Kinetics of Oxygen Ingress into Wine Bottles Closed with Natural Cork Stoppers of Different Qualities

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Vanda Oliveira
University of Lisbon

Paulo Lopes
Amorim

Helena Pereira
University of Lisbon

Research Note

Kinetics of Oxygen Ingress into Wine Bottles Closed with Natural Cork Stoppers of Different Qualities

Vanda Oliveira,[1]* Paulo Lopes,[2] Miguel Cabral,[2] and Helena Pereira[1]

Abstract: The kinetics of oxygen ingress into bottles closed with natural cork stoppers was investigated by a nondestructive colorimetric measurement method using the oxidation of an indigo carmine solution. In order to encompass the natural variability of cork regarding its oxygen ingress into the bottle, 600 natural cork stoppers from different quality classes and produced from cork planks of different calipers were analyzed. The kinetics of oxygen transfer was similar in all cases and could be adjusted to logarithmic models. A significant variability was found for oxygen ingress into the bottles closed with natural cork stoppers: ingress at 12 months ranged from 0.3 to 4.8 mg; 21% of the stoppers reached the limit of oxygen quantification along the experiment. The results suggest that the variation of oxygen ingress is a consequence of the natural differing features in the cell dimensions and air volume within the stopper's structure.

Key words: oxygen ingress rate, colorimetric oxygen measurement, natural cork stoppers, cork permeability

The contact between wine and oxygen is of critical importance for wine conservation and bottle aging processes, during which wine characteristics evolve toward the appearance of developed characters (Godden et al. 2005). Wine postbottling development is complex: red wines benefit from a small degree of oxygenation as it contributes to color stabilization, astringency reduction, and aroma improvement (Lopes et al. 2005, Silva et al. 2011); white wines are less resistant to oxygen, leading to oxidative off-flavors and browning that reduce wine quality (Escudero et al. 2002, Karbowiak et al. 2010). However, a tight sealing and lack of oxygen can also lead to negative sensory attributes (Karbowiak et al. 2010).

The closure is the most obvious factor that influences in-bottle wine development. The main function of a closure is to ensure an appropriate seal, preventing liquid leakage and sensory deterioration during storage. The sealing performance of closures is strictly related to their permeability properties that are commonly used for evaluating their barrier efficiency (Godden et al. 2005, Karbowiak et al. 2010). Exposure to oxygen of bottled wines is usually low but can be variable depending, primarily, on the amount of oxygen in the headspace at bottling, the oxygen permeability of the bottle closure, and the storage conditions (Caillé et al. 2010, Mas et al. 2002, Lopes et al. 2005, 2006, Kontoudakis et al. 2008, Silva et al. 2011).

Cork is a cellular material with chemical inertia and a set of physical and mechanical properties that allow its use as a sealant (Fortes et al. 2004, Pereira 2007). Several studies have compared the oxygen permeability performance of different closure systems. The permeability to oxygen of natural cork stoppers decreased along time during a 36-month wine storage experiment, totaling 2.43 and 3.29 mg of oxygen for cork stoppers of two visual quality grades (Lopes et al. 2006). Technical cork stoppers (agglomerated cork topped by two discs of natural cork) allowed ingress of 1.0 to 1.2 mg of oxygen over 36 months, with low and constant rates 0.003 mg O_2/month (2 to 36 months). After 36 months postbottling, natural cork stoppers reached a mean permeation of 9.33 mg O_2/stopper/year and Altec technical closures reached 0.52 mg O_2/stopper/year (Godden et al. 2005). Screwcaps were the closures least permeable to oxygen (Godden et al. 2005). Synthetic stoppers were the least successful barriers to oxygen, reaching a mean oxygen ingress of 1.60 mg O_2 in the first month (Lopes et al. 2005). Despite their lower permeability to oxygen, chemical and sensory analyses showed that oxidation developed faster with plastic stoppers and metal screwcaps (Mas et al. 2002).

