Performance Evaluation of Nine Wine Bottle Closures During 18 Months of Cellaring a White Wine

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Abstract: A wine's evolution and aging potential once bottled may be affected by the type of closure used. By virtue of a closure's physical characteristics, it may allow more or less oxygen to enter the bottle, both through and around the closure. Oxygen can then interact with phenolic substances, ethanol and other wine compounds and consume sulfur dioxide (SO_2), and alter the wine's chemistry including taste (aromas and flavors). Initially, these alterations may improve a wine's quality, but excessive oxygen can cause premature oxidation and have detrimental effects on quality, therefore shortening the shelf life of the wine. This study examined closure performance over an 18-month cellaring period of a Chardonnay bottled under nine closures: a microagglomerate (Gültig Carat), two natural corks (Bosa UF25 and Bosagrape's Best), a Mosti Mondiale twin-disc technical, and five synthetics (Nomacorc Select 900, Select Bio and Classic Green, and Cork Supply VINC$^+$ and VINC^{NEO}). Each bottle was analyzed monthly for headspace, dissolved and total package oxygen, and free and total SO_2 to determine total oxygen consumed and what portion can be attributed to oxygen ingress due to the closure. Color evolution was also analyzed. Although oxygen and total SO_2 were consumed differently in wines under different closures and with uncharacteristically high levels of binding, the SO2:O2 consumption ratio was lowest in the wine under a UF25 closure, the only ratio under the mean and median. However, closures can transfer varying and significant amounts of oxygen into the headspace upon compression and insertion into bottles; natural cork UF25 and Bosagrape's Best closures contained, or at least transferred, the least amount of oxygen. A non-blind tasting after 18 months did not reveal any differences in aromas or flavors; none demonstrated flaws. This study demonstrates that, although closures performed differently from an oxygen ingress perspective based on oxygen and total SO_2 consumption analyses as well as color analysis, all closures are deemed appropriate for cellaring a fruity-style white wine for up to 18 months. Better bottling equipment and process, including inerting headspace, can extend shelf life significantly.

Key words: wine closures, oxygen transfer rate (OTR), headspace oxygen (HSO), dissolved oxygen (DO), total package oxygen (TPO), total consumed oxygen (TCO), free sulfur dioxide (FSO2), bound sulfur dioxide (BSO2), total sulfur dioxide (TSO2), wine oxidation

Introduction. The choice of bottle closure (stopper) can have a significant impact on wine quality and aging potential depending on physical characteristics, primarily type of material, density, length and diameter, and oxygen content. A poor closure can allow the uptake of higher than desirable amounts of oxygen and cause premature oxidation reactions that can alter aromas, flavors and color, and also increase the risk of microbial spoilage of wines that are not bottled under sterile conditions as free sulfur dioxide (SO_2), or FSO2, falls to critically low levels, and therefore reduce shelf life.

Winemakers have a vast choice of closure types including cork, microagglomerate, synthetic, screw caps, and glass, and designed to allow the uptake of different amounts of oxygen, or what is referred to as oxygen transfer rate (OTR), to match specific styles of wine and their intended aging. OTR are typically expressed in mL or mg of oxygen (O_2) per day, month or year. For any given closure, OTR can change with closure age during cellaring, for example, it may decrease as efficacy diminishes, or increase as in the case of most natural cork closures.

The focus of this study is cork, microagglomerate and synthetic closures.

Cork closures include those manufactured entirely from cork, the material extracted from the outermost layer of the bark of *Quercus suber* oak trees, and comprise three broad categories, as defined in Pereira (2007) to avoid confusion since the terms are often used interchangeably in the industry: natural, agglomerated, and technical closures.

Natural closures are punched out as single pieces entirely from reproduction cork, the highest grade cork that has the least amount of physical defects but possibly the highest variability due to the natural aspect and the most proneness to cork taint, or what is known as TCA, short for 2,4,6-trichloroanisole, the compound responsible for giving affected wine a moldy character or wet-dog smell.

Agglomerated closures are manufactured using coarsely or finely ground granules derived from virgin and second cork, from rejected reproduction cork or manufacturing leftovers, or from other cork by-products (Pereira 2007), which have a higher rate of defects. Agglomerated closures manufactured from very finely ground granules are also known as microagglomerates. The granules are glued together with an acid-tolerant binding agent,

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such as food-grade polyurethane or a plant-based compound, and then hot-press molded into their cylindrical shape.

Technical closures are composites manufactured with an agglomerated body and then finished with a natural disc at one or both ends so that only natural cork material is in contact with wine during bottle storage. Twin-disc technical closures, also called 1+1 stoppers, have a natural disc at each end.

Since agglomerated and technical closures contain natural cork, these are also at risk of TCA contamination.

