Bottle ageing of three white wines as a function of the stopper's permeability to oxygen

The case of Viognier and Chardonnay

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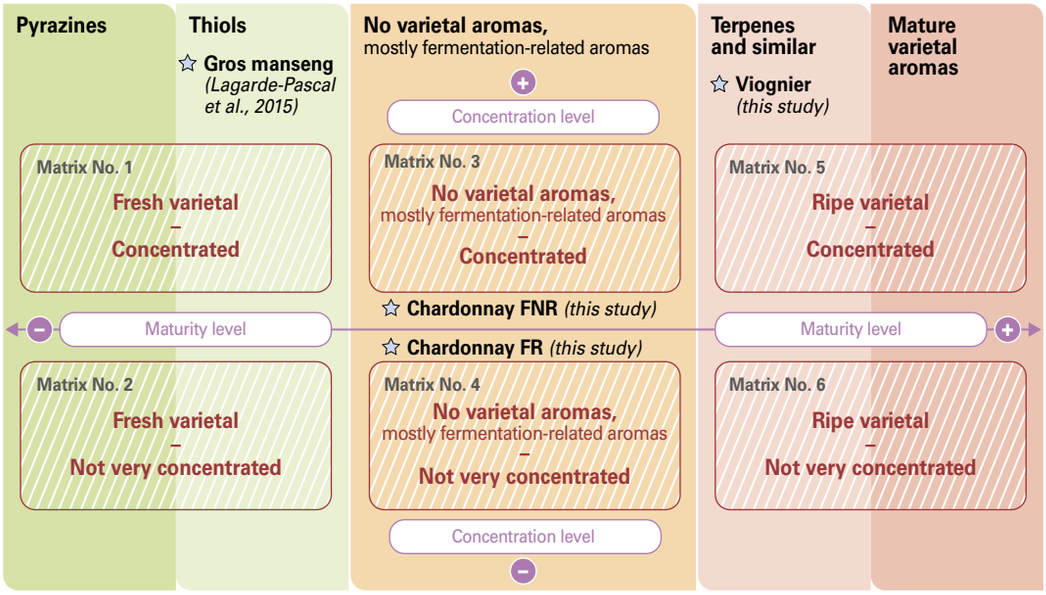
TECHNICAL

The choice of stopper has a great impact on the proper storage and ageing of wines in bottle. In addition to ensuring perfect sealing for liquids as well as maintaining organoleptic neutrality, the stopper must also enable perfect control over permeability to oxygen. In fact, Godden et al. (2001) demonstrated the essential role played by the stopper in the manifestation of premature oxidative ageing of wines, even though this phenomenon is above all determined by the quality of the raw material and by the care with which the winemaker treats it *(Lavigne et al., 2008; Pons et al., 2010).* These days, it is well-known that micro-agglomerated cork stoppers may be defined by two essential values in terms of oxygen additions: OIR (Oxygen Initial Release), which represents the oxygen released by the stopper, and OTR (Oxygen Transfer Rate), which represents the oxygen coming from the outside via the stopper *(Chevalier et al., 2019).* A series of articles published in the past few years have demonstrated, thanks to sensory analysis and specific marker analysis, the significant influence of the stopper on the evolution of aromas in various white and red wines *(Lagarde-Pascal et al., 2015; Pons et al., 2019 a, b).* It has been shown that the choice of stoppers with controlled oxygen transfer is a critical action in winemaking as well as a precious tool for the winemaker or oenologist. This choice must be examined as a function of the wine being bottled but also of the desired wine profile when released on the market on a specific date. Vivelys has defined a matrix field, which classifies wines according to their level of maturity and their overall concentration level, as well as their reductive or non-reductive potential ***(Figure 1).*** Each of these matrices has different needs, especially with regards to the oxygen transferred *via* the stopper during wine ageing.

■ **Figure 1: Vivelys wine matrix.**



In 2015, an initial study was carried out on a matrix rich in varietal thiols: Gros Manseng *(Lagarde-Pascal et al., 2015).* Our project is intended to study the aroma and analytical evolution of three white wines resulting from different matrices over three years, as a function of stoppers with different permeabilities.