Several studies have focused on the mechanisms and main routes of oxygen ingress through closures. Lopes et al. (2007) found no significant differences in the oxygen ingress rates of cork-stopped bottles when comparing uncovered, interface-covered, and fully covered natural cork stoppers, suggesting that oxygen diffuses mainly out of the cork into the wine due to the high pressure in the cork cells. The high internal pressure (from 0.6 to 0.9 MPa) created when natural cork

[1]Centro de Estudos Florestais, Instituto Superior de Agronomia, Universidade Técnica de Lisboa, Tapada da Ajuda, P-1349-017 Lisboa, Portugal; and [2]Amorim & Irmãos, R&D Department, Rua de Meladas 380, P.O. Box 20, Mozelos, 4536-902, Portugal.

*Corresponding author (email: vandaoliveira@isa.utl.pt; tel: +351 213653491; fax: +351 213645000)

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stoppers are compressed into the bottleneck could force air out of the cork structure, preferentially during the first 12 months (Fortes et al. 2004, Lopes et al. 2007). In a recent study, Faria et al. (2011) investigated the permeability of gases through uncompressed cork and suggested that the gas transport mechanism occurs through minute channels present in the cork cell walls, the plasmodesmata that cross the wall and have a diameter of ~0.1 µm (Teixeira and Pereira 2009). The transfer of oxygen in raw cork may be essentially controlled by a mechanism of diffusion through the cork cell walls (Lequin et al. 2012).

The studies related to oxygen permeability in natural cork stoppers were primarily designed to compare different closure systems or storage positions and the samplings were limited in the number of replicates tested for each type of closure. For example, one study had a design with only four replicates of each natural cork commercial quality (Lopes et al. 2006) and another had 12 cork stoppers per reference class (Godden et al. 2005).

The natural variability of cork in terms of oxygen permeability has therefore not been adequately measured. Here we studied the kinetics of oxygen ingress into bottles closed with natural cork stoppers over 12 months using a large sample of stoppers encompassing the natural variability and a nondestructive colorimetric method (as described in Lopes et al. 2005). We analyzed natural cork stoppers from different quality classes and originating from cork planks of different calipers. The experimental set-up and measurement conditions were rigorously maintained to allow a broad confidence in the results.

Materials and Methods

Chemicals. Deionized water was purified with a Milli-Q water system (Millipore, Bedford, MA) prior to use. Indigo carmine was purchased from Merck (Darmstadt, Germany). Sodium dithionite and sodium benzoate were obtained from Merck and Sigma Aldrich (St Louis, MO), respectively.

Closures. A total of 600 natural cork stoppers (24 mm diameter x 45 mm length; supplied by Amorim & Irmãos, S.A., Santa Maria de Lamas, Portugal) were selected: 300 stoppers were punched out from cork planks of 27–32 mm caliper and 300 stoppers from cork planks of 45–54 mm caliper. The stoppers were randomly sampled from the production line after grading by an automated vision system, subsequently inspected by skilled operators and graded into three final reference quality classes: premium, good, and standard. From each class, 100 stoppers were taken before washing and surface treatment. The stoppers were used for the closure of bottles for oxygen ingress measurements. During the process seven bottles were lost and therefore measurements were taken from 593 bottles.

Bottles. Extra-white (colorless) bordelaise classic bottles (375 mL) were used for cylindrical closures. The bottleneck dimensions complied with the CETIE specifications: 18–19 mm diameter at a depth of 3 mm and 19–21 mm diameter at a depth of 45 mm from the bottle entrance. All bottles were supplied by Saint-Gobain Glass Packaging (Cognac, France). A calibration bottle (without bottleneck) was developed to allow the calibration procedure. This bottle had exactly the same dimensions, volume capacity, and glass thickness as the extra-white bordelaise classic bottles (375 mL). The bottle used for calibration was purchased from Atelier Jean Prémon (Bordeaux, France).

