



# Quantification and identification of oxygen transfer phenomena through barrels

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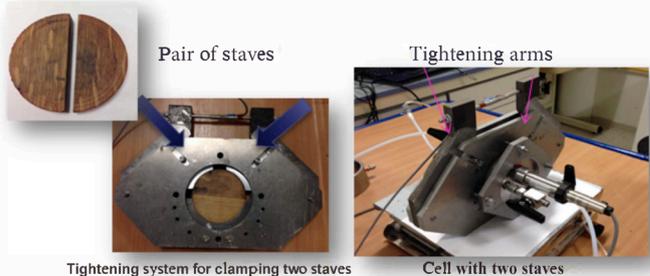
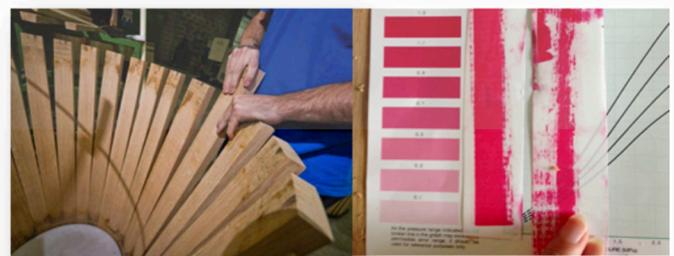
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Due to the intrinsic physical and chemical properties of oak, a barrel is the site of highly complex transfer phenomena. By developing an innovative gas-liquid permeameter, oxygen transfers on both sides of oak samples have been measured and a complete material balance for the barrel has been produced. This study demonstrates that oxygen transfer essentially takes place in low-pressure zones between staves and that oxygen desorption is the main pathway for total oxygen uptake.

The experiments in this study<sup>1</sup> have been performed according to the following material balance:

$O_2$  transferred through staves +  $O_2$  transferred between staves +  $O_2$  transferred at the bung-barrel interface +  $O_2$  desorbed from the oak =  $O_2$  consumed by compounds extracted from the oak +  $O_2$  consumed by the wine + residual  $O_2$ .

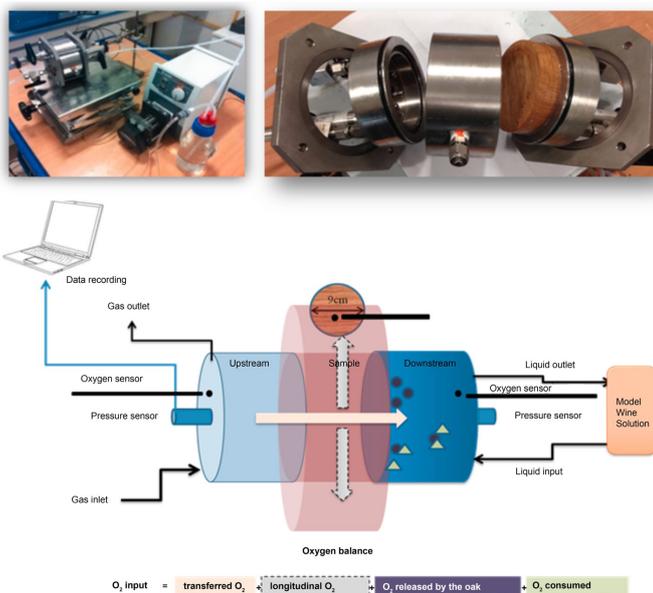
Many authors have already quantified total oxygen uptake and estimate it at between 20 and 27 mg/L/year, but no previous study has provided a complete material balance<sup>2,3</sup>. To do this, an innovative permeameter was developed to reproduce oxygen transfer through barrels at laboratory scale (Figure 1).



**FIGURE 2.** Methodology for studying the pressure between staves. This device was specially designed to measure the oxygen transfer rate through the gaps between staves using the permeameter. A selected piece of oak was cut in half, with the two halves clamped together in the tightening system.

according to the different shades of red on the pressure strips. Two half-staves and a calibrated tightening arm were used to apply the same pressures in the permeameter, to measure gas transfers (Figure 2).

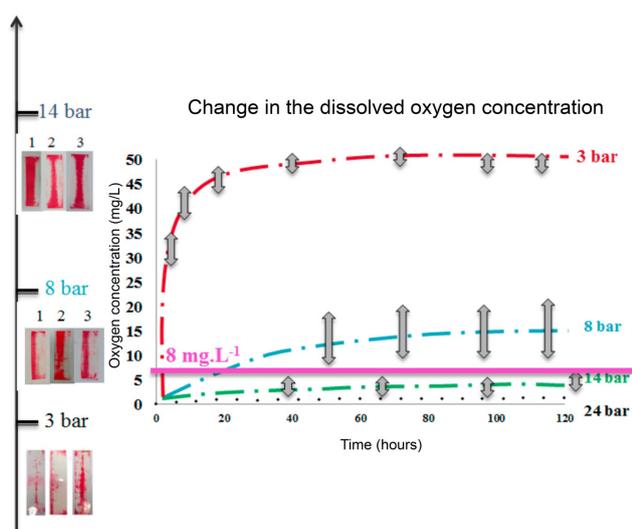
These measurements showed clear disparities. The pressure between staves on the barrel head can reach more than 20 bar while on the body of the barrel it ranges from 4 bar at the center of the stave to 14 bar at the ends. Based on this finding, these pressures were reproduced and transfers measured by the passage of pure oxygen in the course of different experiments (full staves and half-staves with application of a defined pressure between staves). Pure oxygen was used, to avoid interference from gas desorption. The oxygen saturation of wine in air is 8.4 mg/L (at 20°C) but 42 mg/L in pure oxygen, as the partial pressure is 5 times higher. Hence, when oxvaen



**FIGURE 1.** Experimental cell (photograph and schematic diagram) for quantification of gas transfer through wood. The samples were cut from oak staves (untoasted, fine-grained) supplied by the cooperage Taransaud. They were 9 cm in diameter and 2.3 cm thick, either whole or cut in half to replicate the joints between staves.