Equipment and methods

The purpose of this project is to define the oxygen needs and evolution of three white wines (2015 vintage) corked with stoppers that have various oxygen permeability levels. To do so, four stoppers with distinct OIR and OTR levels were selected ***(Table 1).***

The wines studied during this project come from various matrices: a Chardonnay made with reductive fermentation (RF), a Chardonnay made with non-reductive fermentation (NRF) and oak ageing, and a terpenic Viognier (non-reductive varietal). All bottles were stored in a cold room regulated at 14°C.

The evolution of wines was monitored for three years from both analytical and sensory points of view. To do so, oxidative evolution markers of wines were monitored, such as free SO_2 content

■ **Table 1: OIR and OTR values obtained on dry corks.**

	D5 P0,35	D5 P0,15	D30 Origine	D30
OIR (mg)	1.6	1.3	1.1	0.8
OTR (mg/year)	0.6	0.4	0.3	0.3

and dissolved oxygen (by PreSens Pst 3 spot). Moreover, certain aroma compounds associated with the oxidative ageing of white wines were measured *(Bueno et al., 2016; Silva Ferreira et al., 2003):* methional (odour of boiled potatoes), phenylacetaldehyde (withered rose, honey) and TDN (1,1,6-trimethyl-1,2-dihydronaphthalene, odour of petrol); as well as compounds responsible for the fruitiness of wines. Each measurement corresponds to the analysis of one bottle, except for dissolved oxygen measurements, which correspond to the average of five bottles. The wines were also tasted by a panel of expert tasters after the three-year study period.

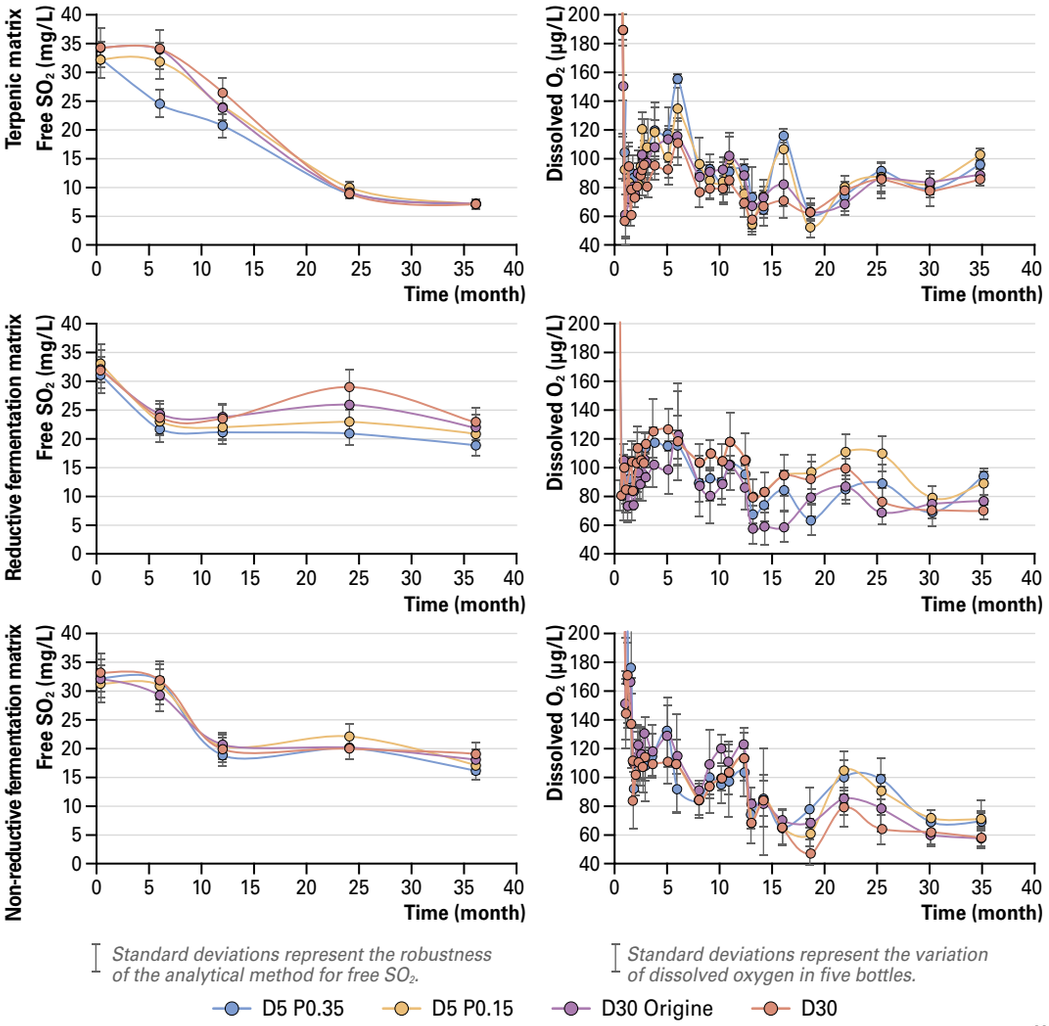
At the end of the three years of storage, we wanted to assess the impact of temperature variations on the transfer of oxygen through stoppers. To do so, we subjected the bottles of the terpenic Viognier matrix to temperature cycles (24 hours at 14°C followed by 24 hours at 28°C) and monitored the dissolved oxygen content in the wines.

