

Changes in the volatile composition of red wines during aging in oak barrels due to microoxygenation treatment applied before malolactic fermentation

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Abstract Microoxygenation is a wine-making technique consisting in the addition of small and controlled amounts of oxygen. This study has examined the effect of this technique on the volatile composition of two red single variety wines during two successive vintages. The microoxygenation treatment was applied at the end of alcoholic fermentation and before beginning malolactic fermentation. Once the microoxygenation treatment had finished, wines were aged in new American oak barrels for 12 months. The results obtained showed that the microoxygenation treatment did not cause significant changes in the varietal and fermentation volatile compounds, however microoxygenation slowed down the extraction of some of the volatile compounds extracted from wood. A varietal and vintage effect was also observed for some of the compounds studied.

Keywords Volatile compounds · Red wine · Microoxygenation · Oxygen

Introducción

Microoxygenation is a winemaking technique developed in France at the beginning of the 1990s which is gaining popu-

larity all over the world. It consists in the addition of tiny and fixed amounts of oxygen to wine at different stages of the winemaking process. The rate at which oxygen is supplied to the wine is equal or lower than the rate at which it is consumed by the wine in order to avoid oxygen accumulation.

The technique is mainly used in red wines and, as such, can be applied during any stage of the winemaking process. However, oxygen is usually sprayed during some of the following stages: during the alcoholic fermentation and at the end of alcoholic fermentation and before beginning malolactic fermentation [1]. Furthermore, oak chips are commonly used in combination with the microoxygenation process in so-called “industrial aging” [2] in order to imitate aging in oak barrels.

Friend or foe, it is clear that oxygen plays a very important role in the vinification process: on the one hand it is responsible for the oxidation of phenolic and volatile compounds and, on the other, the presence of oxygen in the adequate dose improves the sensorial characteristics of the wine. These positive effects of oxygen are: improved intensity and stability of wine colour as well as the palatability and structure [3–10]. This is due, among other factors, to the fact that oxygen takes part in condensation and polymerisation reactions between tannins and anthocyanins. These reactions produce new pigments which can stabilise wine colour and reduce astringency [10, 11–16]. Furthermore, the addition of small and controlled amounts of oxygen enhances the development of fruity flavours, integrates the aroma of the wood, reduces the reductive and vegetal properties (since oxygen can oxidise unwanted sulphur compounds, like H₂S which has a rotten egg smell) and it can also reduce green and herbaceous aromas, especially in red wine, which probably originate from the so-called leaf alcohols [3, 17]. Furthermore, it has also been taken into

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account that greater stability of the phenolic composition of a wine has direct repercussions on the aroma, which should be more fresh and fruity.

Although this technique has been widely applied in recent years in several countries such as France, Italy, Australia, New Zealand, the United States and Chile, it still poses some unanswered questions, for example: varietal effect, optimum doses, when it is best applied for each kind of wine, etc.

However, very few papers have been published on these topics and most of them focus on the influence of oxygen on phenolic compounds in wines [3–5, 7, 10, 18–20] and only some of them focus on sensorial properties: taste and aroma [6, 8, 21]. No paper has been published in scientific journals with regard to the effect of microoxygenation on the volatile composition of wine.

The purpose of this study was to evaluate the effect of the application of small and controlled amounts of oxygen after alcoholic fermentation and before malolactic fermentation on the volatile composition of two single variety red wines. The evolution of the aroma compounds has been studied during aging in barrel for 12 months. This study was carried out in two successive vintages and with wines from two different Appellations of Origin.

Materials and methods

Elaboration process and microoxygenation treatment

Two red-single variety wines were elaborated from two autochthonous Spanish grape varieties: Tinta de Toro (TT) and Mencía (MC), which are the main varieties used to elaborate the wines of with the Appellations of Origin Toro and Bierzo, respectively. These wines were elaborated in the research winery of the Enological Station of Castilla y León and during two successive vintages. The traditional red wine vinification method was followed. The grapes (about 5,000 kg per variety) were harvested in their different production areas in the optimum harvest date and quickly transported to the Enological Station in Rueda in 25 kg plastic boxes. The wines were elaborated according to the following process: grapes were de-stemmed, crushed and sulphited with 50 mg/L. Alcoholic fermentation took

place in stainless steel tanks without yeast inoculation. Temperature was controlled not to rise above 25 °C. Once alcoholic fermentation finished (sugar level under 2 g/L) and after 14–15 days of maceration, the wines were strained off from the tanks and transferred to two tanks per variety; one of the tanks was supplied with small and controlled amounts of oxygen, microoxygenated wine (MO), while the other tank was non-microoxygenated, i.e. the control wine (C). The treatment was carried out by means of microoxygenation equipment supplied by Oenodev (France). Pure oxygen was slowly diffused through a ceramic membrane which was hung inside the tank but without touching the bottom, thus, the small oxygen bubbles were dissolved into the wine by reaching the surface of the tank.