Method calibration procedure. The procedure for reduction and oxidation of indigo carmine solution in the calibration bottle is described in Lopes et al. (2005). In the current study, the procedure was adapted by adding sodium dithionate in a controlled excess in order to reduce the indigo carmine and consume some amount of oxygen (which led to a color change from blue indigo to yellow). This excess of sodium dithionate was predetermined to correspond to the necessary amount to consume the oxygen that enters into bottle due to the bottling operation (1.4 to 1.9 mg). For calibration, controlled amounts of oxygen were injected 28 different times into the reduced indigo carmine bottled solution. Until 1.9 mg of oxygen, the color solution did not change since the oxygen was being consumed by the excess of dithionate. Once the excess of sodium dithionate was consumed, the reduced indigo carmine began to consume the oxygen, resulting in a color change to the original indigo blue of the carmine. Color changes were measured with a colorimeter apparatus. Each point of the calibration curve was obtained by calculating the mean of five replicates. The calibration curve is valid up to a maximal limit of oxygen quantification of 5.7 mg, the amount necessary to fully oxidize the excess of sodium dithionate and to reduce the indigo carmine.

Bottling and storage. For the bottling trials, 600 sterilized commercial bottles with 375 mL were used. These bottles were filled with indigo carmine solution that was reduced with 20 mL sodium dithionite solution (2.9 g/L). Bottles were then sealed with different closures using a single-head corker (Bertolaso Epsilon R/S, Zimella, Italy). All closures were compressed to 16 mm diameter before insertion under vacuum into bottles. At 2 hours after bottling, the internal pressure values were 0 bar. These measurements were carried out in 30 bottles especially prepared for this purpose using a pressure gauge. The final filling level for each bottle was 65 ± 3 mm from the top of the bottle. The temperature of the indigo carmine solution ranged from 17.2 to 21.1°C. All bottles were left upright for 24 hr after bottling and then stored horizontally over 12 months. All bottles were stored under room conditions where temperature varied from 13.6 to 27.8°C.

Bottle colorimetric measurements. The CIELab measurements of the parameters L*, a*, b* were performed by directly scanning the bottled solutions with a Minolta series CM-508i spectrocolorimeter equipped with a transmittance accessory CM-A76 (Osaka, Japan). These measurements were obtained using illuminant D65 and a 10° observer according to CIELab 76 (McLaren 1980). A clean Pyrex bottle filled with water was used to carry out autozero calibration (blank). All bottles were cleaned with ethanol and dried before CIELab measurements. These measurements were carried out in the upright position at 5 cm from the base of the bottle. Four body measurements were collected by rotating each bottle 90° on its vertical axis. All positions were marked on the bottleneck to allow consistent measurement over time. All measurements were made in the dark at room temperature (18 ± 4°C).

Calculation of oxygen ingress. The calculation of the oxygen ingress into the bottle after closure with the cork stopper was made by deducting the oxygen that was already present in the bottle headspace to the values obtained by the colorimetric measurement. This means that the value of 1.50 mg of oxygen (present in the bottle headspace due to the bottling process) was withdrawn from all measurements, corresponding to the assumption of the rapid and total consumption of this oxygen by the indigo carmine (the bottles were shaken immediately after bottling and before each color measurement). Therefore the oxygen ingress was set at zero at time 0. The limit of oxygen measurement by the method was reached at 4.2 mg of oxygen. This value corresponds to the amount of oxygen that the method had the capacity to consume after the oxygen introduced due to bottling. Oxygen ingress rates were calculated on a daily (μg/day) and monthly (mg/month) basis.

For data analysis the natural cork stoppers were clustered into five oxygen ingress classes in function of the total oxygen measured at 12 months: class 1 comprised the stoppers for which the total oxygen measured in the bottle at 12 months was <1.4 mg; classes 2 and 3 comprised the stoppers with total oxygen at 12 months between 1.4 and 2.9 mg and between 2.9 and 4.2 mg, respectively; class 4 comprised the stoppers for which the limit of oxygen quantification (4.2 mg) was reached after the fourth month and class 5 comprised those that reached that limit before the fourth month. The clustering was done following the authors' choice without statistical procedures but ensuring that the distribution of the number of stoppers was approximately the same in the different oxygen ingress classes.

Data analysis. Microsoft Excel 2000 software was used for data analysis. All statistical analyses, namely, the analysis of variance (two-way ANOVA) were performed using SPSS statistical software (ver. 19.0; SPSS Inc., Chicago, IL).

