IVES Technical Reviews
vine & wine

https://doi.org/10.20870/IVES-TR.2024.7952

The bottleneck/cork interface: a key parameter in bottle-aging of wine

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A study of changes in oxygen transfer through micro-agglomerated corks was carried out over 24 months under controlled storage conditions[1]. This work demonstrated that the glass/cork interface can represent a major entry route for oxygen into wine bottles. It also showed that there is no difference in oxygen transfer at 20 °C between bottles stored horizontally and vertically. However, high storage temperature greatly increases oxygen transfer at the interface between the cork and the bottleneck.

Background to the study

The shelf life of wines is a major concern in the wine sector. This is particularly true of wines intended for long cellaring, which are supposed to reach their peak after bottle-aging ranging from a few months to several years or even decades. Low levels of controlled oxygenation through the closure system are generally necessary for the wine to develop its optimum organoleptic characteristics. Bottle-aging is also affected by various environmental storage parameters, such as temperature, humidity and exposure to light[2] (Figure 1a). Previous studies have shown that the interface between the cork and the bottleneck plays a crucial role in the transfer of oxygen into the bottled wine[3, 4]. But how does oxygen transfer through the closure system change over time? How is it affected by storage conditions?

Do the oxygen barrier properties of the closure system change during storage?

For this study, a system of miniature bottles was designed to reduce analysis times (Figure 1b). Surface-treated micro-agglomerated corks were compressed into bottlenecks cut 70 mm from the top. After corking, only a 6 mm thickness of cork was retained in the bottleneck. To assess the effect of the presence of wine on the oxygen barrier properties of the cork, samples were stored with or without 10 mL of a model wine solution and closed under an inert atmosphere (argon) by gluing a glass disk to the base of the bottleneck. The samples were positioned either in contact with the vapor phase of the model wine or directly in contact with the liquid phase, thus reproducing vertical or horizontal storage of bottles. For samples stored horizontally, three temperatures were studied: 20 °C, 35 °C and 50 °C. Oxygen permeation measurements were carried out by manometry, in two successive stages: (i) on the complete system including the cork disk compressed in the bottleneck, (ii) on the disk alone, making it possible to determine the transfer at the interface by subtraction (Figure 1c). The manometric measurement instrument and the protocol used to measure oxygen transfer have been described in detail in previous papers[5, 6]. The oxygen diffusion coefficient through the cork disk D_{disk} and the oxygen diffusion coefficient through bottleneck/cork system D_{total} were determined. Samples were monitored at 3, 6, 9, 12, 18 and 24 months, analyzing a minimum of 4 samples for each storage condition and analysis time, i.e. an experimental design involving more than 150 samples.

The results obtained show that, whatever the storage conditions, the D_{disk} values remain similar over the entire 24-month period studied, with a mean value of 1.3×10^{-11} m^2·s^{-1}. The small variations observed are due to the intrinsic variability of the material rather than an aging effect. Hence, the presence of model wine, the storage position and the temperature have no significant effect on the oxygen diffusion coefficient. This first result highlights the remarkable stability of the intrinsic barrier properties of the corks over 24 months, under these experimental conditions.

In contrast, total oxygen transfer (D_{total}), including not only oxygen transfer through the cork, but also oxygen transfer at the glass/cork

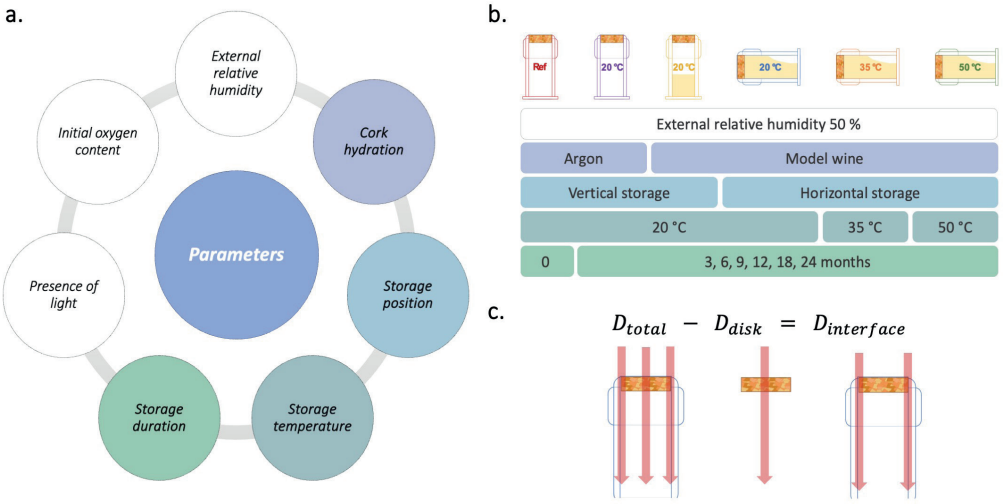


FIGURE 1. a) Main parameters influencing bottle-aging of wine, b) Summary of the different conditions studied, represented by the five experimental conditions and the initial reference, c) Diagram of the different types of oxygen transfer through the complete closure system and the stopper (6 mm thick cork disk), allowing calculation of the transfer at the interface.

The translation of this article into English was offered to you by Moët Hennessy.

