

Wine Closures

Using membrane technology to optimise closure performance

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Abstract

Membrane technology can control the permeation of oxygen into wine. It is used by the wine industry in pre-bottling micro-oxygenation techniques and more recently it is being used post-bottling in membrane-based cork closures. These membrane cork closures are currently being used by a number of leading premium and large wineries. Membrane technology can also be used to inhibit taint and flavour transmission into wine. This paper extends the research reported by Tran *et al.* (2007) and evaluates the current scientific data regarding post-bottling oxygen permeation into wine. This work demonstrates that a wine sealed with different closures can exhibit characters ranging from predominantly oxidised with high permeance synthetic cork to predominantly reduced with low permeance screw cap. The research demonstrates that membrane cork technology works by effectively controlling oxygen ingress thereby inhibiting oxidation, similar to screw cap, while at the same time also inhibiting the reduced character that can develop in wine sealed with the screw cap closure. This work demonstrated that dimethyl disulfide (DMDS) (which is one indicator of 'reduced' wine) can be present in screw cap wine when it is not found in wine from other more permeable closures. This work indicates that membrane technology applied to cork will allow winemakers to find the optimum post bottling oxygen ingress and reduce taint and flavour transmission.

Introduction

The most recent scientific studies confirm what has been generally accepted for the last 400 years: that oxygen is intimately involved in the ageing process of bottled wine. The research has identified that too much oxygen can prematurely oxidise wine, small increased amounts of oxygen through the closure can accelerate wine development/maturation, and that too little oxygen can result in the development of reduced characters in wine [Skouroumounis, 2005; Hart, 2005]. Leading researchers have recently acknowledged that oxygen ingress is actually one of the major factors determining wine development in the bottle and that a complete oxygen barrier is not ideal for wine [Goode, 2006a; Allen, 2006]. It therefore makes sense that membrane technology that can control the transmission of oxygen can play a fundamental role in controlling wine development.

This paper reports independent testing of membrane cork by the Australian Wine Research Institute (AWRI) as well as correlates other published research data on bottled wine development to the characteristic oxygen permeance of the different closure types. The

application of membrane technology to natural cork originated from Australian research during the 1990's. After commercial release in 2004 it became one of the fastest growing new closure technologies, growing to 30 million bottles per annum within 18 months. It is now rapidly spreading into the largest wine producing regions of the world including France and Italy. Membrane technology for cork was actually developed to inhibit taint and flavour transmission into wine, however, it was discovered during this work, that the oxygen transmission properties of the membrane could play an important role in optimising wine development and improve closure performance.

Existing data on post-bottling development of reduction and oxidation

There have been a number of published scientific trials conducted where a range of closures have been compared. In the trials reviewed, a trained tasting panel had been used to conduct a blind evaluation of the sensory characteristics of the wine. In each tasting, both reduced and oxidised aromas and flavours were evaluated.

Several sensory properties have been identified as relating to the 'reduced' sensory character in wine. These sensory properties include rubber, struck flint, burnt match and cooked cabbage [Ribereau-Gayon, 2000; White, 2005]. Low level reduction can present other sensory characteristics like garlic, mineral, leading to bitterness at the back of the pallet which may dull the fruit character [White, 2005; Ribereau-Gayon, 2000; Goode, 2006]. Anecdotal comments at tastings indicate that low level reduction in red wine may also limit the fullness of the tannin feel in the mouth and mask the subtler oak characteristics. Mercaptans have previously been linked to reduced character attributes in wine [Ribereau-Gayon, 2000].

Permeability data corresponding to the closures types used in these trials is now available [Mills, 2005; Hart, 2005; Godden, 2005]. The sensory panel scores for attributes linked to reduced character (such as rubber and struck flint) and oxidised character can be correlated to the relative characteristic oxygen permeance of each style of closure used. This is done by the current authors in Fig. 1, Fig. 2, and Fig. 3 for Shiraz Cabernet, Chardonnay and Riesling. Further data on Semillon is given in Fig. 6 which includes the results of a membrane cork closure.