Evolution of the free SO_2 and dissolved oxygen contents as a function of stopper type and wine matrix

Free SO_2 contents show different behaviour depending on the wine matrix in question as well as on stopper type ***(Figure 2).*** In fact, for the terpenic matrix, the most permeable stopper shows a faster decrease in free SO_2 content than the three others. However, after two years of bottle storage, there is no longer any free SO_2 (< 7 mg/l) in any of the wines. For the reductive and non-reductive fermentation matrices, the evolution of free SO_2 is similar for the different stoppers in the first few months of storage. The first deviations appear after two years for the RF matrix and after three years for the NRF matrix: in general, free SO_2 content is lower when the OTR and OIR values of the stopper are high ***(Figure 2).*** Nevertheless, all wine/stopper pairings for these two matrices present free SO_2 contents that are still very high (16 to 23 mg/L). The wines thus remain protected by the anti-oxidant action of free SO_2.

These results should be compared with the dissolved oxygen measurements present in the wines ***(Figure 2).*** The oxygen added during bottling is very rapidly consumed in all pairings (after about one month and a half), and dissolved oxygen levels tend to stabilise between 50 and 130 µg/l. For the terpenic matrix, a good correlation is observed between the OIR values of stoppers and the dissolved oxygen in the wine after six months of storage, thus confirming the data of Chevalier et al. (2019). With this same matrix, a trend towards increased dissolved oxygen content is observed starting in the second year of storage, in parallel with the disappearance of free SO_2 in the wines. After three years of ageing, the dissolved oxygen content seems to be higher for the most permeable stoppers with the RF matrix, but this trend is more difficult to observe for the NRF matrix. It is probable that these deviations between stoppers will increase as the free SO_2 content in the wines decreases as ageing continues.

■ **Figure 2: Evolution of free SO_2 and dissolved oxygen contents during three years of storage.**