The doses of oxygen applied were set up by sensorial analysis by a group of expert wine-makers and technicians according to the initial characteristics of the wine (structure, astringency, presence of green tannins and vegetal and reductive aromas). The doses of oxygen applied were higher for a brief period of time to eliminate some reductive compounds which sometimes appear just after alcoholic fermentation and the vegetal properties present in wines, allowing improved fruity expression. After that, the flows were reduced to provide colour and tannin stabilisation and to complete the structuring phase before starting malolactic fermentation. The final point of the oxygen addition was also determined by sensorial analysis, when the experts considered that the wines had reached the desired characteristics in the wine: removal of the reductive notes, change of the green tannins into hard tannins and an increase in the structure and body of the wine (Table 1). The concentration of malic acid was evaluated every day during the microoxygenation treatment in order to check that the malolactic fermentation had not commenced. In both kinds of wine, microoxygenated and control, this second fermentation was carried out spontaneously, without the inoculation of lactic acid bacteria and when the malolactic fermentation was finished (malic acid \leq 0.1 g/L) both the microoxygenated and control wine were racked off to three new American oak barrels where they were aged for 12 months. Samples were taken and analysed from two of the three barrels, whilst the third one was used to fill the other two.

The evolution of the wine volatile compounds were studied by analysing the following samples: A, at the end of the

Table 1 Amounts of oxygen in ml/L/month of wine added in the different wines and vintages

Variety grape	First vintage		Second vintage	
	First dose ml/L/month O ₂ (days)	Second dose ml/L/month O ₂ (days)	First dose ml/L/month O ₂ (days)	Second dose ml/L/month O ₂ (days)
Mencía	60 (10)	30 (8)	60 (10)	30 (10)
Tinta de Toro	50 (10)	20 (10)	50 (10)	30 (10)

alcoholic fermentation (initial wine); B, at the end of the microoxygenation treatment; C, at the end of the malolactic fermentation; 0 MB, sample taken when the wine was transferred to the barrel; 4 MB, 4 months of aging in barrel; 8 MB, 8 months of aging in barrel and 12 MB, 12 months of aging in barrel.

Reagents

Dichloromethane (HPLC grade) and the chemical standards used for quantitative analysis were purchased from Sigma-Aldrich (Sigma-Aldrich Corporation, St. Louis, MO, USA).

Analytical method

The volatile compounds were extracted by liquid-liquid extraction following the method described by Moio et al. [22]. Two hundred millilitres of wine, 5 ml of dichloromethane and 50 μ L of an internal standard (a solution of 1000 μ g/L of 2-octanol) was introduced into a special flask in which oxygen had been previously removed by nitrogen. The flask was placed in an ice bath and stirred at 500 rpm for 3 h. This was followed by phase separation and the organic phase was kept and stored at -80°C until analysis. Each sample was extracted twice.

Each extract of volatile compounds was analysed by gas chromatography. A GC (Hewlett-Packard 6890) equipped with an FID detector was used for the quantification of the volatile components. The column was a 50 m \times 0.25 mm cCarbowax BTR with a 0.33 μ m thickness stationary phase. The injector temperature was kept at 220°C and the FID was kept at 250°C . The carrier gas was helium at 1.3 ml/min. The temperature programme was 40°C held for 8 min, raised to 85°C at $10^{\circ}\text{C}/\text{min}$ and held for 1 min, raised to 110°C at $2^{\circ}\text{C}/\text{min}$ and held for 1 min and finally raised to 200°C at $3^{\circ}\text{C}/\text{min}$ and held for 40 min [23].

Isolated peaks were identified by comparing their retention times with the retention time of their respective standards, and using their mass spectra data. A Hewlett Packard 5973 mass detector fitted with a Hewlett Packard 6890 GC was used. The ionisation of the samples was achieved at 70 eV under the SCAN mode. The mass range studied was from 30 to 250 m/z . A Hewlett Packard Chem-station equipped with the Wiley 275 library was used for interpreting MS spectra.

The quantification was carried out following the internal standard quantification method. Quantitative data of the identified compounds were obtained by interpolation of the relative areas versus the internal standard area in the calibration graphs built with the respective standards.

Since the repeatability of the chromatographic method was very good (with variation coefficients lower than 0.5%), only one injection of each dichloromethane extract was carried out.

All the analyses were carried out twice.

The most usual oenological parameters analyzed in wineries and Enological Stations were selected, which are: titrable acidity (TA as g/L tartaric acid), volatile acidity and tataric acid (VA as g/L acetic acid) (TarAc g/L) analyzed following the EC methods [24], ethanol (% v/v), pH and potassium (K^+ mg/L) determined according to the OIV methods [25]. These parameters were determined in duplicate.