The reduced and oxidised sensory scores show what appears to be a characteristic saddle curve where the reduced and oxidised character is minimised at a particular closure permeance. In general the saddle appears to be largely independent of the wine styles tested. Fig. 1, Fig. 2, Fig. 3, and Fig. 6 are experimental evidence of ▶

wine closures

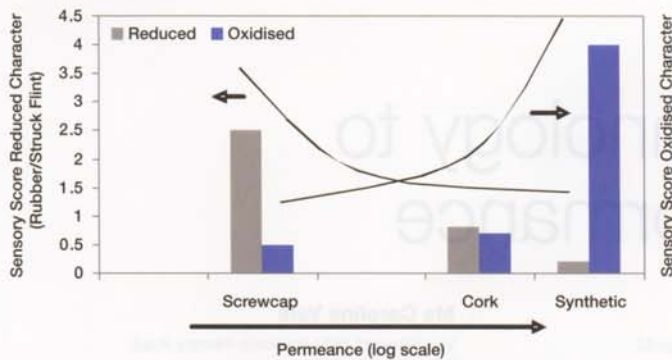


Fig. 1. Reduced and oxidised sensory characteristics versus the relative characteristic closure Permeance in a Shiraz-Cabernet Sauvignon Blend (1996 Penfolds Bin 389), 7 years post bottling. (Scale 0-9 where 1 is barely perceptible) [Hart *et al.* 2005]

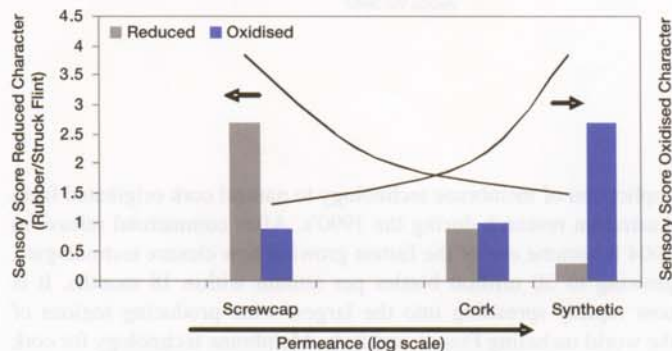


Fig. 2. Reduced and oxidised sensory characteristics versus relative characteristic closure permeance in a Chardonnay (1999 Padthaway South Australia), 3 years post bottling. (Scale 0-9 where 1 is barely perceptible) [Skouroumounis *et al.* 2005]

the "optimum closure oxygen transmission curve" shown at the 2005 Rutherglen Wine Show Seminar [Gibson, 2005] and the "closure zone curve" presented in the book "Wine Bottle Closures" [Goode, 2006b].

Independent testing of membrane cork

The AWRI conducted its first wine bottle closure trial in May 1999. Due to demand by closure producers, a second trial was set up on a commercial basis, and closure producers were invited to submit their closure together with a fee. In return for this fee, the closure producers were presented data on the performance of their closure, together with three reference closures, being screw cap, reference 2 cork and reference 3 cork.

A membrane cork closure developed by ProCork Pty Ltd which uses natural cork combined with membrane technology was

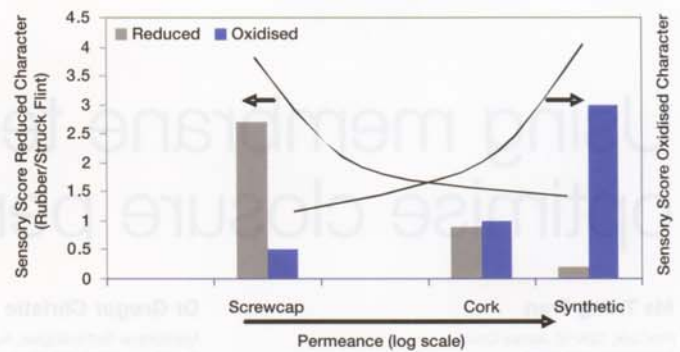


Fig. 3. Reduced and oxidised sensory characteristics versus relative characteristic closure permeance in a Riesling (1999 Eden Valley South Australia), 3 years post bottling. (Scale 0-9 where 1 is barely perceptible) [Skouroumounis *et al.* 2005]

submitted to the AWRI commercial closure trial. The membrane consists of a series of layers which are able to regulate oxygen, reduce taint entering the wine and reduce flavour scalping from the wine. When applied to the cork end, this membrane acts as a barrier to the wine [Tran, 2005].

