Aging of a white wine of the "varietal thiol" type as a function of different oxygen contributions *via* cork closures and by micro-oxygenation

Christine Lagarde-Pascal[1], Vincent Lieubeau[1], Christophe Loisel[2], Dominique Rabiot[2]
[1] *Vivelys SAS – Villeneuve-Lès-Maguelone – France.*
[2] *Diam bouchage – Espace Tech Ulrich – Céret – France.*

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During the past 15 years, several studies on the effect of wine closures have been published. They aimed to compare the evolution of analytical criteria over time in wines *(Lopes et al., 2009; He et al., 2013; Dimkou et al., 2011; Brajkovich et al., 2001; Wirth et al., 2010)*, organoleptic profiles of wine *(Godden et al, 2001; Skouroumounis et al., 2005; Lopes et al, 2009; Caille et al., 2010)* or even to develop techniques to measure closure permeability *(Sanchez et al., 1998; Lopes et al., 2005; Skouroumounis et al., 2007; Dieval et al., 2009)*. The measurement of permeability enables us to compare the closures with each other, but under no circumstances does it provide information about the effect of the closure on the wine's profile over time, which is indeed the most important piece of information for the winemaker. To our knowledge, no study has compared the evolution of wine profiles due to the permeability of a closure during a long period of aging in bottle and the evolution of the same wines when subjected to low doses of micro-oxygenation in sealed containers. Micro-oxygenation is a widely used technique today, and its effects on wines are well-known. This parallel between bottle aging and micro-oxygenation could help winemakers predict the aging profile of their wines with different types of closures, in order to optimize their choice of closure, thus enabling them to target a specific wine profile at a given release date.

The goal of this study is to establish a correlation between micro-oxygenation flow rates and oxygen permeability of closures, as measured by pressure gauge methods *(Sanchez et al., 1998)* and Mocon Ox-Tran®, and also to monitor how the profile of this white wine of the "varietal thiol" type evolves during its aging in bottle. A wine with a concentrated thiol profile (citrus, boxwood) was bottled using several different closures, and it was also micro-oxygenated with several oxygen flow rates. An analytical and organoleptic monitoring of the wines was conducted for 3 years. A comparison between micro-oxygenation dose and oxygen permeability of closures will be presented here, and the effect of these different oxygen contributions on the profile of wines will be discussed.

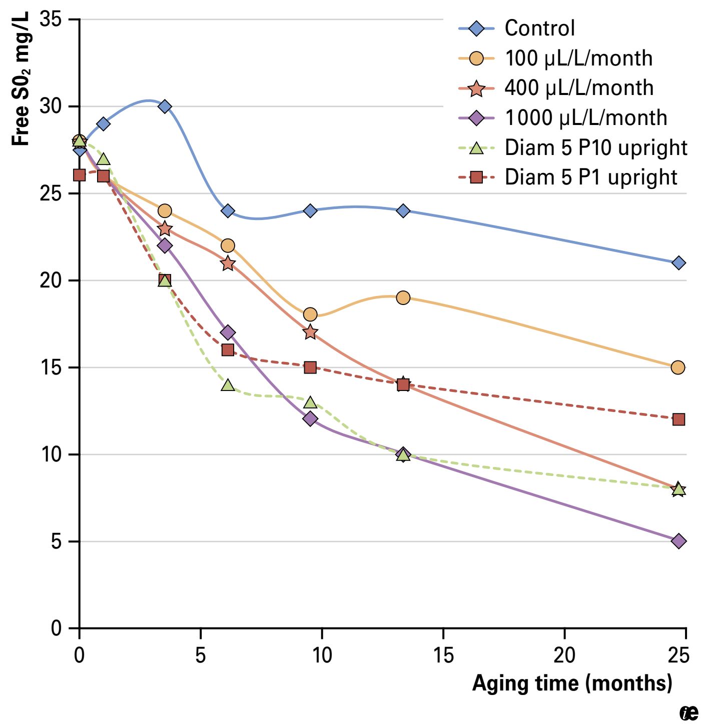
Materials and Method

A Gros Manseng wine from Jurançon (2011) was chosen for its high concentration of thiols (6,600 ng/l of 3-mercaptohexanol and 620 ng/l of 3-mercaptohexanol acetate).