Results

As noted above, the natural cork stoppers were clustered into five oxygen ingress classes in terms of the total oxygen measured at 12 months in order to simplify the data analysis. Twenty-one percent of the total cork stoppers reached the limit of oxygen quantification along the experiment and were assigned to oxygen classes 4 (11%) and 5 (10%). Classes 1, 2, and 3 included 23%, 32%, and 24%, respectively, of the total sample of cork stoppers.

The kinetics of oxygen ingress during the 12 months of storage clustered by cork plank caliper and oxygen class (1, 2, and 3 oxygen classes) of stoppers was determined (Figure 1). Despite the wide variability between stoppers (the coefficient of variation of the mean was 67% and 49% for the oxygen ingress measured at 5 and 360 days, respectively), a common behavior along time was found corresponding to a logarithmic kinetics of oxygen ingress with time. There was rapid and high oxygen ingress in the first days after bottling, with an initial high ingress rate, followed by decreasing ingress rates until the first month and further on, until stabilizing a low and rather constant ingress rate from the third to twelfth months. On average, 35% of the overall ingress of oxygen occurred in the first five days, 59% in the first month, and 78% in the first three months.

The kinetics models were all similar corresponding to logarithmic curves and differing mainly on their position on the y axis. For instance, on average, the oxygen ingress after 90 days was 0.77 mg, 1.50 mg, and 2.66 mg for classes 1, 2, and 3, respectively, and at the end of the 360 days, it was 0.99 mg, 1.92 mg, and 3.44 mg, respectively. Differences in bottle oxygen ingress were found for the three oxygen classes between the cork stoppers punched out from cork planks of different calipers. On average, the cork stoppers from the thicker caliper had a higher oxygen ingress of 3% (for class 1) and 8% (for classes 2 and 3) than the corks from the thinner caliper.

The evolution over time of the total oxygen ingress into the bottle averaged for the stoppers obtained from cork planks of the two calipers and the three reference quality classes is shown (Table 1). In these calculations, we only considered the stoppers that did not reach the limit of oxygen quantification within the 12 months. The two-way ANOVA of oxygen ingress indicated significant differences between cork stoppers of the two caliper thicknesses ($p = 0.00$) but no interaction between cork plank caliper and stopper quality class. For each cork plank caliper, the differences between quality classes had a low statistical significance ($p = 0.04$) and no between-class trend was found.

There were cork stoppers that reached the limit of oxygen quantification for the colorimetric method within 12 months (Figure 2). Within the 360 days of storage, 25% of the cork stoppers from the thicker caliper and 17% of the stoppers from the thinner caliper had reached the limit of oxygen quantification (6% and 15% of the stoppers, respectively, within 120 days).

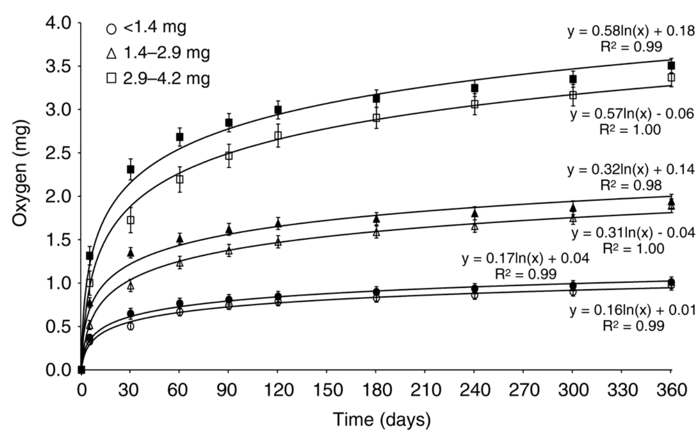

Figure 1 Kinetics of oxygen ingress into the bottle closed with cork stoppers of three oxygen classes divided into cork plank calipers (open symbols represent stoppers from 27–32 mm cork planks and filled symbols represent stoppers from 45–54 mm cork planks). Error bars represent the 95% confidence interval for the mean.