The standard closure size for standard 750-mL bottles is 24 mm (15⁄16 inch) in diameter and 45 mm (1¾ inches) in length with, possibly, chamfered rims to ease insertion into bottles when used with manual corkers. Premium wines typically use 54-mm (2-inch) long natural closures while 38-mm (1½-inch) agglomerates and technical closures are most popular in home winemaking.

Synthetic closures are manufactured entirely from synthetic polymers or plant-based biopolymers, such as sugar cane, with no natural cork material, and therefore free of possible TCA contamination.

During bottle aging, the chemistry of the wine, and therefore its quality, changes based on a number of factors, primarily, oxygen consumption and the availability of SO_2 to modulate the rate of oxidation reactions.

At bottling, wine contains a certain amount of oxygen, referred to as dissolved oxygen, or DO, which varies depending on the type of equipment and care taken directly before and during the bottling operation. If the bottle and headspace are not purged with an inert gas, such as nitrogen, the headspace (ullage) too will contain a certain amount of oxygen, referred to as headspace oxygen, or HSO, from not only atmospheric oxygen but also from air trapped within the closure material and which is transferred into the headspace when the closure is compressed and inserted in the bottle neck, a phenomenon known as outgassing.

A standard wine bottle with a natural cork rim with an opening diameter of 18.5 mm (¾ in) and a headspace of 12 mm (½ in), representing a headspace volume of approximately 3.2 mL measured at 20°C (68°F), the amount of oxygen due to 100% air in non-purged headspace is approximately 0.7 mL, or 0.9 mg, thus contributing approximately 1.2 mg DO/L. And outgassing from a 45×24 natural cork can contribute 3 mL or more (Stelzer et al. 2005; Lopes et al. 2007), or the equivalent of approximately 5.3 mg DO/L in a standard bottle.

The sum of DO and HSO is known as total package oxygen, or TPO. Under sound packaging practices in a well-controlled environment and purged headspace (HSO = 0 mg/L), TPO values immediately after bottling should be lower than 2 mg/L, while some authors recommend levels below 1 mg/L, for example, 0.5 mg/L (Stelzer et al. 2005). As a best practice, DO should be below 1 mg/L, and also depending on the level of FSO2 at bottling.

Once bottled and the closure applied, TPO is completely consumed within an amount of time depending on initial TPO and FSO2 levels and temperature, after which the oxygen concentration should be relatively low and only maintained by ingress through the bottle closure; however, OTR depends on the type and integrity of the closure utilized (Lopes et al. 2007) and the type of corking equipment. In home winemaking, because of the type of bottling equipment and method used and usually not purging the headspace, TPO can attain levels close to 8 mg/L.

When closure OTR data is known, total consumed oxygen (TCO) in a wine under that closure can be determined from TPO measurements in mg/L at bottling (TPO_b) and at a measurement point in time (TPO_p), the closure's OTR in mg/L/days, and the aging period in days, as per the following equation (Dimkou et al. 2011; Waterhouse et al. 2016; Pascal et al. 2019):

$$TCO = TPO_b - TPO_p + OTR \times days$$

When OTR data is not known, the amount of oxygen entering bottles from around the closure or diffusing through its material over the course of the cellaring period can be calculated by reworking the above equation as follows:

$$OTR \times days = TCO - TPO_b + TPO_p$$

TCO can be determined analytically by measuring the decrease in total SO_2 (TSO2), which is proportional to the amount of oxygen consumed, whilst the decrease in FSO2 is not because, when oxidation occurs and oxygen reacts with FSO2, FSO2 decreases due to oxidation to sulfate, and some SO_2 is released from the pool of bound SO_2. The drop in TSO2 is then divided by the theoretical stoichiometric ratio between SO_2 and O_2, where 1 mg of O_2 consumes 4 mg of SO_2 (FSO2). However, Diéval et al. (2013) reported in a study that 1 mg of O_2 consumes 2–2.5 mg of SO_2 while, more recently, Schneider (2019) reported a median of 2.86 mg. Wine contains many weak SO_2 binders, but more important, DO can oxidize ethanol into acetaldehyde, a very strong SO_2 binder, which can result in different binding behaviors depending on the initial TPO.

TPO can be derived by measuring DO and HSO non-intrusively and non-destructively with a DO meter equipped with an optical sensor.

Color evolution too can be assessed in white wines by measuring absorbance in absorbance units (a.u.) at 420 nm, denoted A_{420}, although A_{420} cannot be used to determine the amount of oxygen consumed since the concentration of the color-producing compounds, flavonoid phenols, also contributes to the result. Under comparable conditions (identical SO_2), browning depends on the concentration of flavonoid phenols (Lee and Jaworski 1988; Schneider 2019).