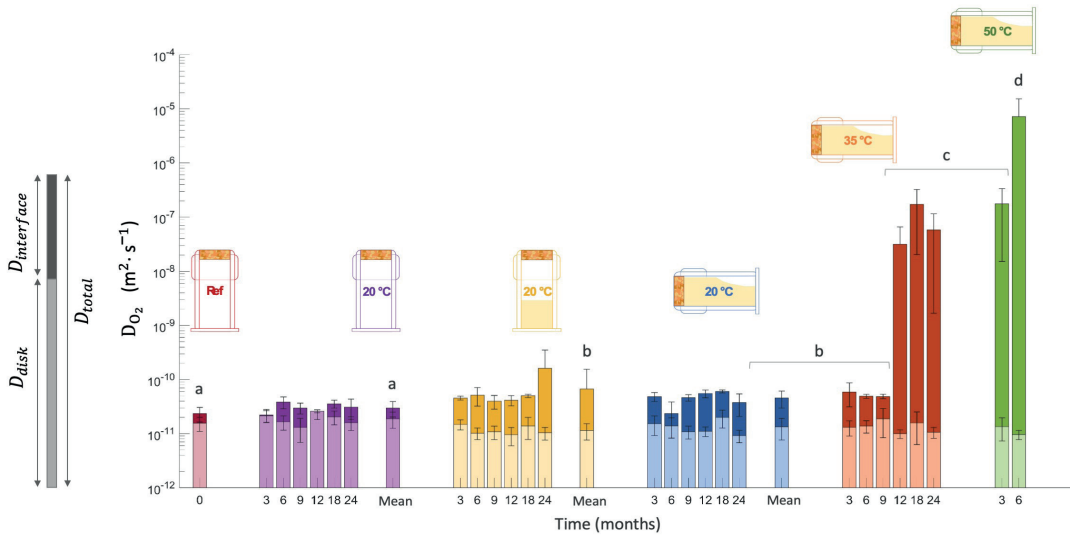


FIGURE 2. Change in the oxygen diffusion coefficient through the micro-agglomerated cork disk (D_{disk}), through the compressed disk in the bottleneck (D_{total}) and at the glass/cork interface ($D_{interface}$), over 24 months under different storage conditions.

The experimental measurements were performed on 6 mm thick cork disks, first inserted into a bottleneck, then on their own. Red: reference without model wine. Purple: storage at 20 °C without model wine. Yellow: vertical storage at 20 °C with model wine. Blue: horizontal storage at 20 °C with model wine. Orange: horizontal storage at 35 °C with model wine. Green: horizontal storage at 50 °C with model wine. The results are presented as the sum of the diffusion coefficient through the disk alone (D_{disk}, light color) and the diffusion coefficient at the interface ($D_{interface}$, dark color), thus representing the total diffusion coefficient of the complete closure system D_{total}. Statistically significant differences between the mean values calculated for each experimental condition are indicated by different letters (a, b).

interface, was modified by the presence of model wine, with an increase in D_{total} from 2.3×10^{-11} $m^2 \cdot s^{-1}$ to 4.7×10^{-11} $m^2 \cdot s^{-1}$, after the first three months of storage at 20 °C (Figure 2, yellow bars). This effect could be due to the sorption of water and ethanol by the cork, resulting in a plasticizing effect[7] [8]. Cork hydration also affects the mechanical properties of closures, with a significant reduction in the elastic modulus to half its initial value at between 50 % and 100 % relative humidity[9]. It can thus be assumed that the force applied by the stopper on the glass wall of the bottleneck tends to decrease during the initial hydration phase of the material, leading to an increase in oxygen transfer at the interface. Furthermore, the horizontal or vertical storage position did not affect oxygen transfer (Figure 2, yellow and blue bars). The oxygen diffusion coefficients D_{total} for the two different storage positions remain similar. These observations are valid for all durations studied, from 3 to 24 months. Once the sorption equilibrium of water and ethanol on the cork has been reached, a relatively stable diffusion coefficient is observed over the following months[7] [8]. Hence, this study clearly shows that, under the experimental conditions studied and in the presence of model wine, the position of wine bottles during 24 months' storage at 20 °C has no effect on oxygen transfer through the closure system. Lastly, temperature has a considerable effect on total oxygen transfer. At 20 °C, the oxygen diffusion coefficient D_{total} remains unchanged at 3 months and 24 months. At 35 °C, the oxygen diffusion coefficient D_{total} remains similar to that measured at 20 °C, but only for storage up to 9 months. Beyond this period, the total oxygen diffusion coefficient D_{total} increases by a factor of 1,000 after 12 months of storage (Figure 2, orange bars). Lastly, for samples stored at 50 °C, a 10,000-fold increase in transfer occurs at the interface as from 3 months, with the oxygen diffusion coefficient D_{total} reaching 1.8×10^{-7} $m^2 \cdot s^{-1}$. This phenomenon is further accentuated after 6 months, with a value of 7.2×10^{-6} $m^2 \cdot s^{-1}$, close to the diffusion coefficient of oxygen in air. At this temperature, the mechanical properties of the closure may be modified and also the stability of the surface treatment, with partial melting of the paraffin coating promoting oxygen transfer at the bottleneck/cork interface.

Conclusions

These results offer a novel perspective by demonstrating the influence of the glass/cork interface on the shelf life of bottled wines under controlled storage conditions. They provide concrete answers to questions such as the influence of the presence of a liquid, and the storage position and temperature of the bottle, which may be of interest to both wine producers and consumers concerned with conserving the quality of wines during bottle aging. To transpose these results from a model system to the storage of bottled wines by means of a global model describing oxygen transfer, various parameters still need to be integrated, such as the type of cork, its hydration state over a greater length, the product used for surface treatment, and the change in mechanical properties over time. ■

Acknowledgements: *The authors would like to thank Diam Bouchage (France) and the Association Nationale de la Recherche et de la Technologie (ANRT, France) for their financial support and Julie Chanut's PhD grant (CIFRE 2018-1278). They also thank GAI for the loan of an industrial corking machine, and the DIVVA platform for access to analytical equipment.*

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