & editing. **Marta Bueno-Herrera:** Formal analysis, Investigation. **Ignacio Nevares:** Conceptualization, Methodology, Formal analysis, Writing – review & editing, Supervision, Project administration, Funding acquisition. **María del Alamo-Sanza:** Conceptualization, Methodology, Formal analysis, Writing – original draft, Writing – review & editing, Supervision, Project administration, Funding acquisition.

Declarations of competing interest

None.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2023.114786.

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