Results and discussion

Taking into account the large number of volatile compounds identified, the results were analysed by grouping some of these quantified volatile compounds. The compounds were grouped according to chemical similarities. Furthermore, in order to avoid information loss, it was ensured that compounds in the same group showed a similar pattern of evolution. Thus, the following groups were formed: ethyl esters, (ET), ethyl butyrate, ethyl hexanoate, ethyl octanoate, and ethyl decanoate; fusel alcohol acetates, (AA), isomyl acetate, hexyl acetate, phenylethyl acetate; fatty acids, (AC), hexanoic, octanoic, decanoic and dodecanoic acid and fusel alcohols, (FA), isobutyl, isoamyl and β -phenylethyl acetates. The rest of the volatile compounds were studied one by one.

Regarding ethyl esters and fusel alcohol acetates, compounds responsible for the fruity aroma of young wines [26, 27], the addition of small and controlled amounts of oxygen did not cause significant changes in the concentration of these compounds (ET and AA), in MC Wines (Fig. 1a). However significant differences were found in the concentration of AA between the microoxygenated and the control TT wines in the two vintages studied. The effect of the supply with oxygen on the concentration of AA in these wines was different in the 2 years, showing lower levels than the control wines in 2002, and higher in wines of 2003 vintage. However, as for the levels of ethyl esters significant differences were only found in some of the samples taken along the elaboration and aging process.

Then, taking into account the data of the two vintages and the two single-variety wines together, it is possible to assert that microoxygenation did not induce important changes in ET levels. However, the effect of this technique on the concentration of AA should be more deeply studied since the variability of the obtained results, did not allow to establish any general conclusion.

Therefore, from the point of view that the microoxygenation treatment only increased significantly the levels of these compounds in one of the eight cases studied, it is not possible to assert that the microoxygenation treatment enhanced the development of the volatile compounds

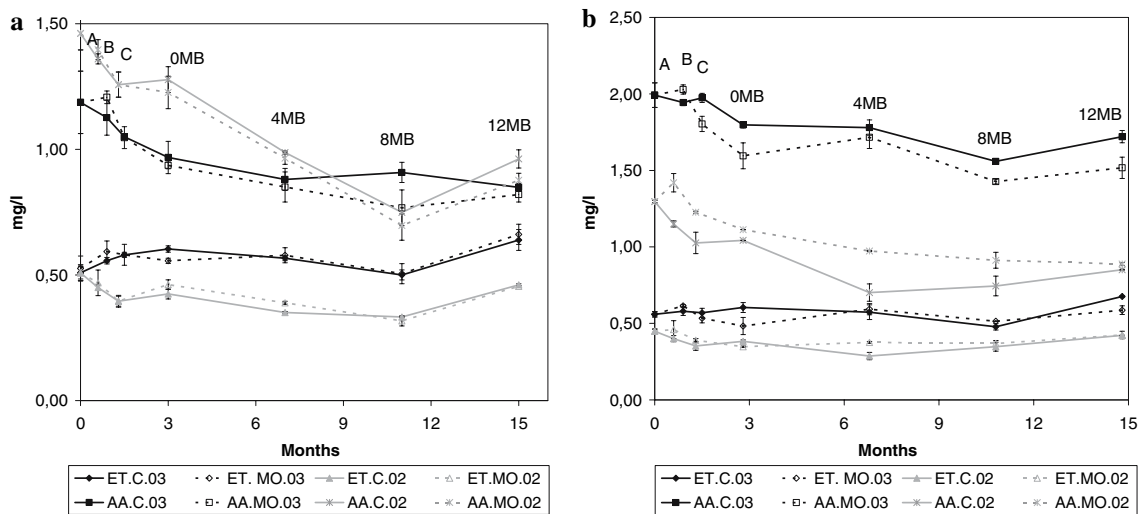


Fig. 1 Evolution of the ethyl esters (*ET*) and fusel alcohol acetates (*AA*) in the MC (**a**) and TT wines (**b**) in the two vintages studied