The purpose of this study was to assess and compare the performance of nine different cork and synthetic closures by measuring and analyzing dissolved oxygen, headspace oxygen and total package oxygen data in conjunction with free and total SO_2 data, as well absorbance measurements to assess any color impacts, and a non-blind tasting by the author.

Materials and Methods

Closures. Nine different closures from different manufacturers/suppliers were used: Carat (Gültig Corks and Closures, Heinrich Gültig Korkwarenfabrikation GmbH); UF25 and Bosagrape's Best (Bosagrape Winery Supplies Ltd); Technical twin-disc (obtained from a Mosti Mondiale Meglioli Kit), referred to as MM Technical, hereafter; Nomacorc Select 900, Nomacorc Select Bio, Nomacorc Classic Green, VINC$^+$, and VINC^{NEO} (Cork Supply USA). Table 1 lists closure types, specifications, if available (N/A=not available), and characteristics, including recommended aging periods, obtained from manufacturers' or suppliers' data specification sheets (corrections, e.g., length, were made once confirmed by measurements). OIR is oxygen initial release, i.e., the maximum amount of oxygen transferred from the closure material into the headspace upon compression, and OTR is the oxygen transfer rate. For analysis and calculations in this study, oxygen data expressed in mL has been converted to mg using a conversion factor of 1.33 (20°C, 1 atm).

Wine. The wine used for this study was a 2020 unoaked Chardonnay with pre-bottling parameters as per Table 2. No ascorbic acid was added, and therefore it had no impact on oxygen consumption. After cold settling, before yeast inoculation, a gallotannin preparation, Laffort Tanin Galalcool SP, was added at a rate of 10 g/hL. Although gallotannins are oxygen scavengers, their impact on oxygen consumption should be minimal.

Bottles and Bottling: Standard 750-mL flint Bordeaux-style bottles with 18.5-mm (¾-in) opening fitted with pre-calibrated 5-mm PreSens technology oxygen-sensitive spots (PSt3) glued with silicone, one on the glass inside in the headspace volume and one in the main body.

Bottles were filled with a gravity-type filler and immediately corked with a brass-jawed floor corker and without inerting or purging the headspace. Bottles were cellared in an upright position for 24 hours to allow to reach equilibrium, then the first set of oxygen measurements were made – this represents time 0 (T0). Oxygen measurements were then made on the same day at one-month intervals for 18 months, or a total of 549 days; monthly measurement points are represented as T0+1, T0+2, . . ., T0+18. Bottles were cellared in an upright position at 13°C (55°F). At T0+18, oxygen measurements were made as in all previous months, then bottles were uncorked to take samples to measure free and total SO_2 and absorbance parameters. Only one

Table 1: Closure types, specifications and characteristics

	Type	Diameter (mm)	Length (mm)	OIR	OTR	Other characteristics and recommended aging period
Carat	Microagglomerate	24.0	44.0	N/A	N/A	2-mm chamfers
UF25	Natural cork	24.0	44.0	N/A	N/A	3–5 years
Bosagrape's Best	Natural cork	24.0	44.0	N/A	N/A	5+ years
MM Technical	Technical, twin-disc	23.5	39.5	N/A	N/A	
Nomacorc Select 900	Synthetic, polymer	22.0	44.5	N/A	3.2 mg O_2 after 12 months	1-mm chamfers
Nomacorc Select Bio	Synthetic, plant-based biopolymer	22.0	47.0	N/A	N/A	1-mm chamfers
Nomacorc Classic Green	Synthetic, plant-based biopolymer	22.5	43.0	N/A	1.70 mg O_2 after 3 months 2.22 mg O_2 after 6 months 3.12 mg O_2 after 12 months 1.74 mg O_2/year after 1 year	1-mm chamfers
VINC$^+$	Microagglomerate	24.0	49.0	N/A	0.0023 mL O_2/day	2-mm chamfers <5 years
VINC^{NEO}	Microagglomerate	24.0	44.0	N/A	0.0029 mL O_2/day	2-mm chamfers <2 years

Table 2: 2020 unoaked Chardonnay pre-bottling parameters

pH	TA (g/L)	VA (mg/L)	%ABV	RS (g/L)	BSO2 (mg/L)	FSO2 (mg/L)	TSO2 (mg/L)	DO (µg/L)	A_{420} (a.u.)
3.21	6.30	324.0	12.6	4.54	67.6	28.0	95.6	775	0.038

measurement was made for each parameter. Wines were also tasted by the author, non-blind, at T0+18.