To minimize oxygen uptake during bottling, the wine was pushed with nitrogen into the bottling tank. The bottles were first filled with nitrogen gas and then vacuum corked (Durfo corker) using Diam 5 closures with 2 different permeability values (P1 and P10). The total oxygen (dissolved and gaseous) in the 5 bottles stored upright and 5 bottles stored lying down with each of the closures was monitored throughout the non-invasive measurement test (Fibox 3 LCD trace, Presens). This same wine was put into 6 sealed stainless steel containers, blanketed with nitrogen and then subjected to micro-oxygenation flow rates (Vivelys) at 0, 20, 100, 400, 700 and 1,000 µL/L/month. The samples of wine were taken during the test under nitrogen blanketing, in order to replace the volume of liquid sampled with this inert gas. The concentrations of free and total SO_2 were monitored by iodometric titration (Œno 20, Œnobio). The concentrations of 3-mercaptohexanol (3MH) and 3-mercaptohexanol acetate (3MHA) were analyzed by the Nyseos laboratory at the start of the test, and then after 1, 6, 12, 24 and 36 months.

A sensory evaluation of the wines was performed initially and then again after 1, 3, 6, 9, 12, 24 and 36 months by a panel composed of 8 trained persons, using the Syriel tool (Vivelys). Though a misnomer, the term "permeability" of closures will be used in this article in place of the word "flow rate" (transfer of oxygen per unit time), as is usually done in the profession.

Figure 1: Evolution of free SO_2 (mg/L) in bottles of Gros Manseng with P1 and P10 closures, kept upright and in stainless steel tanks, both as a control and micro-oxygenated at 100, 400 and 1000 µL/L/month.



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Results and discussion

Evolution of free SO_2 and total oxygen in bottle

The monitoring of free ***(figure 1)*** and total SO_2 shows a faster drop in oxygen in the bottles and in the stainless steel containers during the first 6 months of aging. In parallel, total oxygen (dissolved + gaseous) in the bottles stored lying down ***(figure 2)*** increases during the first few days after bottling. This increase is greater in the bottles with P10 closures than in those with P1 closures (which have lower permeability). These phenomena can be explained by a transfer of the oxygen contained in the pores of the closures into the bottle, as described by Ugliano et *al.* (2011): the more permeable closures have more interstitial zones and release more oxygen than the less permeable closures.

The monitoring of dissolved oxygen ***(figure 2)*** in the bottles kept upright does not show any increase in total oxygen during the first days after bottling. In this case, the wine/gas interface through which oxygen gas can be dissolved is smaller than in the case of bottles kept lying down, and its speed of dissolution is lower than the rate of oxygen consumption by the wine.

Correlation between micro-oxygenation flow rate and the permeability of closures

As expected, the drop in thiol concentration during the 3 years of aging is greater when the amount of added oxygen is high. The analyses performed after two years on wines from the stainless steel containers show a correlation between 3MH concentrations and the flow rate of oxygen added via micro-oxygenation ***(figure 3)***.

Based on this correlation and relative concentrations of 3MH measured in the bottles with the P1 and P10 closures (average of one measurement on one bottle stored upright and one bottle stored lying down). We can calculate an equivalent flow rate between micro-oxygenation and the transfer of oxygen via the closure. This extrapolation leads to an oxygen flow rate of about 40 μL/L/month (57 μg/L/month) *via* the P1 closures. The permeability measurement via the Mocon Ox-Tran® method gives a value of 0.0008 cm³/J, i.e. 32 μL/L/month. The P10 closures have a permeability of 0.0019 cm³/J, i.e. 76 μL/L/month measured by this same method, versus 160 μL/L/month (217 μg/L/month) according to the present extrapolation. The permeability values measured by the two methods are comparable even if the deviation is more significant for the P10 closure, likely owing to the precision of the extrapolation.

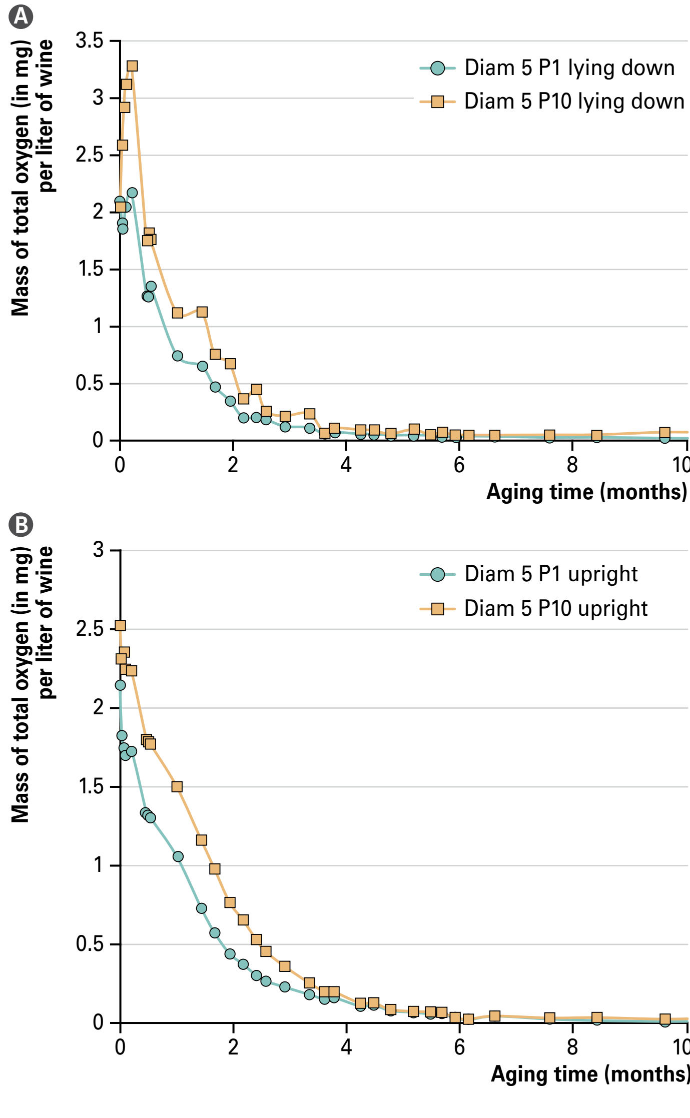
Owing to its different design, the values obtained with the pressure gauge method are clearly greater than the previous values (0.15 cm³/J, i.e. 6000 μL/L/month for P1, and 0.35 cm³/J, i.e. 14,000 μL/L/month for P10), since the transfers are accelerated by the pressure differences between the two ends of the closure during analysis.