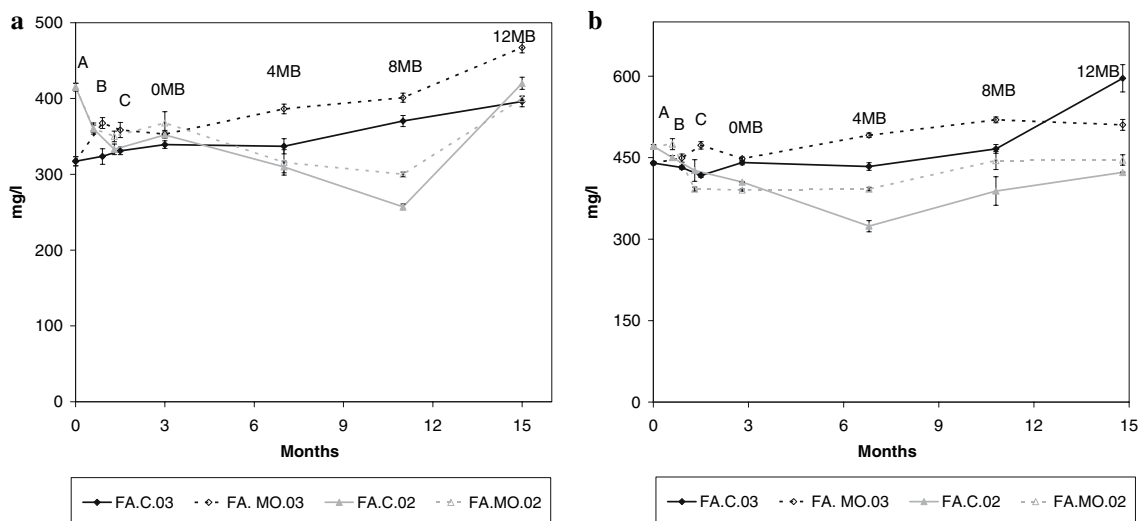


Fig. 2 Evolution of the fusel alcohol (*FA*) in the MC wines (**a**) and TT wines (**b**) in the two vintages studied

responsible for the floral and fruity notes of the wines. So, the more intense fruity aroma in the microoxygenation wines detected previously [1, 28] could be a complex result in which all the changes induced in these and other groups of volatile compounds would be implicated.

As it was expected, both, ethyl esters and fusel alcohol acetates tended to decrease during the storage in barrel, although this reduction was more important for the latter. These results coincide with those found by other authors [29, 30] and they can be explained by the different hydrolysis esterification equilibrium [31], which showed that fusel alcohol ester hydrolyses more rapidly than ethyl esters, with the exception of ethyl decanoate.

Fusel alcohols are, from a quantitative point of view, the major group in the volatile compounds present in wine.

Except for 2-phenylethanol with an aromatic note of roses, the rest of fusel alcohols are associated with not very pleasant aromas described as alcoholic, pungent or fusel (the origin of their name). For that reason, if their concentration in wine is higher than 400 mg/L, wine aroma can be affected in a negative way, although below 300 mg/L these compounds contribute to its complexity [32].

In general, microoxygenation treatment always produced important increase of fusel alcohols levels. Therefore, it could be asserted that the microoxygenation treatment enhanced the formation of these compounds (Fig. 2). Neither a vintage nor a varietal factor was found. The effect of the microoxygenation treatment on these compounds were, in general, the same in the two vintages and for the two single variety wines studied.

It was also observed that in general, the differences between the control and the microoxygenated wines were getting bigger during the aging in wood. Furthermore, during the last months of the maturation in barrels, the concentration of fusel alcohols increased in all the wines. This fact could be correlated with the hydrolysis of the AA cited previously; however there is not a quantitative correspondence between the decrease of AA and the increase of fusel alcohol levels.

According to the results obtained, it could be asserted that microoxygenation intensifies the olfactory notes correlated with these compounds. Therefore, according to what has been said before, in the case of the MC wines this treatment would enhance the wine aroma complexity, but in the TT wines, microoxygenation would increase the fusel and pungent notes, since the concentrations of fusel alcohols found in these wines were higher than 400 mg/L.

Short chain fatty acids are also formed during alcoholic fermentation by yeast metabolism and they are associated with lacteal and soapy (octanoic acid) notes. However, it seems that these acids play a positive role in the quality of wine aroma as long as they are not present in high concentrations [33].

These compounds showed some results similar to those obtained for the fusel alcohols and others similar to those commented for fusel alcohol acetates and ethyl esters. As such, a vintage effect was found, mainly for the TT wines, although this effect was not as important as for the AA. Neither a variety factor nor differences between the levels of fatty acids between the MC and TT wines were detected. The effect of the microoxygenation treatment was more important for the MC wines whilst in the TT wines significant differences between the control and the microoxygenated wines were only found in some cases (Fig. 3).

In general, it can be said that the microoxygenation treatment enhanced the presence of fatty acids. This fact could be related with a higher hydrolysis of their corresponding ester or even with a low initial formation of esters. However, once again, even if a qualitative correlation could be detected, it is not possible to establish a quantitative correlation between changes in ethyl esters and fatty acids levels.

The slight increase in the levels of fatty acids in microoxygenation wines should induce slight or insignificant differences in the aromatic characteristics of wines associated with them.

Terpenic compounds are characteristics of the kind of grape used. They are responsible for the floral and fruity notes present in wine and, although they are typical for the so-called “aromatic” varieties such as Muscat, Gewürztraminer [34], they can also be present in low concentrations in other varieties known as “neutral” varieties. A vintage and a varietal effect were observed in both MC and TT wines, but only in some of the sample points of the wine-making process and during the period of aging in barrel.

Significant differences were also found in the concentrations of citronellol between the MC and TT wines, the MC wines being the richest ones in this compound. However, no differences were found for linalool between both kinds of single variety wines. Therefore, it is difficult to establish if the addition of small and controlled amounts of oxygen favoured the presence of these compounds or not. A clear positive effect of the microoxygenation on the concentration of citronellol was only detected for MC wines of the second vintage. This increase could mean that the addition of small and controlled amounts of oxygen could increase the hydrolysis reactions from the odourless precursors of citronellol (Fig. 4).