Table 1 Evolution in time of total oxygen ingress (mg) into the bottle closed with the natural cork stoppers grouped by cork plank caliper (27–32 mm and 45–54 mm) and reference quality classes (premium, good, and standard) after different periods of time (5 days, 1, 3, 6, and 12 months) and considering the oxygen classes 1 to 3 (n = 467). Parentheses enclose standard deviations.

	5 days	1 month	3 months	6 months	12 months
27–32 mm caliper	0.56 (0.41)	0.97 (0.60)	1.39 (0.76)	1.60 (0.87)	1.88 (0.99)
Premium	0.63 (0.44)	1.03 (0.59)	1.52 (0.76)	1.74 (0.87)	2.04 (0.95)
Good	0.49 (0.38)	0.90 (0.54)	1.32 (0.70)	1.53 (0.82)	1.76 (0.94)
Standard	0.55 (0.39)	0.97 (0.65)	1.32 (0.79)	1.55 (0.91)	1.85 (1.07)
45–54 mm caliper	0.90 (0.50)	1.58 (0.74)	1.92 (0.86)	2.10 (0.93)	2.35 (1.02)
Premium	0.92 (0.53)	1.72 (0.71)	2.06 (0.81)	2.26 (0.89)	2.46 (0.94)
Good	0.92 (0.53)	1.50 (0.77)	1.71 (0.85)	1.89 (0.92)	2.17 (1.06)
Standard	0.87 (0.46)	1.53 (0.72)	2.03 (0.88)	2.16 (0.94)	2.43 (1.04)

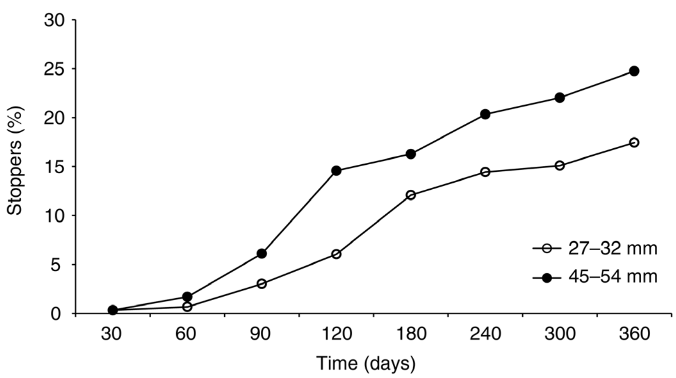

Figure 2 Percentage of natural cork stoppers from the two cork plank calipers that reached the limit of oxygen quantification.

Table 2 Oxygen ingress rates (μg/day) into the bottle closed with cork stoppers of each oxygen class (1 to 5) during 12 months.

	Oxygen class					
Time	1	2	3	4	5	Total
0–5 days	68.02	130.25	236.61	287.39	420.96	188.19
5–30 days	8.19	20.70	35.29	41.26	57.04	27.25
Month 1	18.16	38.96	68.84	82.28	117.70	54.07
Month 2	5.05	7.07	13.76	20.57	13.19	10.29
Month 3	2.18	4.06	7.00	11.65	10.43	5.75
Month 4	1.39	2.77	6.12	8.85	12.84	4.57
Months 4–12	0.76	1.38	2.41	5.48	–	2.51

Results confirmed that the oxygen ingress rate of natural cork stoppers depends on time (Table 2), as stated previously by Lopes et al. (2006). The rates of oxygen ingress in the first month of storage were statistically different ($p = 0.00$) than the rates from the second until the twelfth month, independently of the oxygen class considered. The oxygen ingress rates in the first month were 18.16 and 68.84 μg/day for classes 1 and 3, respectively. The average oxygen ingress rates after the fourth month of storage were 0.76, 1.38, and 2.41 μg/day, respectively, for classes 1, 2, and 3.