Test Equipment. A NomaSense O_2 P300 Oxygen Analyzer was used to measure headspace oxygen (HSO) and dissolved oxygen (DO) from the PSt3 oxygen-sensitive spots; total package oxygen (TPO), given in mg/L, was calculated from the sum of HSO (mg/L) and DO (mg/L). HSO (mg/L) values were calculated from HSO measurements made in hPa and temperature (°C), bottle volume (750 mL), and headspace volume (calculated from the bottle opening diameter, 18.5 mm, and headspace length (measured with a ruler).

A Hanna Instruments HI 902C Potentiometric Titrator with an ORP electrode and Hanna reagents were used to measure free SO_2 (FSO2) and total SO_2 (TSO2) using the Orienting Ripper Method. Bound SO_2 (BSO2) was calculated by subtracting FSO2 measurements from TSO2 measurements. FSO2 and TSO2 were only measured in the wine pre-bottling, and then in each wine at T0+18.

A Thermo Scientific Genesys 10S UV-Vis spectrophotometer was used to measure absorbances at 420 nm using 10-mm (pathlength) quartz cuvettes using undiluted wine samples and against a blank (distilled water).

Data Analysis and Limitations. For each closure, DO and HSO data was analyzed and charted over the 18-month period of this study to assess oxygen ingress and consumption, and then FSO2 and TSO2 measured at the end of the study and compared to measurements in the wine pre-bottling to quantify total consumed oxygen (TCO). The SO2:O2 ratio used to determine TCO from SO_2 consumption analysis was determined as an average of all closures from TSO2 consumed divided by TPO at bottling. From this analysis, the approximate total amount of oxygen due to ingress over the duration of the study was estimated. Absorbance data for each closure was also charted for comparison analysis.

These analyses are limited in that only one measurement was made for each parameter.

Results and Discussion

Oxygen Initial Release (OIR). Bottling resulted in DO increases between 1.15 mg/L and 1.95 mg/L, with DO at T0 in a fairly narrow range of 1.92–2.72 mg/L. These values and variability are typical of bottling with a gravity filler and without inerting or headspace purging.

TPO ranged from 2.78 mg/L (Bosagrape's Best) to 6.99 mg/L (Select Bio), and this large range is due to the large range in HSO measurements at T0; HSO was lowest with a Bosagrape's Best closure at 0.86 mg/L, and highest with a Select Bio closure at 4.58 mg/L. Only UF25 and Bosagrape's Best closures had HSO below 2.0 mg/L, and below the mean of 3.33 mg/L and the median of 3.68 mg/L.

Table 3 lists calculated OIR per closure. OIR, calculated as the difference between HSO at T0 and the estimated amount of oxygen prior to inserting the closure, but not accounting for any oxygen that may have become dissolved during the 24-hour equilibration period (hence the negative values), were lowest in the natural cork closures, i.e., UF25 and Bosagrape's Best, and therefore it is concluded that these contain, or at least transfer, the least amount of oxygen into the headspace upon compression and insertion. All other closures had calculated OIR values above 1 mg/L, with the Select Bio highest at 2.78 mg/L. More accurate OIR values can be obtained by measuring HSO immediately upon inserting closures.

Table 3: Calculated oxygen initial release (OIR) per closure

CLOSURE	HSO at T0 (mg/L)	Estimated oxygen prior to closure (mg/L)	OIR (mg/L)
Carat	3.83	2.10	1.73
UF25	1.48	1.70	-0.22
Bosagrape's Best	0.86	1.70	-0.84
MM Technical	3.17	2.00	1.16
Select 900	3.68	1.70	1.98
Select Bio	4.58	1.80	2.78
Classic Green	3.54	1.80	1.74
VINC^{+}	4.30	2.20	2.09
VINC^{NEO}	4.54	2.60	1.94

Headspace and Dissolved Oxygen. Table 4 lists HSO, DO and TPO measurements per closure at each month interval.

Over the course of the 18-month cellaring period, for the wine bottled with a Carat closure, DO dropped to 0 mg/L within 7 months while HSO and TPO dropped to 0 mg/L within 16 months. The HSO drop was gradual and never increased during cellaring, suggesting that the wine was consuming oxygen at a faster rate than oxygen ingress through and around the closure, and that oxygen ingress was very low. These results are similar to those for the MM Technical closure.

For the wine bottled with a UF25 closure, DO dropped to 0 mg/L within 5 months, the quickest of all closures, while HSO and TPO never dropped to 0 mg/L, suggesting a very low but measurable amount of oxygen ingress through and around the closure, and that the wine was consuming oxygen at a slightly slower rate than oxygen ingress.