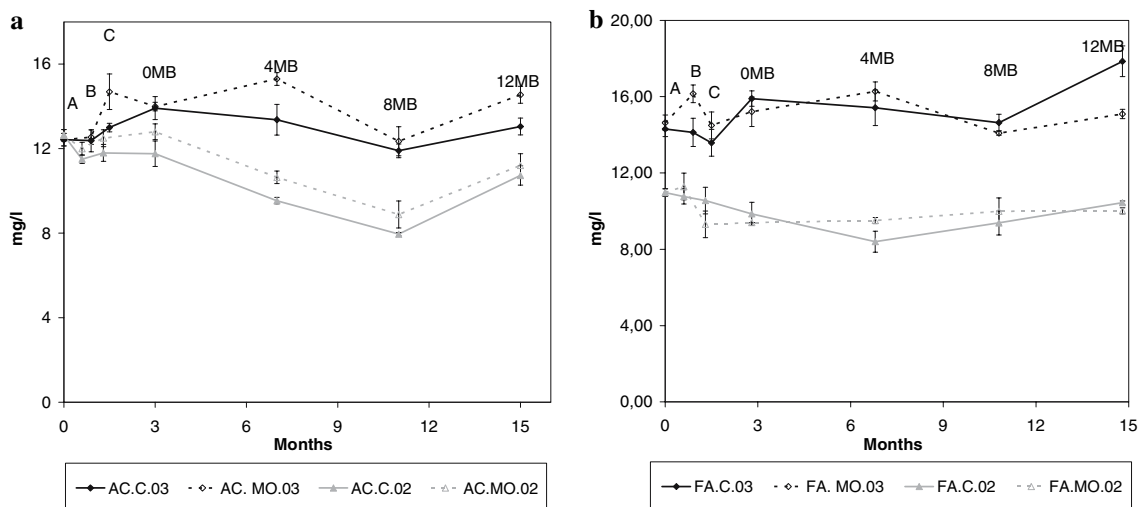


Fig. 3 Evolution of the fatty acids (AC) in the MC wines (a) and TT wines (b) in the two vintages studied

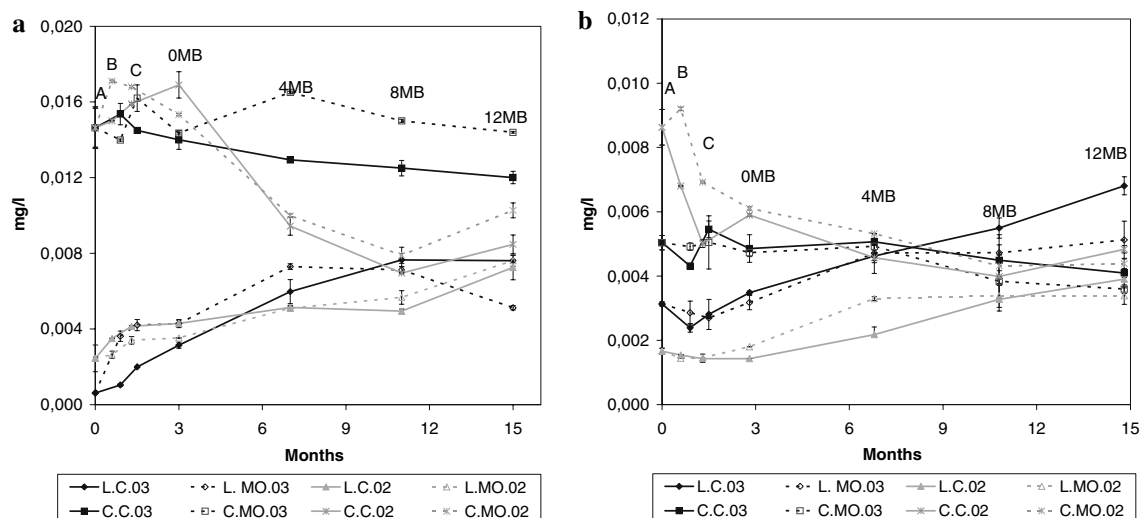


Fig. 4 Evolution of the linalool (L) and citronellol (C) compounds in the MC wines (a) and TT wines (b) in the two vintages studied

In the rest of the wines studied a more irregular behaviour was found. Therefore, it is difficult to know if microoxygenation treatment enhances the presence of the compounds responsible for the floral notes in wines or not.

C6 alcohols are responsible for the green and herbaceous aromas present in wines, and although one of the aims of microoxygenation is to reduce the presence of these notes in wines, no scientific paper supporting this fact has been found.

