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Overcapping wax: an aesthetic or functional element

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An in-depth study was carried out to assess oxygen transfer through different waxes and resins, either alone or used in combination with cork-based stoppers. The research provided the answer to a crucial question being asked by many winemakers and consumers: does the application of overcapping wax play a protective role against oxidation of the wine during bottle ageing?[1]

Background to the study

The use of waxes and resins in oenology for corking goes back several thousand years, as several archaeological discoveries attest. This empirical use is mainly associated with the idea that they can limit the wine's exposure to oxygen. However, despite this historical use, the exact role of waxes and resins as oxygen barriers has rarely been studied, and even less so as overcapping materials. The only studies into the oxygen permeability of different types of wax (including beeswax, microcrystalline wax and paraffin) were carried out over two decades ago on wax films of relatively thin thickness for applications where wax is used as a food coating[2] or as a surface treatment agent for cork stoppers[3]. The aim of this study was to evaluate the oxygen barrier properties of different waxes and resins, either alone or in combination with cork-based stoppers. Oxygen transfer through corked bottles, with and without cork wax, was monitored over a one-year storage period.

Does the sealing wax act as an effective oxygen barrier?

A preliminary step in this study was to characterise the oxygen permeability of various waxes and resins (beeswax, shellac, microcrystalline wax and a commercial blend of waxes), in the form of thin films approximately 350 µm thick. The results show that not all waxes and resins offer an equivalent level of protection (Figure 1). Their effectiveness as oxygen barriers depends mainly on their intrinsic chemical composition and their physical state[4 5 6].

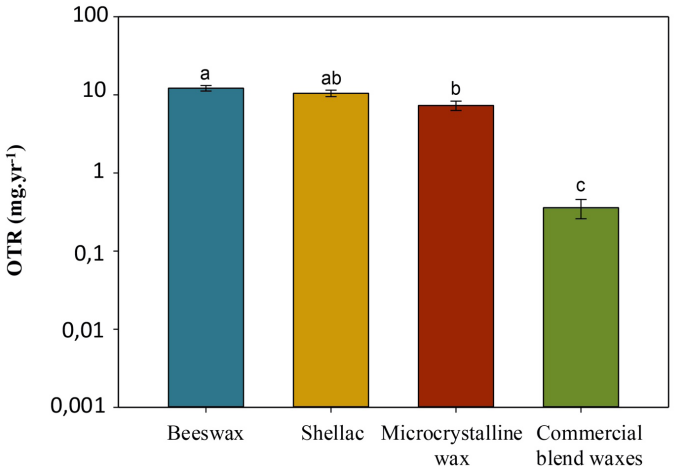


FIGURE 1. Oxygen barrier properties (in mg.yr^{-1}) of four waxes and resins, measured at 25°C and 50 % relative humidity. The OTR (Oxygen Transmission Rate) values were calculated for a film with a thickness of 350 µm and a diameter of 18.5 mm at an oxygen differential pressure of 212 hPa. The letters in the histogram indicate significant differences between the mean values ($p \leq 0.05$, $n = 3$).

Following this characterisation, the material with the highest oxygen barrier, in this case the wax blend, was selected to evaluate the performance of this wax as an overcapping system on clear glass bottles (37.5 cL capacity). A sensor was placed inside the bottles beforehand to enable the quantity of oxygen entering the bottle to be detected by chemiluminescence. The bottles were used empty to eliminate the influence of the wine and to focus solely on characterising the closure system (bottle + cork +/- wax). Before corking, the bottles were inerted with nitrogen to eliminate the oxygen initially present inside. They were then capped under vacuum using a semi-automatic Bottling machine (GAI 4040WL). The initial partial pressure of oxygen inside the bottles remained below 0.5 hPa. Two types of stoppers were used in the experiments: natural corks, 49 mm long and 24 mm in diameter, and micro-agglomerated corks, 44 mm long and 24 mm in diameter. Some of the corks were also used without surface treatment to simulate a leak at the glass/cork interface. The corks were inserted 3.5 mm below the neck of the bottle to allow sufficient space for subsequent wax deposition. Six replicates were prepared for each condition with micro-agglomerated corks and nine replicates for those with natural corks. Once the bottles had been corked, the wax was deposited, melting it at 120°C before it was deposited on the cork up to the neck of the bottle, which had been heated to 70°C beforehand. The quantity of wax deposited was approximately 1 g, corresponding to a thickness of approximately 3.5 mm once solidified. In the case of surface-treated corks, the wax was applied one month after corking to avoid deformation of the wax disc caused by outgassing of the cork following its compression in the bottleneck. In contrast, for corks without surface treatment (TDS), the wax was added immediately after corking to avoid immediate and excessive oxygen transfer at the glass/cork interface. The samples were stored under controlled conditions at 25°C and 50 % relative humidity for the duration of the study. The kinetics of oxygen transfer from the outside to the inside of the bottle were monitored for one year.