For the wine bottled with a Bosagrape's Best closure, DO dropped to 0 mg/L within 8 months while HSO and TPO never dropped to 0 mg/L, suggesting a very low but measurable amount of oxygen ingress through and around the closure. Given the longer period to reach zero DO and the higher levels of HSO, this natural cork closure performed less efficiently than the UF25 from the same vendor, but similar to the Select 900 closure. The period to reach zero DO was however comparable to all other closures in spite of having the lowest initial HSO.

For the wine bottled with a MM Technical closure, DO dropped to 0 mg/L within 6 months while HSO and TPO, for all practical purposes, dropped to 0 mg/L within 16 months, mirroring the performance of the Carat closure.

Table 4: HSO, DO and TPO measurements per closure at each month interval from T0 to T0+18

TIME	Carat			Bosa's UF25			Bosa's Best			MM Technical		
	HSO (mg/L)	DO (mg/L)	TPO (mg/L)	HSO (mg/L)	DO (mg/L)	TPO (mg/L)	HSO (mg/L)	DO (mg/L)	TPO (mg/L)	HSO (mg/L)	DO (mg/L)	TPO (mg/L)
T0	3.83	2.33	6.16	1.48	2.15	3.63	0.86	1.92	2.78	3.17	2.03	5.20
T0+1	1.87	2.05	3.92	0.84	0.88	1.72	0.89	0.50	1.38	1.53	0.96	2.49
T0+2	0.90	1.60	2.50	0.60	0.39	0.99	0.84	0.17	1.01	0.66	0.94	1.60
T0+3	0.44	0.96	1.40	0.35	0.16	0.51	0.75	0.10	0.85	0.28	0.51	0.79
T0+4	0.26	0.48	0.74	0.24	0.04	0.27	0.67	0.07	0.74	0.16	0.15	0.31
T0+5	0.13	0.22	0.35	0.18	0.00	0.18	0.54	0.05	0.59	0.10	0.03	0.12
T0+6	0.07	0.08	0.15	0.10	0.01	0.11	0.43	0.08	0.51	0.04	0.00	0.04
T0+7	0.04	0.00	0.04	0.06	0.00	0.06	0.22	0.08	0.29	0.02	0.00	0.02
T0+8	0.03	0.00	0.03	0.05	0.00	0.05	0.13	0.00	0.13	0.02	0.00	0.02
T0+9	0.02	0.00	0.02	0.05	0.00	0.05	0.14	0.00	0.14	0.02	0.00	0.02
T0+10	0.03	0.00	0.03	0.06	0.00	0.06	0.13	0.00	0.13	0.03	0.00	0.03
T0+11	0.03	0.00	0.03	0.09	0.00	0.09	0.15	0.00	0.15	0.04	0.00	0.04
T0+12	0.03	0.00	0.03	0.08	0.00	0.08	0.13	0.00	0.13	0.03	0.00	0.03
T0+13	0.02	0.00	0.02	0.10	0.00	0.10	0.15	0.00	0.15	0.04	0.00	0.04
T0+14	0.01	0.00	0.01	0.11	0.00	0.11	0.16	0.00	0.16	0.04	0.00	0.04
T0+15	0.01	0.00	0.01	0.10	0.00	0.10	0.13	0.00	0.13	0.02	0.00	0.02
T0+16	0.00	0.00	0.00	0.03	0.00	0.03	0.04	0.00	0.04	0.01	0.00	0.01
T0+17	0.00	0.00	0.00	0.01	0.00	0.01	0.02	0.00	0.02	0.00	0.00	0.00
T0+18	0.00	0.00	0.00	0.02	0.00	0.02	0.03	0.00	0.03	0.01	0.00	0.01