These compounds also showed varietal differences and, in some cases, vintage and microoxygenation variability factors were also observed. In general, TT wines were richer than MC ones in these kind of compounds and *cis*-3-hexenol was only detected in TT wines. These data coincide with others found in the bibliography, which observed that these compounds play a very important role in the varietal characterisation of wines [35, 36].

Levels of hexanol and *cis*-3-hexenol of wines from the first vintage were significantly higher than those of wines from the second vintage. However, during the aging in barrel the cited differences disappeared. Levels of *trans*-3-hexenol did not show significant differences between vintages (Fig. 5).

As for the effect of the microoxygenation treatment in these compounds, except for the MC wine in the first vintage, microoxygenated wines showed similar or higher concentrations of C6 alcohols than the control wines (Fig. 5). In this way, from these analytical data, it is not possible to assert that microoxygenation reduces the levels of C6 alcohols. Then, the described decrease of grassy notes observed in microoxygenated wines [6, 8, 21] should be correlated with other compounds and not only with changes in the levels of C6 alcohols.

The effect of microoxygenation treatment was very important on the levels of volatile compounds extracted from wood, which were in general more intensive extracted in control wines.

Furfural aldehydes studied in this work were furfural and 5-methylfurfural. These compounds are mainly formed during the toasting of the cask by thermic degradation of the celluloses and hemicelluloses present in wood [37]. The role of these compounds in wine aroma does not seem to be very important due to their high perception threshold [38] although it is thought that these compounds give notes of caramel, almond and toast.

The control wines showed higher concentrations of furfural than the microoxygenated wines in the two vintages studied for the TT wines and in the first vintage for the MC wines. Being these differences very important in all the cases (Fig. 6). The highest concentrations of 5-methylfurfural were also found in the control wines, but these differences were less remarkable, and even in some cases were not significant (Fig. 6). These results seem to indicate that the control wines showed a higher extraction capacity of furfural derivatives compounds than the microoxygenated ones. Other possible explanation to the lower concentrations of furfural and 5-methylfurfural in the microoxygenated wines could be the fact that these compounds are implicated in the aldehyde-generating reactions with flavanols or anthocyanins, producing new colour or colourless pigments [39–40]. However these studies were carried out in model solutions and described adducts have not been detected in wines.

Similar results to those found for furfural compounds were also found for eugenol and *cis*- β -methyl- γ -octalactone, also known as *cis*-whiskey lactone, (Figs. 7, 8). Eugenol is responsible for the clove notes of wines aged in barrel, while *cis*-isomer of whiskey lactone contributes to wine bouquet with notes of coconut, wood, vanillin and toast [41].

Therefore, during the aging in barrel, the control wines showed significantly higher levels of these compounds than the microoxygenated ones in the two single variety wines

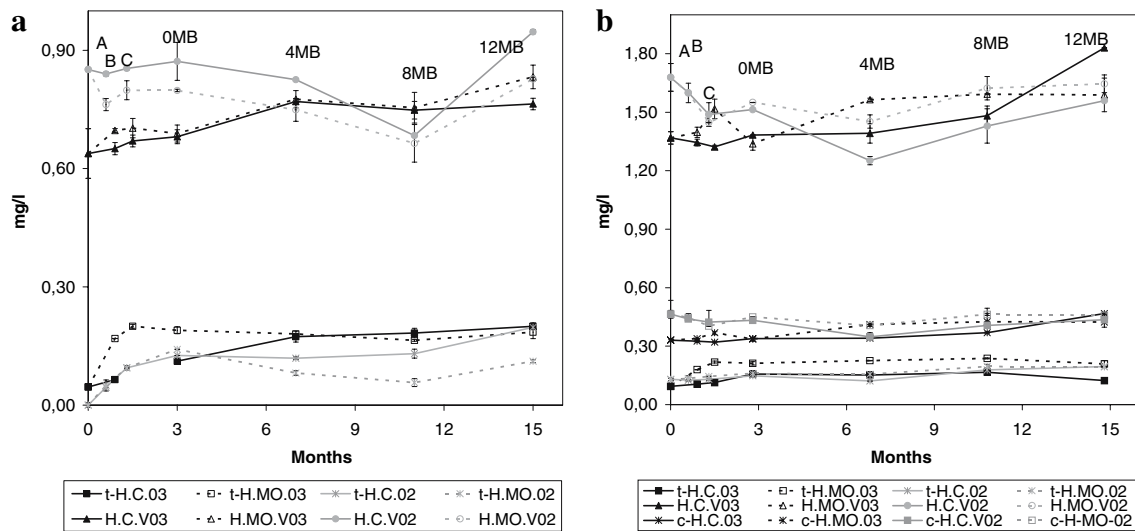


Fig. 5 Evolution of the hexanol (*H*), trans-3-hexenol (*t-H*) and cis-3-hexenol (*c-H*) in the MC wines (a) and TT wines (b) in the two vintages studied