The AWRI Commercial Closure trial commenced in September 2002. The wine was a premium 2002 Semillon from the Clare Valley, the same wine used in the 1999 trial. It was made under normal winemaking conditions. The wine was bottled at Vinpac International, Angaston (ISO 90001:2000 certified and ISO 17025:1999 certified).

The detailed methodology and results are given in a poster summary published in the Conference Proceedings of the 12th Australian Wine Industry Technical Conference 2004 [Tran, 2005].

Wine development under membrane cork

AWRI carried out sensory and chemical analysis on the wine from the trial (described above) on a 6 monthly basis. The free SO₂ data at 24 months is given below (Fig. 4) for the membrane cork closure (ProCork), Diam, screw cap and cork of two grades and lengths. It can be seen that the membrane technology when applied to cork has the highest average level of free SO₂ as well as a high consistency with the lowest standard deviation of 4.0%. As a comparison, screw cap, which is also noted for its consistency, had a standard deviation of 4.3%. This indicates a very tight and consistent seal with the membrane cork closure.

Table 1 gives methyl mercaptan and dimethyl disulfide (DMDS) concentration measured by ETS Laboratories, St Helena, California. This table also details the corresponding AWRI sensory scores of the 24 month sensory data. The sensory data highlights the struck flint/rubber character is more obvious in the screw cap, than any

Table 1. 24 month: sulfide concentration (ETS Laboratories) and some sensory data (average of 4 replicates, standard deviation in brackets) AWRI Commercial Closure Trial [Eichinger, 2004a; Eichinger, 2004b]

	ETS analysis		AROMA (sensory)				PALATE (sensory)			
	Dimethyl Disulfide (ng/l)	Methyl Mercaptan (ng/l)	Struck flint/rubber	oxidised	Floral	Citrus	Overall Fruit	Honey	Overall fruit flavour	Fruit flavour persistence
Membrane Cork Closure (ProCork)	nd	nd	0.7 (0.3)	0.1 (0.1)	2.9 (0.4)	4.6 (0.3)	5.1 (0.5)	0.9 (0.2)	5.4 (0.2)	5.2 (0.4)
**Diam P0	nm	nm	0.1 (0.1)	0.3 (0.3)	2.6 (0.3)	4.1 (0.2)	4.4 (0.5)	1.5 (0.4)	4.9 (0.3)	4.9 (0.3)
**Diam P1	nm	nm	0.2 (0.1)	0.3 (0.0)	2.8 (0.4)	4.1 (0.2)	4.3 (0.4)	1.6 (0.2)	4.8 (0.0)	4.7 (0.4)
Screw cap	1100	600	1.7 (0.1)	0.2 (0.2)	2.7 (0.3)	4.3 (0.3)	4.5 (0.5)	1.0 (0.3)	5.5 (0.2)	5.1 (0.2)
Cork Ref 2	nd	nd	0.3 (0.2)	0.3 (0.1)	3.0 (0.3)	4.2 (0.3)	4.9 (0.3)	1.5 (0.3)	5.1 (0.3)	5.1 (0.2)
Cork Ref 3	nd	nd	0.1 (0.2)	1.0 (0.8)	2.3 (0.4)	3.6 (0.5)	4.0 (0.5)	1.5 (0.1)	4.6 (0.4)	4.5 (0.4)

*nd = Not detected, nm = not measured

**The Oenoc UK web site and a previous publication [Banister, 2005] both indicate the Altec closures in the report [Eichinger, 2004b] are Diam closures. The data for the more permeable Diam P2 is not shown here. (Sensory scale 0-9 where 1 is barely perceptible)

wine closures

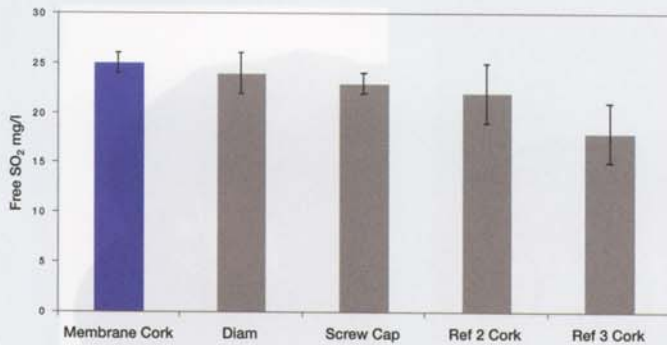


Fig. 4. Average free SO₂ and standard deviation (12 bottles tested) - AWRI commercial closure trial [Eichinger, 2004; Banister, 2005]