Discussion

In order to understand and explain the variability found in oxygen ingress into the bottle, it is necessary to analyze the cork cell structure and the amount of oxygen contained therein, as oxygen ingress occurs mainly out of the cork due to the high internal pressure in the cork cells created when the cork stoppers are compressed into the bottleneck (Ribéreau-Gayon 1933). Natural cork stoppers with 24 mm diameter x 45 mm length have a volume of 20.4 mL, of which 80 to 85% is air contained in the cell lumen, implying 4.9 to 5.2 mg (3.4–3.6 mL) of oxygen within their structure (Fortes et al. 2004). Therefore, the average oxygen ingress into the wine bottles (Figure 1), in relation to the theoretically estimated total oxygen within the stoppers, represented 27 to 29% (1.0 mL, or 1.43 mg) in the first month of storage, and 42 to 45% (1.5 mL, or 2.15 mg) at the end of the 12-month storage.

There are no published data relating the thickness of the cork planks and the quality and permeability to oxygen of cork stoppers produced from them. Cork planks with thicker calipers have larger growth rings than thinner cork planks, and they also differ in the proportion of the earlycork and latecork cells, since the number of latecork cells in a radial row is independent of the cork ring width (Pereira et al. 1992). Earlycork cells are larger than latecork cells and have thinner cell walls, and consequently they differ in their lumen empty (air-filled) volume: approximately 91 to 92% in the earlycork region and 78 to 85% in the latecork region (Pereira 2007). The average cell prism height (radial direction) was 22% higher in the cells of larger growth rings (Pereira et al. 1992). Therefore, cork planks with thinner caliper have a higher proportion of smaller cells with thicker cell walls, and less air-filled empty volume which increases the barrier to the diffusion of gases, explaining the significant differences found in oxygen ingress of the stoppers produced from the thinner and thicker calipers (Table 1, Figure 1). Our results show that, on average, 1.88 mg and 2.35 mg of oxygen diffuses from natural cork stoppers from 27–32 mm and 45–54 mm calipers, respectively (Table 1), representing 36 to 38% and 47 to 50% of the theoretically total oxygen in the cell structure. These results are in agreement with others (Faria et al. 2011), who noted that samples from thinner planks on average permeate less than those from thicker planks.

The results showed no significant correlations between the reference quality classes and the oxygen ingress (Table 1). These results are in accordance with others (Lopes et al.

2005), who found no significant differences in the overall oxygen diffusion of stoppers of different quality.

The occurrences of structural discontinuities, like lenticular channels or woody inclusions, which may cause variation in the mechanical properties of cork, eventually explain this higher oxygen ingress. The cork stoppers from thicker caliper reached the limit of oxygen quantification in higher number and earlier than the cork stoppers from thinner caliper (Figure 2). The thickness of the cork plank is also a factor of mechanical behavior variation, with larger caliper planks showing lower strength in compression for all strains (Pereira et al. 1992).

Independently of the oxygen class of the stoppers, the oxygen ingress rates were higher in the first month of storage, and particularly high in the first days after bottling, decreased thereafter, and stabilized at rather constant values after 3 months (Figure 1), suggesting a decrease in the permeability of the cork closure over time (Karbowiak et al. 2010). The higher air pressure within the cells due to the compression of the stopper in the bottleneck certainly will increase ingress rates immediately after bottling as oxygen ingress progresses, the pressure decreases and so the ingress rates, until reaching a rather constant value.

The rates of oxygen ingress found for the cork stoppers of oxygen classes 1, 2, and 3 (Table 2) are within the range and exhibit similar trends to those reported by Lopes et al. (2006), who published values of 35.8 to 64.6 μg/day (25 to 45 μL/day) in the first month and ranging from 2.43 to 8.73 μg/day (1.7 to 6.1 μL/day) from the second to twelfth months for the bottles stored horizontally. For natural cork stoppers, Godden et al. (2005) reported a mean oxygen permeation of 25.6 μg/day (17.9 μL/day), with a range from 0.1 to 175.7 μg/day (0.1 to 122.7 μL/day), tested after ~36 months postbottling.

Conclusion

The variability of oxygen ingress into the bottle closed with the cork stoppers led us to classify the stoppers in oxygen ingress classes. The kinetics of oxygen ingress was similar regardless of the oxygen class and could be adjusted to similar logarithmic models with high statistical significance. The results suggest that this variation of oxygen ingress is a consequence of the natural differing features in the cell dimensions and air volume within the stopper's structure.

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