The results showed that the use of an overcapping wax leads to different results, depending on the initial oxygen barrier performance of the wax and that of the corks. On one hand, in the case of the micro-agglomerated corks used in this study, the barrier performance of the cork was already found to be very high, being of the same order of magnitude as that of the wax disc (Figure 2a). Consequently, the addition of wax to these stoppers did not confer any additional protection against oxygen (Figure 2b). On the other hand, the greater variability in oxygen permeability of natural cork stoppers (Figure 3a), widely described in the literature[7 8 9], does not allow the role of wax addition to be accurately assessed (Figure 3b). Finally, in the case of corks without surface treatment (Figure 2c and 3c, "without TDS"), whether natural or micro-agglomerated, very significant oxygen transfer at the glass/cork interface occurs rapidly and is equivalent to a leakage-type defect. In this case, the addition of wax can

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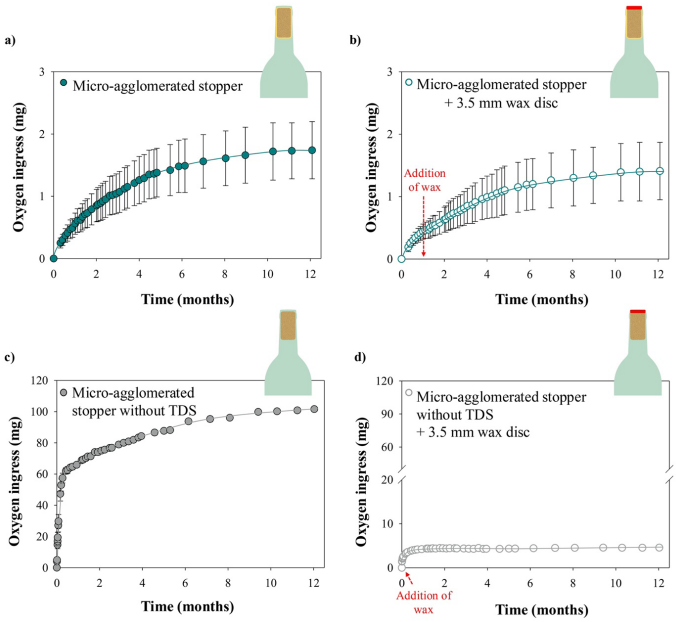


FIGURE 2. Quantity of oxygen entering bottles corked with micro-agglomerated corks (n = 6 for each case studied). (a) Stoppers with surface treatment (TDS) and without the addition of wax. (b) Stoppers with TDS and with the addition of a disk of wax (3.5 mm thick). (c) Stoppers without TDS and without wax. (d) Stoppers without TDS and with the addition of a disk of wax. For waxed conditions, as indicated by the red arrow, the wax was applied 1 month after corking for corks with TDS and the same day for corks without TDS. Mean values and standard deviations are reported for each kinetic.

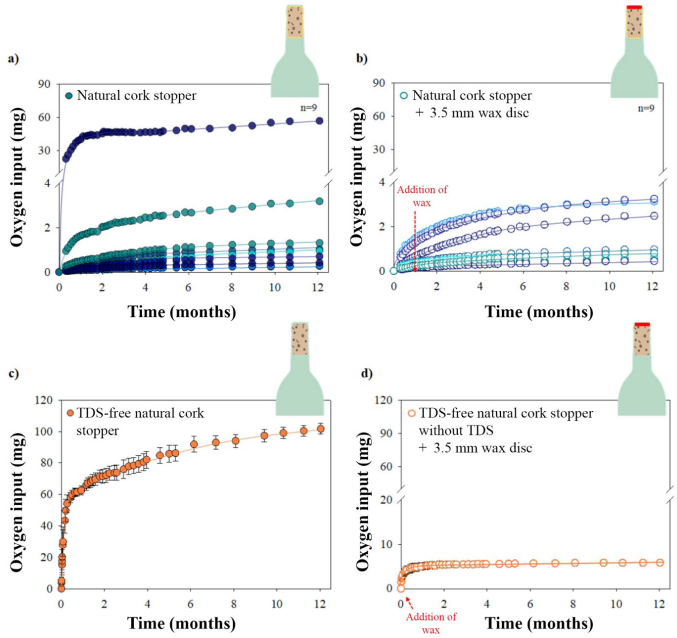


FIGURE 3. Quantity of oxygen entering bottles corked with natural cork stoppers (n = 9 for each case studied). (a) Stoppers with surface treatment (TDS) and without the addition of wax. (b) Stoppers with TDS and with the addition of a disk of wax (3.5 mm thick). (c) Stoppers without TDS and without wax. (d) Stoppers without TDS and with the addition of a disk of wax. For waxed conditions, as indicated by the red arrow, the wax was applied 1 month after corking for corks with TDS and the same day for corks without TDS. Due to the variability observed for study cases a and b, the kinetics for each sample are reported individually, whereas for cases c and d, mean values and standard deviations are reported.

considerably limit leakage at the glass/stopper interface (Figure 2d and 3d), thus compensating for the absence of surface treatment[10 11].

Conclusions

Waxes and resins have oxygen barrier properties that differ according to their chemical composition. For their application as an overcapping agent, regardless of the type of stopper considered, if the stopper has a lower permeability than the wax and a surface treatment that effectively limits transfer at the glass/stopper interface, the wax will play an aesthetic rather than a functional role.

If we extrapolate the results obtained on corks without surface treatment to the case of defective corks that would lead to high oxygen transfer (high intrinsic permeability and/or inadequate surface treatment), the application of wax would limit excessive oxygen ingress. However, in practice, the wax should not be applied immediately after corking to avoid any deformation of the cork, contrary to what was done in this study for corks without surface treatment. Instead, a period of at least one month should be observed, a critical period during which a significant ingress of oxygen would already have occurred.

This study was carried out over a period of one year under laboratory conditions (on empty bottles with constant external temperature and relative humidity). With regard to the longer-term storage of wine in bottles, other questions remain, such as the impact of cork hydration on the oxygen barrier properties of the cork and wax, and the evolution of the physico-chemical properties of the cork and wax over time as a function of storage conditions. ■

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