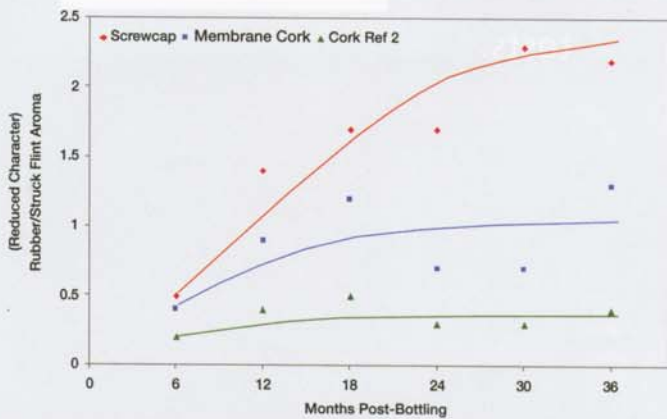


Fig. 5. Reduced character development over time AWRI commercial closure trial [Buick, 2003; Eichinger, 2003, 2004, 2004b, 2005; Taylor, 2005]

other closure. This data was supported by the ETS Laboratories results which showed DMDS and methyl mercaptan are present in the screw cap wine.

Methyl mercaptan and DMDS are some of the indicators of reduced wine [Ribereau-Gayon, 2000] and struck flint/rubber is also one of a number of sensory attributes that are considered to indicate a wine is reduced [Ribereau-Gayon, 2000; White, 2005].

The reduced character was not observed directly after bottling. However, as the wine developed in the bottle so did the reduced character and presumably the DMDS and methyl mercaptan. This post bottling reduced character developed to be more evident in the screw cap closure as shown in Fig. 5.

The results in Fig. 5 indicate that reduced compounds can develop after bottling with a screw cap closure where they wouldn't have if a more permeable closure was used. To further evaluate the implication of this, three bottles of older screw cap Riesling were purchased from retail outlets in 2006. These bottles were sent to ETS Laboratories, and the results are shown on Table 2. There can be no way of identifying where the DMDS came from, however, the data in Fig. 5 indicates it is possible that it occurred after bottling and that a more permeable closure could have reduced its level.

The 24 month sensory data showed the membrane cork closure scored the highest average score for citrus and overall fruit aromas when compared to Diam, and the reference screw cap and cork closures. Some of the sensory data from Table 1 is plotted in Fig. 6 demonstrating the membrane cork closure sits in the optimum region

Table 2. DMDS levels in older screw cap wines.

Wine	Years since bottling	DMDS (ng/L)
A	4	1900
B	4	1200
C	3	1700

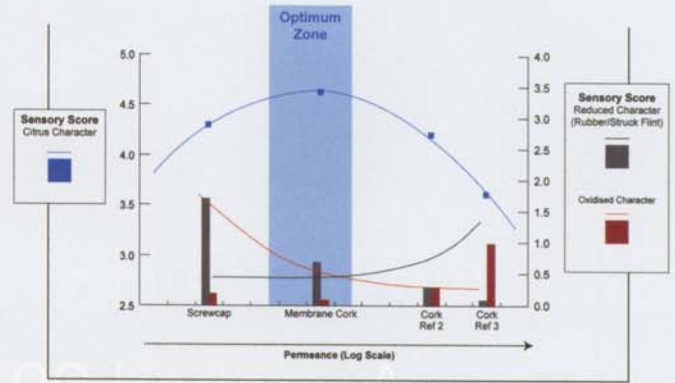


Fig. 6. Reduced, oxidised and citrus sensory attributes (Scale 0-9 where 1 is barely perceptible) plotted against the relative characteristic closure permeance (Log Scale): AWRI Commercial Closure Trial (2002) - 24 months for screw cap, membrane cork, cork Ref 2 - 44mm, cork Ref 3 - 38mm

where oxidised character is minimised, reduced character has not become too intense and the citrus sensory attribute is maximised. It has a significantly lower reduced character than screw cap and a lower oxidised character than cork. The membrane cork has the highest average score for citrus aromas when compared to the other closures. The determination of the bottom of the reduction/oxidation saddle is subjective. It can be interpreted, that the optimum zone is where the fruit attributes are maximised or plateau, oxidation is low and reduced characters are only barely perceptible. It is within this optimum zone that the membrane cork closure performs. The membrane cork closure benefits from lower oxidation without the quality loss caused by the higher reduction.