Nevertheless, one can observe that the ratios between the permeability vales of closures P1 and P10 are maintained and are between 2 and 4, depending on the method used. This new comparative approach between the oxygen permeability of the closures and aging with micro-oxygenation enables us to foresee the changes that wines undergo in the bottle, by comparison with the effects of micro-oxygenation aging, which today are much better known to winemakers.

Impact of added oxygen on the sensory evolution of wine

The evaluation of the organoleptic profiles of the different wine/closure pairs has enabled us to observe that none of them show any reduction up through 9 months of bottle age.

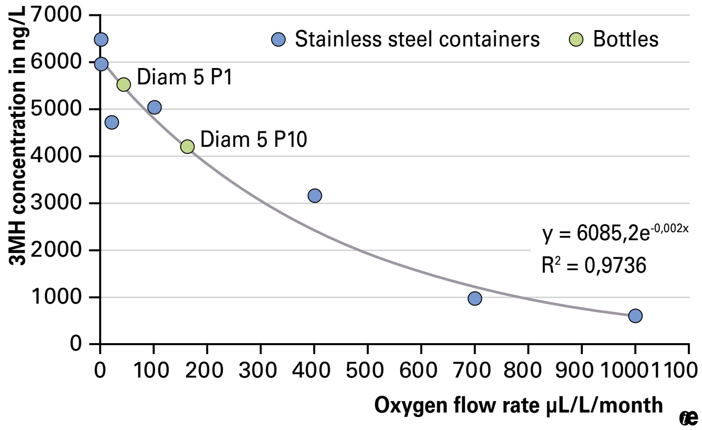
Figure 2: Evolution of average total oxygen (gaseous O_2 + dissolved O_2) (out of 5 bottles) in bottles capped with P1 and P10 closures, kept upright Ⓑ and lying down Ⓐ. Expressed in mg of total O_2 per liter of wine.



After 12 months, notes of reduction were described in the wines bottled with the 2 types of closures, as well as in the wine from the least oxygenated stainless steel containers (less than 100 μL/L/month). With these same pairs, "fire-related/burnt" notes were described for the first time. These notes could be related to the formation of aging-related thiols, such as benzene methanethiol (smoky, flint), 2-methyl-3-furanthiol (cooked meat) and furfurylthiol (roasted coffee) *(Ribéreau-Gayon et al., 2012)*, but these compounds were not tested. However, none of the test wines were deemed to be oxidized, even the one micro-oxygenated at the highest flow rate of 1000 μL/L/month.

After 24 months of aging, the fire-related/burnt notes were again described on the least oxygenated test wines (bottles and micro-oxygenated wines at less than 100 μL/L/month), with the complete absence of notes of reduction this time. However, the wines that were micro-oxygenated at 700 and 1000 μL/L/month were found to be oxidized. After 36 months of aging ***(figure 4)***, very significant differences in profiles were observed. The wines that were micro-oxygenated at 700 and 1000 μL/L/month had highly oxidative profiles. In contrast, the control wine, which did not receive any added oxygen, and the bottle capped with P1 closures and stored lying down both still

■ Figure 3: Correlation between concentrations of 3MH quantified after 2 years of aging, and flow rate of oxygen added via micro-oxygenation. Flow rate equivalent to the oxygen added via the P1 and P10 closures calculated from their 3MH concentration and from this correlation.