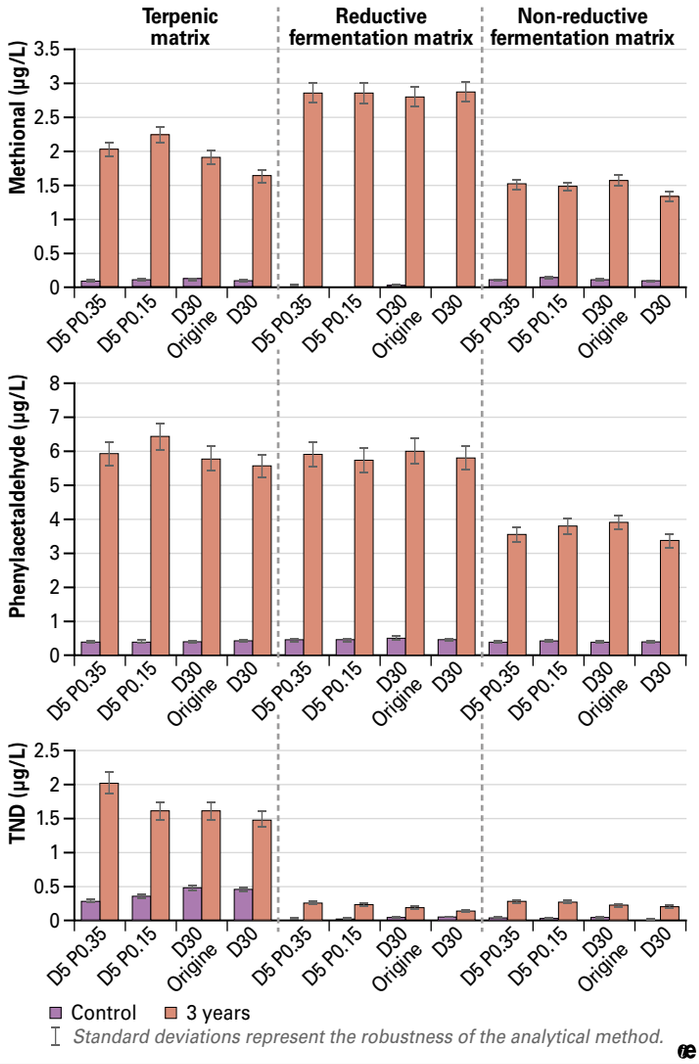
Evolution of oxidative ageing markers in white wines

To complete our compositional analysis for these wines, we monitored the evolution of several aroma compounds that are ageing markers in wines ***(Figure 3).*** Methional and phenylacetaldehyde are good markers of the oxidative evolution of wines. Their perception thresholds are 2.4 µg/l and 25 µg/l, respectively. Just after bottling, these compounds were found in much lower concentrations than these thresholds (< 0.2 µg/l for methional and ≤ 0.5 µg/l for phenylacetaldehyde) for all of the wines in question ***(Figure 3).*** After three years of storage, methional is measured at higher concentrations than this threshold, thus reflecting the oxidative ageing of the wines ***(Figure 3).*** Phenylacetaldehyde concentrations significantly increase