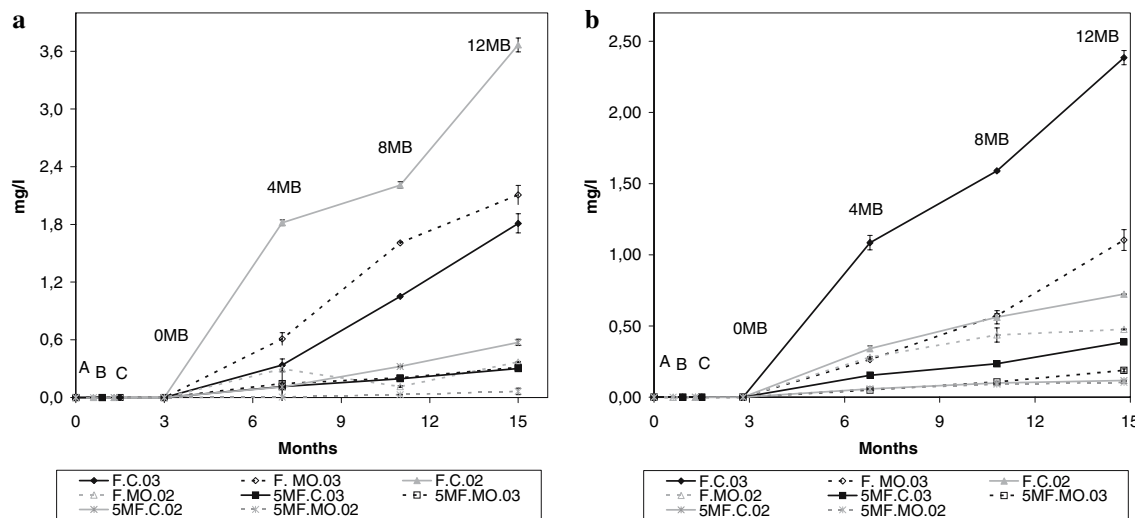


Fig. 6 Evolution of the furfural (*F*) and 5-methyl furfural (*5MF*) in the MC wines (a) and TT wines (b) in the two vintages studied

studied and in the two vintages (except in the MC wines at 4 months in barrel). Once more, the differences found between the control and microoxygenated wines were quite remarkable.

These results do not coincide with those by Lapeña et al. [42] who found higher concentrations of furfural, 5-methyl-furfural and *cis*-whiskey lactone in Cabernet Sauvignon microoxygenated wines in which the addition of oxygen was also done, before malolactic fermentation and aged in barrel for 12 months.

Guaiacol and vanillin also play an important role in the aroma of wines aged in oak. The first one gives smoky flavours whereas “vanillin notes” of wine are also associated to other volatile compounds extracted from wood apart

from vanillin [43]. Furthermore, these compounds showed opposite behaviour to the other volatile compounds extracted from wood.

The addition of small and controlled amounts of oxygen enhanced the extraction of these two compounds from wood, with the exception of MC wines of the first vintage (Figs. 7, 8). Moreover, the differences between the microoxygenated and non-microoxygenated wines became greater as the time of aging in barrel increased.

In order to explain the differences in the compounds extracted from wood found between the microoxygenated and control wines, the oenological classic parameters were studied. These parameters were studied due to their influence on the extraction process (Table 2).

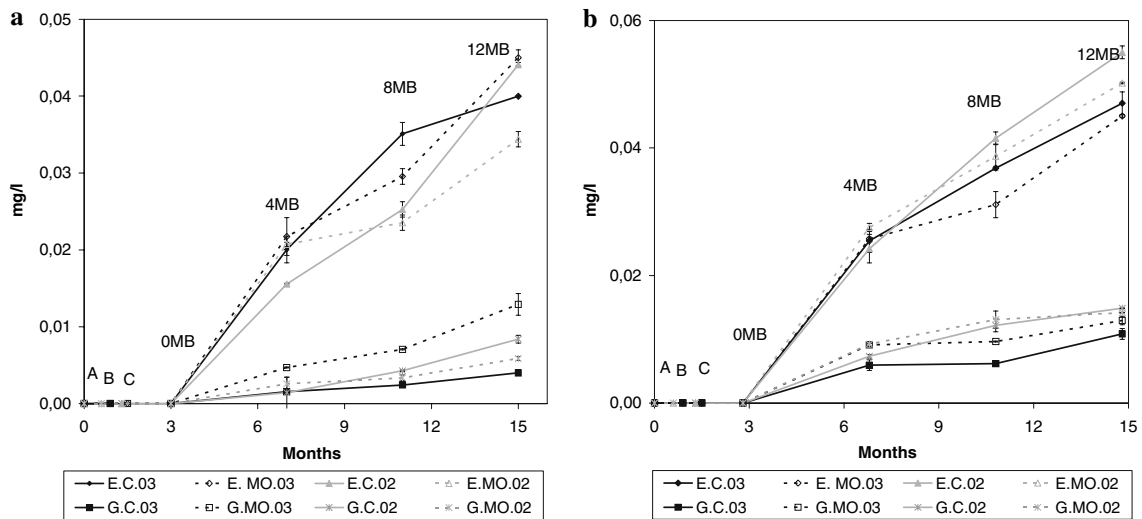


Fig. 7 Evolution of the eugenol (*E*) and guaiacol (*G*) in the MC wines (a) and TT wines (b) in the two vintages studied