had notes of varietal thiols and fire-related/burnt notes from aging, without any reduction. The other wines (P10 closures stored upright and lying down, P1 closure stored upright, and the wines micro-oxygenated at 20, 100 and 400 µL/L/month) were described as having the greatest aroma complexity, with varietal thiol notes (grapefruit, boxwood), signs of reduction/bottle age (fire-related/burnt) and candied citrus notes. The latter appeared for the first time during bottle aging. The 400 µL/L/month and P10 upright wines still have less varietal thiol character and more oxidative notes. The evolution of this wine with a "varietal thiol" profile during this aging test demonstrates that reduction can be a passing defect which leads to the formation of more complex wine profiles, in particular with notes of reduction/bottle age. This aroma complexity was not observed when the wines were not subjected to a reduction phase.

Conclusion

This study clearly shows that the choice of a closure, and in particular its permeability to oxygen, is a winemaking decision, much like the last step of the winemaking journey, described elsewhere for thiol wines *(Lagarde-Pascal et al., 2013)*. This choice must be based on the type of wine being bottled, but also on the desired wine profile when it is released on the market on a specific date. For example, in the case of this wine with a "concentrated varietal thiol" profile, the following strategies can be envisaged:

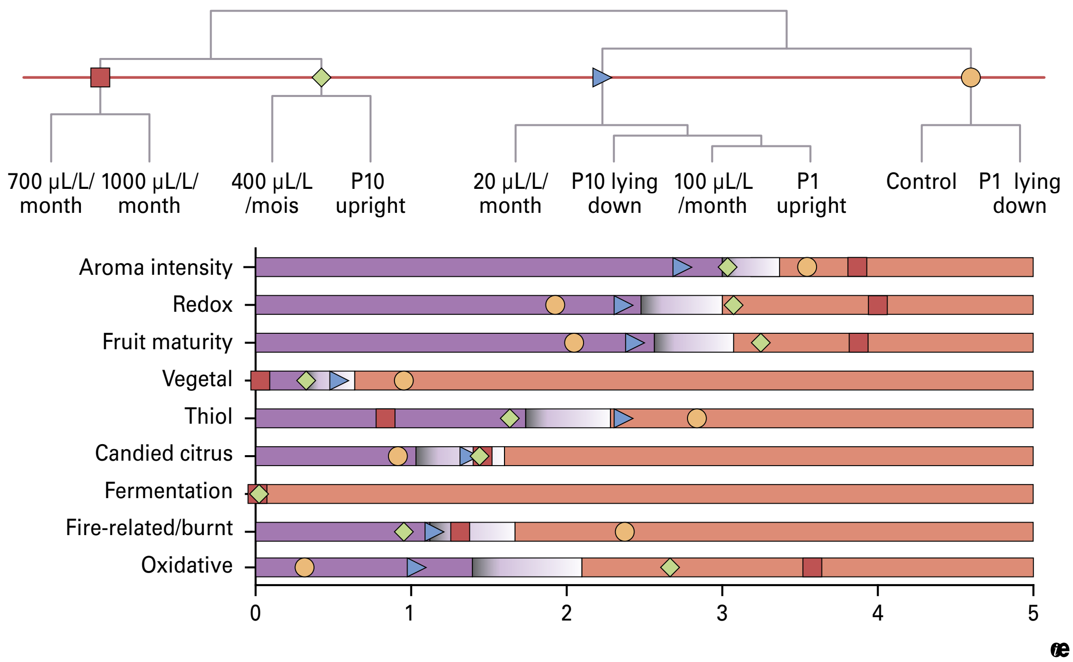
– bottling with a closure that is very permeable to oxygen (close to 700 µL/L/month) would help avoid any appearance of reduction, but with a deterioration of the "concentrated varietal thiol" aroma profile towards an oxidative profile, for aging periods longer than 24 months,

– bottling with a closure that is less permeable to oxygen (less than 400 µL/L/month, as is the case with P10) would help, after a reduction phase, to obtain more complex wines with a reduced bouquet after 24 months of bottle aging. After 36 months, these wines would have a high level of complexity, combining varietal notes and reduced/bottle age notes with a hint of oxidation,

– bottling with closures that have very low permeability to oxygen (case of P1 and the control) helps to preserve aroma freshness for more than 36 months (duration of this study), with co-existence of varietal and reduced/bottle age notes. ■

Note: The bibliographical references concerning this article are available on request from the Revue des Œnologues.
- By mail: enclose a stamped envelope, with the references of the article
*- On the Internet : **www.oeno.tm.fr***

■ Figure 4: Increasing hierarchical ranking of the wine/closure pairs after 36 months of aging, calculated from the tasting notes by the 8-person panel, and aroma profile of each of the groups (represented by a symbol on the ranking). Color bars for each descriptor: ANOVA at 5% with respect to the average of the samples.



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