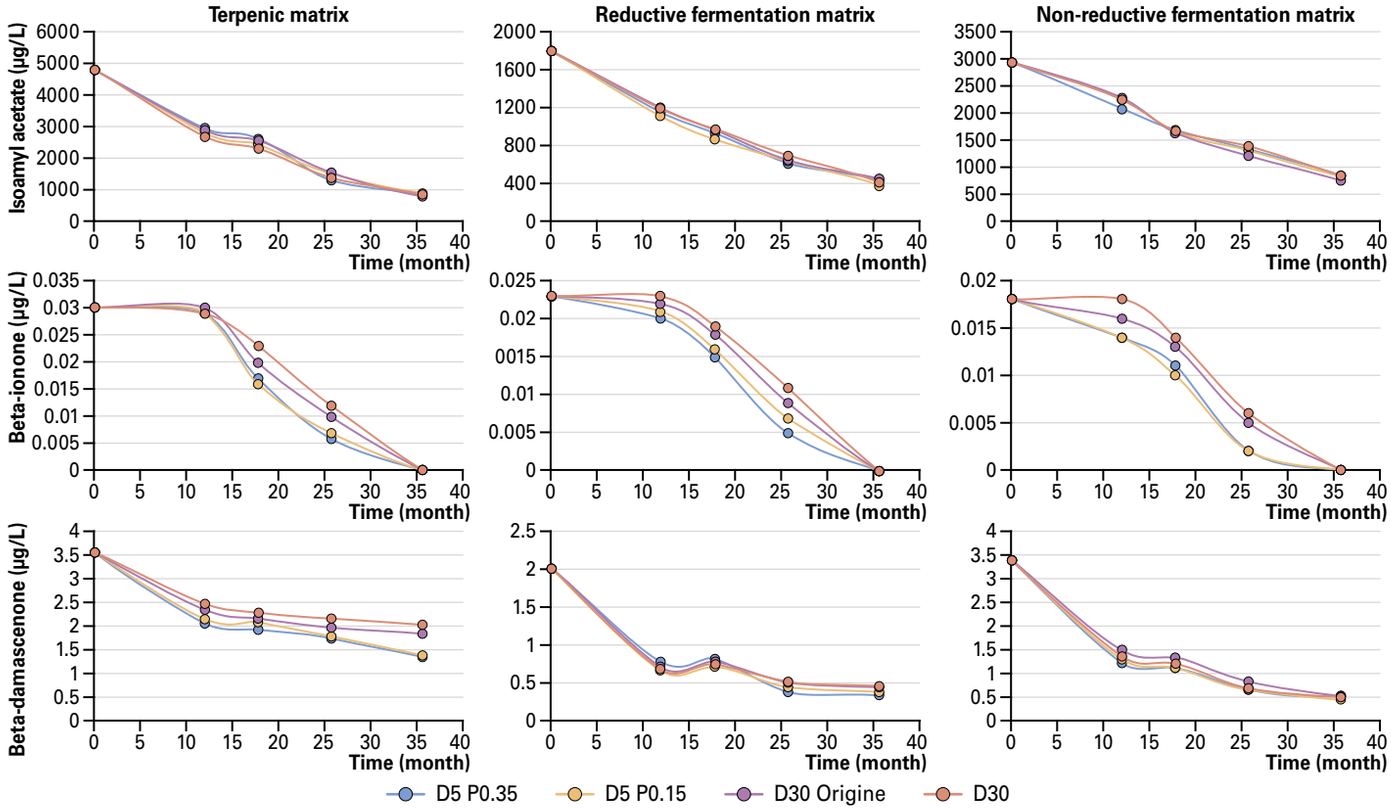
(by a factor of 8 to 12) during storage but do not exceed the perception threshold. However, it remains difficult to establish a link between the concentration of these compounds and the permeability of stoppers, except for the terpenic matrix, where a trend seems to appear: the more permeable the stoppers are, the higher the concentrations. This seems consistent with the fact that there is no longer any detectable free SO_2 in this wine resulting from the terpenic matrix, which is therefore more sensitive to oxidation.

TDN has also been described as an ageing aroma *(Silva Ferreira et al., 2003).* Its perception threshold is around 2 µg/L. During bottling, TDN concentrations are observed to be much lower than this threshold (< 0.05 for the RF and NRF matrices and < 0.5 for the terpenic matrix) ***(Figure 3).*** After three years of ageing, these concentrations remain low for RF and NRF matrices, even though an increasing trend is observed. Wines resulting from the terpenic matrix show concentrations near or above the perception threshold. For the three matrices, a correlation with the permeability of the stopper is observed: the higher OTR and OIR values of the stopper, the higher the TDN concentration is ***(Figure 3),*** which is the sign of a more extensive oxidative evolution for stoppers that let more oxygen enter the bottle. In addition to the appearance of these molecules related to the oxidative ageing of wines, a decrease in aroma compounds involved in the fruitiness of wines was observed, such as β-ionone, β-damascenone and acetate esters ***(Figure 4).*** The loss of fruitiness is often observed during the premature ageing of white wines. For the five compounds being monitored, a constant decrease over time was observed, whatever the stopper or matrix in question. It is often difficult to highlight differences according to the permeability of stoppers.

■ Figure 3: Methional, phenylacetaldehyde and TDN contents in wines after three years of storage.



■ Figure 4: Acetate ester and norisoprenoid contents in wines after three years of storage.