To reproduce operating conditions in the cell, it was necessary to determine the pressures between staves. Colorimetric pressure strips (Prescale) were used on a real barrel to assess the gradients existing within it. The forces exerted between staves were determined

concentrations exceed 8.4 mg/L, it is certain that pure oxygen is actually passing through the wood and that this transfer is not due to air desorption. Pressures of 3, 8, 14, and 24 bar were applied and the oxygen concentration was monitored over time (Figure 3).



**FIGURE 3.** Oxygen transfer between staves at different tightening pressures (3, 8, 14 and 24 bar). When working in the cellar, it is often observed that the weakest contact points in a barrel are around the bung hole and in the middle of the staves, where they are most strongly curved. A pressure of 3 bar can be observed at these points while pressures of 8, 14 and 20 bar are found along the length of a stave, with 20 bar at each end.

Permeameter measurements showed that a pressure of 24 bar applied between staves strongly limits the transfer of pure oxygen between the half-staves. Reducing the applied pressure to 14 bar and 8 bar facilitates gas transfer due to reduced contact between the staves: after 5 days under these conditions, the dissolved oxygen concentration can reach 10 mg/L. At an applied pressure of 3 bar between staves, the oxygen concentration can reach 40 mg/L in 5 hours. It should be noted that the leak-tightness of the barrel head is ensured through the use of high pressures (25 to 30 bar between staves). The pressure of 3 bar corresponds to the pressure between staves at the center of the barrel.

This study has also shown that stave-stave contact conditions (surface roughness) has an impact on transfer phenomena. Indeed, this parameter should be taken into account to improve contact conditions, to limit the preferential passage of oxygen.

It has also been found that a complete oak stave (which is between 23 and 27 mm thick) is impermeable to gas when in contact with liquid. Gas transfer does not therefore take place through the wood. This impermeability is due both to the structure of the wood and to its interconnections. The presence of tyloses observed on the ligneous plane<sup>4</sup> limits longitudinal transfer. The limiting connections are the perforations and pits, which are filled with liquid during aging. The oak is thus made impermeable, as diffusion phenomena are very slow across these interconnections.

Hence, the air permeability of the barrel depends mainly on the quality of contact between staves (surface roughness and pressure). It seems that the weak points of the barrel (where most oxygen transfer takes place) are found where there is low pressure (3 bar) between staves. These points are located at the middle of the staves. Ultimately, it would seem wise for coopers to modify hoop positioning to provide barrels with better regulated permeability (uniform pressure between staves across their length).

## Most oxygen transfer takes place by desorption of the gas contained in the oak

To quantify all the parameters in the equation, the oxygen desorption provided by the staves to the solution in contact was also determined. Staves were used in model solutions with an oak surface area/matrix ratio equivalent to that found in a barrel (225 L / 2 m<sup>2</sup>). The results show that the desorption of the gas contained in the oak represents a substantial part of the 20-27 mg.L<sup>-1</sup>.year<sup>-1</sup> provided by a barrel over a full one-year aging period (between 35 and 50 %). This phenomenon takes place concurrently with impregnation of the liquid in the oak during the first month. This impregnation is explained by the fact that the wine penetrates the vessel to a depth of up to 4 mm in a few days (rapid impregnation phenomenon) and reaches 7 mm after 1 month. A strong correlation has been observed between these impregnation kinetics and oxygen release by desorption. Hence, 10 mg/L is added to the wine during the first month, which represents a very significant quantity.

This study has also shown that gas transfer at the bung (silicone) represents only a few mg/L, which is very low compared with the desorption data and the total transfer. However, these values depend strongly on the type of bung and how it is used.

Overall, these results allow for better understanding of the factors that influence oxygen transfer during barrel aging, to offer better guidance to barrel users and to provide coopers with data to improve their manufacturing procedures. The management of dissolved gases is essential and requires careful attention whatever the level of winemaking.

## Further research needed

It would be useful to carry out additional studies including (i) the influence of alcohol content on oak impregnation kinetics and oxygen desorption from the oak and (ii) the influence of cleaning the barrel on oxygen desorption from the oak and the impregnation kinetics. ■

Sourced from the research article: "Oxygen desorption and oxygen transfer through oak staves and oak stave gaps: an innovative permeameter" (OENO One, 2018).

**1** QiuY., LacampagneS., MirabelM., Mietton-PeuchotM., & GhidossiR. (2018). Oxygen desorption and oxygen transfer through oak staves and oak stave gaps: an innovative permeameter. *OENO One*, 52(1). <https://doi.org/10.20870/oeno-one.2018.52.1.909>

**2** Giraud, W. (2009). Étude physico-chimique de l'interface bois-vin pendant l'élevage en barriques (Doctoral dissertation, Université de Toulouse, Université Toulouse III-Paul Sabatier).

**3** Nevares, I., & del Alamo-Sanza, M. (2015). Oak Stave Oxygen Permeation: A New Tool To Make Barrels with Different Wine Oxygenation Potentials. *Journal of agricultural and food chemistry*, 63(4), 1268-1275.

**4** Hansmann, C. H., Wimmer, W. G. R., & Teisinger, A. (2002). Permeability of wood-A review. *Drevarsky Lyskum*, 47(4), 1-16.