TIME	Select 900			Select Bio			Classic Green			VINC+			VINC-NEO		
	HSO (mg/L)	DO (mg/L)	TPO (mg/L)	HSO (mg/L)	DO (mg/L)	TPO (mg/L)	HSO (mg/L)	DO (mg/L)	TPO (mg/L)	HSO (mg/L)	DO (mg/L)	TPO (mg/L)	HSO (mg/L)	DO (mg/L)	TPO (mg/L)
T0	3.68	2.72	6.40	4.58	2.41	6.99	3.54	2.59	6.13	4.30	2.45	6.75	4.54	2.27	6.81
T0+1	2.11	1.83	3.94	2.67	1.51	4.18	2.06	1.53	3.59	2.23	2.20	4.43	2.42	2.04	4.46
T0+2	1.47	1.12	2.59	1.73	1.00	2.73	1.34	0.90	2.23	1.06	1.81	2.87	1.39	1.60	2.99
T0+3	0.92	0.76	1.68	0.98	0.74	1.71	0.77	0.64	1.41	0.53	1.20	1.73	0.80	1.12	1.92
T0+4	0.63	0.47	1.10	0.64	0.39	1.04	0.56	0.29	0.85	0.34	0.65	1.00	0.53	0.65	1.18
T0+5	0.40	0.33	0.72	0.36	0.26	0.62	0.33	0.23	0.56	0.22	0.37	0.59	0.33	0.41	0.73
T0+6	0.26	0.21	0.47	0.18	0.17	0.35	0.16	0.16	0.32	0.14	0.22	0.35	0.17	0.26	0.43
T0+7	0.15	0.13	0.27	0.10	0.06	0.16	0.10	0.04	0.14	0.09	0.09	0.18	0.10	0.11	0.21
T0+8	0.11	0.04	0.15	0.07	0.00	0.07	0.07	0.00	0.07	0.06	0.03	0.09	0.07	0.02	0.10
T0+9	0.13	0.00	0.13	0.07	0.00	0.07	0.07	0.00	0.07	0.06	0.00	0.06	0.07	0.00	0.07
T0+10	0.14	0.00	0.14	0.07	0.00	0.07	0.07	0.00	0.07	0.06	0.00	0.06	0.05	0.00	0.05
T0+11	0.15	0.00	0.15	0.09	0.00	0.09	0.09	0.00	0.09	0.06	0.00	0.06	0.06	0.00	0.06
T0+12	0.17	0.00	0.17	0.09	0.00	0.09	0.10	0.00	0.10	0.06	0.00	0.06	0.05	0.00	0.05
T0+13	0.17	0.00	0.17	0.10	0.00	0.10	0.12	0.00	0.12	0.06	0.00	0.06	0.05	0.00	0.05
T0+14	0.15	0.00	0.15	0.09	0.00	0.09	0.11	0.00	0.11	0.05	0.00	0.05	0.04	0.00	0.04
T0+15	0.09	0.03	0.11	0.05	0.00	0.05	0.10	0.00	0.10	0.02	0.00	0.02	0.02	0.00	0.02
T0+16	0.04	0.01	0.05	0.02	0.00	0.02	0.03	0.00	0.03	0.01	0.00	0.01	0.00	0.00	0.00
T0+17	0.03	0.01	0.03	0.01	0.00	0.01	0.02	0.00	0.02	0.00	0.00	0.00	0.00	0.00	0.00
T0+18	0.05	0.00	0.05	0.02	0.00	0.02	0.03	0.00	0.03	0.01	0.00	0.01	0.00	0.00	0.00

For the wine bottled with a Select 900 closure, DO dropped to 0 mg/L within 9 months with non-zero DO for a three-month period, while HSO and TPO never dropped to 0 mg/L, suggesting that the wine was consuming oxygen at a slower rate than oxygen ingress through and around the closure. These results are similar to those for the Bosagrape's Best closure, and very similar to other synthetic closures although HSO was slightly higher.

For the wine bottled with a Select Bio closure, DO dropped to 0 mg/L within 8 months while HSO and TPO never dropped to 0 mg/L, suggesting a low but measurable amount of oxygen ingress through and around the closure was very low but that the wine was consuming oxygen at a slower rate than oxygen ingress. These results are similar to those for other synthetic closures.

For the wine bottled with a Classic Green closure, DO dropped to 0 mg/L within 8 months while HSO and TPO never dropped to 0 mg/L, also suggesting a very low but measurable amount of oxygen ingress through and around the closure but that the wine was consuming oxygen at a slower rate than oxygen ingress. These results are similar to those for other synthetic closures.

For the wine bottled with a VINC$^+$ closure, DO dropped to 0 mg/L within 9 months while HSO and TPO never dropped to 0 mg/L, also suggesting a very low but measurable amount of oxygen ingress through and around the closure but that the wine was consuming oxygen at a slower rate than oxygen ingress. These results are similar to those for other synthetic closures.

For the wine bottled with a VINC^{NEO} closure, DO dropped to 0 mg/L within 9 months while HSO and TPO dropped to 0 mg/L within 16 months. The HSO drop was gradual and never increased during cellaring, suggesting that the wine was consuming oxygen at a faster rate than oxygen ingress through and around the closure, and that oxygen ingress was very low. These results are similar to those for other synthetic closures although it is the only one clearly dropping to zero HSO.

Of importance in the above analysis is closure reaching and maintaining zero HSO as the time to zero DO depends on the initial HSO.

Sulfur Dioxide. Figure 1 illustrates SO_2 levels for the base wine and wine under each closure comparing FSO2 and BSO2 (and TSO2) at the start (T0) and end of the study (T0+18); Figure 2 illustrates TSO2 consumption amounts for each closure; and Figure 3 illustrates approximate SO2:O2 ratios based on oxygen and total SO_2 consumptions.