Impact of cork permeability on the perception of oxidative evolution during tasting

Throughout this work, a correlation between free SO_2, dissolved oxygen, the presence of oxidation markers and the oxygen permeability of stoppers was found. In addition to the analytical approach, we assessed the oxidative evolution of wines using sensory analysis. Tasters were asked to freely describe the wines, while paying specific attention to the oxidised character of those wines. Their comments are listed in ***Table 2.***

These results tend to confirm that the choice of stopper will greatly depend on the wine matrix. In fact, it is observed that for a terpenic matrix, despite a very slight reduction, D30 was preferred over the other corks. Moreover, it appears that the use of this same type of stopper with a non-reductive fermentation matrix, where oak was used, prevented oxidative evolution and preserved the vanilla character after three years of storage. Finally, more permeable stoppers seem to be the best suited to three years of bottle ageing for wines resulting from the reductive fermentation matrix.

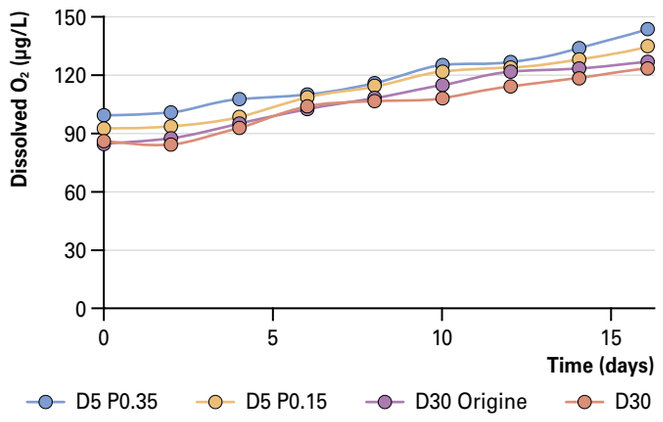
Impact of wine storage temperature on oxygen transfer

In this study, bottles were stored under «ideal» conditions in a temperature- (14°C + /- 1°C) and humidity-regulated cold room, protected from light. To get as close as possible to real conditions encountered by a wine from bottling until its sale to the consumer, we decided to subject the bottles to cyclical temperature variations: 24 hours at 14°C followed by 24 hours at 28°C.

■ Table 2: Descriptors most often cited during sensory analysis after three years at 14°C.

	Terpenic matrix	Reductive fermentation matrix	Non-reductive fermentation matrix
D5 P0.35	Fresh Open Not very aromatically intense	Slightly oxidative	Fresh Slightly oxidative Not very oaky
D5 P0.15	Fresh Open Not very aromatically intense	Open Discreet on the nose	Fresh Slightly oxidative Not very oaky
D30 Origine	More closed Dried fruit notes	Slight reduction	Open Slightly oaky
D30	Very slight reduction Peach and apricot notes More aromatically intense	Reduced	Oaky Vanilla More aromatically intense

■ Figure 5: Evolution of dissolved oxygen in a Viognier wine (terpenic matrix) when subjected to temperature variations.



Dissolved oxygen in the wines was measured at regular intervals ***(Figure 5).*** We observed an increase in dissolved oxygen over time for the four stoppers. After 15 days of cycles, bottles corked with the most permeable stoppers presented the highest dissolved oxygen rates. This absolutely confirms the effect of day/night cycles on bottle storage. Therefore, it will be of interest during future studies to use this cycling method in order to maximise the differences between stoppers.

Conclusion

Our study is part of a project initiated in 2008, which is aimed at increasing knowledge on the impact of the stopper's oxygen permeability on wine quality during bottle ageing. To do so, an approach combining analytical data and sensory analysis was implemented. Therefore, on three white wines coming from different matrices, we assessed the impact of the stopper on classic markers of oxidation (dissolved O_2, free SO_2), but also on aroma markers for oxidative ageing (TDN, methional, phenylacetaldehyde).

Finally, our work shows the importance of controlling the oxygen supply throughout bottle storage and confirms the importance of the stopper/wine matrix pairing. We have demonstrated that the choice of a low-permeability stopper is required for a terpenic matrix in order to conserve the varietal aroma of such wines and minimise the compounds related to oxidation (TDN, methional, etc.). However, for a reductive fermentation matrix, a more permeable stopper will prevent the appearance of reduction notes and conserve the freshness of the wine. ■

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