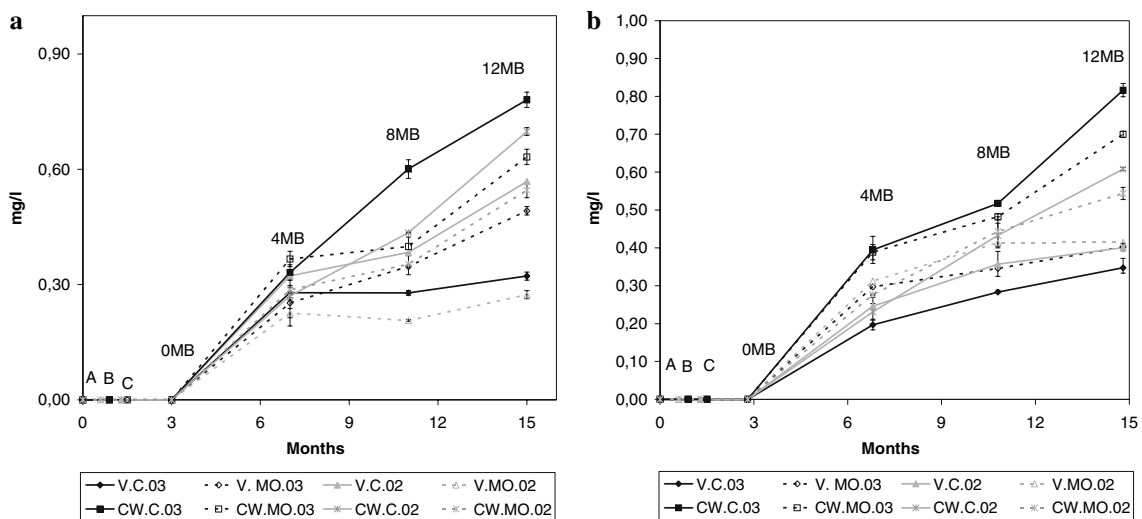


Fig. 8 Evolution of the vanillin (*V*) and cis-whiskey lactone (*CW*) in the MC wines (a) and TT wines (b) in the two vintages studied

Statistical significant differences were not found for the ethanol content, pH, total acidity, tartaric acid and potassium levels between the control and microoxygenated wines (Table 2). However, significant differences were found for the volatile acidity levels, showing the microoxygenated wines higher values than the control ones. This fact was detected on the two kinds of varietal wines and in the two vintages. The higher volatile acidity values of the initial microoxygenated wines could explain the higher extraction of guaiacol [44]. This recent paper describes a positive correlation between the volatile acidity of wines and the levels of guaiacol extracted from barrels. However, it was also observed a similar effect on the extraction of eugenol, although this fact was not found in this work.

Conclusions

According to the results obtained, it can be said that the effect of microoxygenation on some of the wine volatile compounds depends on the kind of wine (variety, origin, etc.), and the vintage. These facts can be related with the differences in the phenolic composition between grape varieties and therefore the doses of oxygen should be determined according to the initial phenolic composition of wines.

Since the behaviour of the compounds extracted from wood was quite similar in all the wines studied; it is possible to assert that the addition of oxygen after alcoholic fermentation slowed down the extraction of furfural

Table 2 Mean values of the oenological classic parameters determined in the initial wines (just before being transferred to the barrels)

Vintage	Wine	pH	Ethanol (%v/v)	TA (g/L)	VA (g/L)	TarAc (g/L)	K* (mg/L)
2002	MC-C	3.62	13.7	4.60	0.39*	1.5	1375
2002	MC-MO	3.65	13.8	4.60	0.49*	1.2	1395
2003	MC-T	3.59	15.4	4.85	0.49*	1.2	1270
2003	MC-MO	3.57	15.3	5.00	0.63*	1.4	1220
2002	TT-C	3.72	13.6	4.60	0.34*	1.2	1405
2002	TT-MO	3.76	13.7	4.45	0.49*	1.4	1405
2003	TT-C	3.65	14.6	4.85	0.53*	1.6	1325
2003	TT-MO	3.65	14.7	4.80	0.59*	1.5	1350

Values marked with * means that statistical differences ($\alpha = 0.05$) between control and microoxygenated wines per variety and per vintage

compounds, cis-whiskey lactone and eugenol, and enhanced the extraction of vanillin and guaiacol. This fact could be correlated with the positive effect of microoxygenation on the integration of wood notes with fruity and varietal aromas.

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