Conclusion

The trials above show that there is an optimum closure permeability that allows wine to age without excessive development of post-bottling reduction or oxidation.

Correlation of tests carried out by ETS Laboratories to the sensory characters have shown that a build up of dimethyl disulfide (DMDS) and methyl mercaptan is one symptom of post-bottling reduction which appears to be more evident in the screw cap closure compared to any other closures reported here.

The independent closure trial showed that the membrane cork closure has the optimum oxygen permeability for the wine tested. The membrane technology has retained high levels of free SO₂ and fruit without the quality loss associated with higher levels of reduced characters seen in the screw cap control or oxidised characters seen in the cork control.

The membrane technology used in the commercial membrane cork closure tested will allow winemakers to optimise oxygen permeation post bottling and obtain bottle development uncompromised by oxidation or reduction.

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** Participation in and reporting of the independent AWRI "Commercial Closure Trial" does not imply an endorsement by AWRI. The data used in this article should be read in conjunction with the full AWRI report*

National Water Plan: is this the answer?

Early last month, Prime Minister John Howard unveiled a \$10 billion plan to take control of Australia's water resources away from State governments.

The proposal in fact takes a very broad view of water use in Australia and not only includes a massive investment in irrigation infrastructure, but also plans for water saving measures for the farming community and new exploration of water development in Australia's north.

However, central to the plan, and the cause of the controversy surrounding it, is the Prime Minister's bid to take control of the Murray-Darling river system from State governments.

"This 10-point plan opens a new chapter of national water management in Australia," Prime Minister Howard said on announcing the plan. "It is a large but prudent investment, especially given the importance to Australia of the Murray-Darling Basin and the scale of the water crisis that confronts it."

Premier problem

At the time of going to press, New South Wales is the only State involved to have pledged to relinquish their control of the Murray-Darling, with Mike Rann in South Australia, Steve Bracks in Victoria and Peter Beattie in Queensland making their opposition to the plan felt. The Victorian Premier has demanded that his State's interests are met before talks on a federal water takeover can begin, and he pointed out, "There's no new water created by the proposals."

New South Wales Premier Morris Iemma is in favour of a national approach and will cooperate fully with Canberra.

MDA support

Meanwhile the Murray Darling Association (MDA) is also calling for cooperation with the Prime Minister's plan. MDA chairman Cr Bill Gorman of Corowa Shire has also welcomed the Prime Minister's proposal.

MDA General manager, Ray Najar, told *Grapegrower & Winemaker*, "We do believe that the plan has the capacity to provide better security to all aspects of water use in irrigation, urban and environmental components, simply by dealing with compensation to farmers with cuts in their allocations – the over allocations in NSW in particular – so that this money will mostly go back into on-farm investment in best practice, water use efficiency upgrades."

"The MDA does support the plan in principle, subject to following conditions;

- that the MDBC is given the authority to carry out its full responsibility of managing the infrastructure programs necessary under the PM's \$6billion plan.
- that the PM and the Premiers agree to a joint team of practitioners (not all bureaucrats and politicians) to manage the water resource allocations, and set up a uniform strategy on licences and water trading rules, across the entire basin."

Critical issues

The newly-created Drought Management Taskforce also responded to the plan saying, in a statement to *Grapegrower & Winemaker*, "It is widely accepted that the current approach to managing the Murray-Darling system could be improved, and the taskforce supports appropriate initiatives for improvement. In addition, the taskforce supports appropriate initiatives to address the following critical issues: water over-allocation, water losses through both the supply system and on-farm use, and greater efficiencies in the delivery and application of water.

"Addressing these issues will lead to healthier river systems through greater flows and ultimately benefit Australia's winemakers and grapegrowers as well as other agricultural industries."

Discussions are ongoing, see the April issue of Grapegrower & Winemaker for an update on the National Water Plan.



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