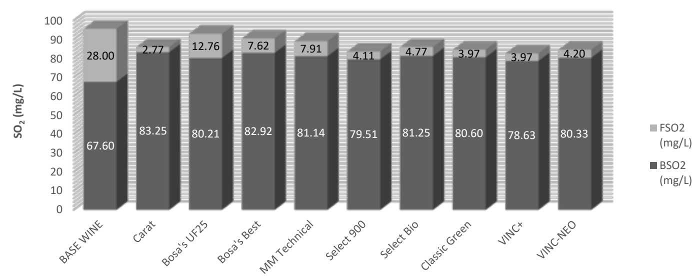

Figure 1: SO_2 levels for the base wine and wine under each closure comparing FSO2 and BSO2 (and TSO2) at T0 and T0+18

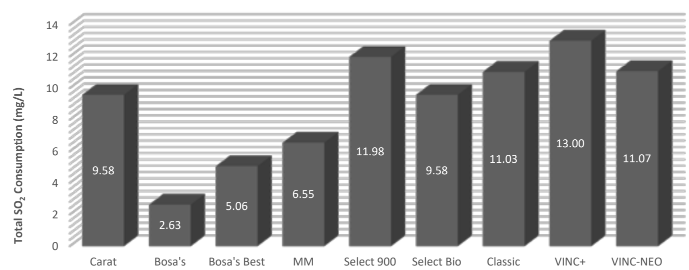

Figure 2: Total SO_2 consumption between T0 and T0+18 per closure

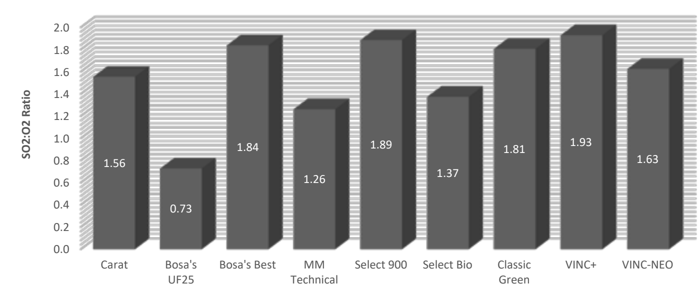

Figure 3: Calculated approximate SO2:O2 consumption ratio per closure

After the 18-month cellaring period, the wine with a Carat closure had the lowest FSO2 at 2.77 mg/L, a significant drop of more than 25 mg/L but an actual consumption of 9.58 mg/L of FSO2 based on TSO2 drop; the wine with a UF25 closure had the highest FSO2 at 12.76 mg/L, representing a drop of approximately 15 mg/L, and the lowest FSO2 consumption (2.63 mg/L). Bosagrape's Best and MM Technical closures had similar FSO2 at 7.62 and 7.91 mg/L, respectively, representing a drop of approximately 20 mg/L, and actual FSO2 consumptions of 5.06 mg/L and 6.55 mg/L. All synthetic closures had similar FSO2 and in the range 3.97–4.77 mg/L, representing a drop of approximately 23–24 mg/L; however, there were some small differences in FSO2 consumptions. The wine with a Select Bio closure had consumed 9.58 mg/L, identical to the Carat closure, then the Select 900, Classic Green and VINC^{NEO} with similar consumptions (11.98, 11.03, and 11.07 mg/L). The wine with a VINC^{+} closure had the highest FSO2 consumption (13.00 mg/L) though not significantly different from the latter three synthetic closures when factoring in instrumentation errors.

Since the amount of oxygen ingress during the cellaring period could not be measured, TPO measurements were used to determine an approximation of the total consumed oxygen (TCO) to obtain approximate SO2:O2 ratios for each wine and closure, and compare those with theoretical (stoichiometric) and literature-reported ratios. Ratios are calculated from the amount of FSO2 consumed divided by the difference in TPO between T0 and T0+18. The calculated ratios are in the range 0.7:1 to 1.9:1 with a mean of 1.6:1. The wine with the UF25 closure had the lowest ratio and the highest difference compared to the mean. These ratios are significantly lower than those reported in the literature, 2–2.5:1 (Diéval et al. 2013), 2.86:1 (Schneider 2019), and particularly that there is uncharacteristically significant binding in the wine in all bottles. Plausible explanations are that oxygen oxidizes ethanol to acetaldehyde, which would quickly bind free SO_2, or there are other SO_2 binders present in the wine. However, these results are contrary to the explanations reported by Waterhouse et al. (2016) in their study and which state that the reduced oxidation rates they observed affected not the free SO_2 as expected, but affected also bound SO_2, and that the results indicate that when low levels of free SO_2 (below 10 mg/L) were reached, the bound SO_2 started to dissociate, sustaining a minimal level of free SO_2 that reacted with oxidation products, with the result that bound SO_2 levels dropped more than free SO_2 levels did. The authors did however observe that there was also a loss of FSO2 not fully compensated by dissociation of bound SO_2.

Total Consumed Oxygen: Table 5 compares total consumed oxygen (TCO) per closure based on the HSO, DO and SO_2 analyses, approximations and assumptions described above, to TCO calculated from manufacturers' OTR data where available.

Based on SO2:O2 analysis, wine bottled with the UF25 closure consumed the least amount of oxygen over the 18-month cellaring period, followed by wines bottled with Bosagrape's Best and MM Technical closures. Wines botted with Carat and Select 900 closures consumed the same amount of oxygen, while wines bottled under Select 900, Classic Green, VINC^{+} and VIN^{NEO} closures consumed the most oxygen.

Based on manufacturers' OTR data, where available, measured TCO (7.70 mg/L) for the Select 900 was significantly below the calculated TCO (12.79 mg/L) based on OTR over 18 months (549 days), and similarly for the Classic Green (7.09 vs. 11.42 mg/L), while there was better correlation for the VINC^{NEO} closure (7.11 vs. 9.63 mg/L), and very good correlation for the VINC^{+} closure (8.35 vs. 8.98 mg/L).

Table 5: TCO comparisons based on SO2:O2 ratio analysis and on manufacturers' OTR data

CLOSURE	TCO (mg/L) based on SO2:O2	TCO (mg/L) based on OTR data
Carat	6.16	N/A
Bosa's UF25	1.69	N/A
Bosa's Best	3.25	N/A
MM Technical	4.21	N/A
Select 900	7.70	12.79
Select Bio	6.16	N/A
Classic Green	7.09	11.42
VINC+	8.35	8.98
VINC-NEO	7.11	9.63

Color. Figure 4 illustrates absorbance measurements for the base wine and wine under each closure comparing absorbance at 420 nm (A_{420}) at T0 and T0+18.

All wines measured higher absorbances at 420 nm consistent with color evolution during aging. Although absorbances varied with good correlation to total consumed oxygen (TCO), there were no significant differences. The wine with the UF25 closure had the lowest absorbance followed by Bosagrape's Best and MM Technical closures. The Gültig Carat as well as synthetic closures all had similar absorbances.

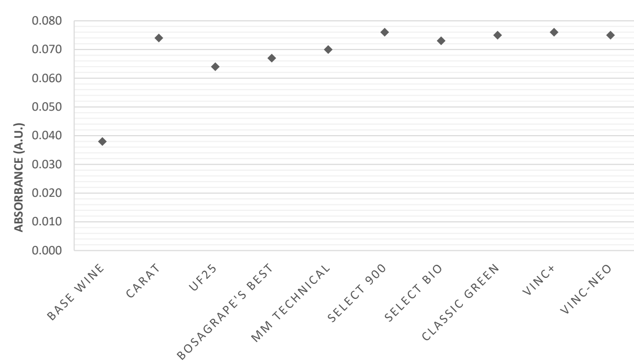

Figure 4: Absorbance measurements for the base wine and wine under each closure comparing A_{420} at T0 and T0+18

Taste. A non-blind tasting after 18 months did not reveal any differences in aromas or taste; none demonstrated flaws.

Conclusions

This study demonstrates that, although closures performed differently from an oxygen ingress perspective based on oxygen and total SO_2 consumption analysis as well as color analysis, all closures are deemed appropriate for cellaring a fruity-style Chardonnay for up to 18 months. Better bottling equipment and process, including inerting headspace, can extend shelf life significantly.

Closures were found to contain or transfer varying and, in some cases, significant amounts of oxygen into the headspace upon compression and insertion into bottles, with natural cork closures transferring the least amount of oxygen.

Only the Carat, MM Technical and VINC^{NEO} closures achieved zero headspace oxygen, from which we conclude that these closures have either very low OTRs or that dissolved oxygen was being consumed at a faster rate, although the VINC^{NEO} closure took a while longer to reach zero dissolved oxygen. All other closures were very close to zero headspace oxygen after 18 months of cellaring and would likely reach zero within the next month or two.

Total SO_2 consumption analysis demonstrated that consumption varied under different closures. Consumption was lowest in wine with the UF25 closure and highest in the wine with the VINC^{+} closure; however, this impact is due to total package oxygen (TPO) at bottling, which was highly impacted by the amount of oxygen transferred from closure material, and not necessarily from oxygen ingress through and around closures.

Now combining oxygen and SO_2 analyses, wine under the UF25 closure had a very low and lowest SO2:O2 ratio, and the Bosagrape's Best, Select 900, Classic Green, and VINC+ closures having the highest ratios.

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