# **Technical notes**

# The impact of oak additions on the colour and phenolic profile of red wines

#### Introduction

The use of oak in winemaking has a long history and is integral to the production of many wine styles. Over recent years, the production of oak barrels has become more technologically advanced; at the same time, the adoption of oak adjuncts in winemaking has increased. A number of questions still remain, however, on the role that oak products can play in enhancing red wine colour and phenolics.

The AWRI has undertaken two separate studies on the impact of oak additions on the colour and phenolic profile of red wines:

- 1. To determine if the addition of oak chips or oak powder at various rates during red wine fermentation has any short to medium term impact on the development of colour and phenolic profile in red wine.
- 2. To determine the relative impact of new and used oak barrels on the evolution of phenolics and colour in red wine over the short to medium term.

## Study 1: Evaluating the impact of oak addition during ferment

The extraction of phenolic and volatile compounds during barrel maturation and their impact on the sensory attributes of red wines has been well characterised. However, the impact of oak on the colour and phenolic profile of red wines over time is less widely understood. Whilst we know that the majority of tannins in wine are grape derived and that oak-derived tannins exist in much smaller amounts, the latter tend to be more chemically complex and little is known about the interactions between these different tannin species over time.

Whilst the use of oak additions during wine maturation is commonplace, the use of oak additions during fermentation is a somewhat novel concept and there is limited information on its effect on resultant wine style (Rodriguez-Bencomo et al. 2010 and Gomez-Garcia-Carpintero et al. 2012).

#### Methodology

A small-scale experiment was carried out to investigate the influence of different additions of oak adjuncts on red wine properties. Shiraz grapes were fermented in coffee plungers (200 g/plunger) with oak chip additions of 1 g/kg, 3 g/kg and 5 g/kg grapes. A sample of the oak chips was ground to a powder and an addition of this powder at 1 g/kg was included as

an additional treatment. To investigate the possible influence of oxygen present in the oak chips, a treatment of deoxygenated chips (3 g/kg grapes) was also included. All five treatments and control (with no additions) were fermented in triplicate at 26°C using the same yeast. After primary fermentation was completed, the ferments were racked to remove the oak and gross solids, cold settled and stored at ambient (~21°C) conditions. The resulting wines were analysed after 1 month, 3 months and 7 months for free anthocyanins, total pigments, total phenolics, total tannins, non-bleachable pigments, chemical age and hue, using the rapid spectral method utilised by the AWRI WineCloud<sup>™</sup> (AWRI publication #1436).

#### Results

Significant variability among replicates was observed, most likely due to the small scale of the ferments and the corresponding variability in juice volume. The initial results one month after fermentation showed very little difference between treatments with significant differences only becoming apparent at later time points. Results collected after four months were highly variable, probably reflecting the transition of the wine components to their equilibrium state, which can occur at slightly different rates for each replicate.

Results collected at seven months post-ferment showed that the biggest changes observed between 1 month and 7 months post-ferment were increases in % non-bleachable pigments and chemical age indices, and decreases in anthocyanins, total pigments, tannins and colour density over the six-month period (Figure 1). This is to be expected, as the available colour components bind with the available tannins to form stable pigmented tannins.

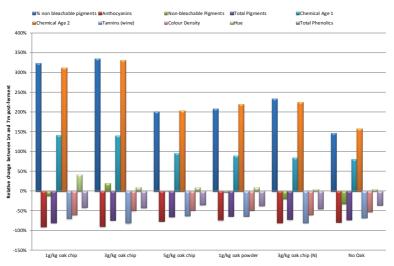


Figure 1. Relative change in colour and phenolic components between one and seven months post-ferment for all five treatments relative to the control treatment (no oak).

Figure 2 highlights the comparison between different treatments after seven months postferment. Key trends observed were that:

- Oak chip additions of 1 g and 3 g showed decreased levels of free anthocyanins and total pigments in relation to the control, whilst the oak powder addition resulted in comparatively high levels of both.
- Increased colour density was seen for all treatments in relation to the control, most noticeably for the 3 g and 5 g additions, as well as the oak powder addition.
- Hue levels were highest for the 1 g oak chip addition, with the 1 g powder addition and 3 g deoxygenated addition exhibiting the lowest levels.
- Increased chemical age was observed for all treatments, relative to the control, with the 1 g and 3 g additions showing the biggest increases.
- All treatments showed higher proportions of non-bleachable pigments than the control, particularly the 1 g and 3 g oak chip additions.
- Higher tannin concentration was seen only for the 5 g treatment and oak powder treatments, whilst the 3 g deoxygenated addition showed decreased tannin levels, relative to the control.
- The oak chip additions of 1 g and 3 g and the 3 g deoxygenated addition resulted in lower total phenolics than the control.

Overall, samples treated with deoxygenated oak chips were more similar to the control (no oak) than to the samples with standard oak chip additions. The entrained oxygen that would normally be present in oak additions appears to play a significant role in achieving colour stability in the form of non-bleachable pigments and in retaining tannins between 1 month and 7 months post-ferment.

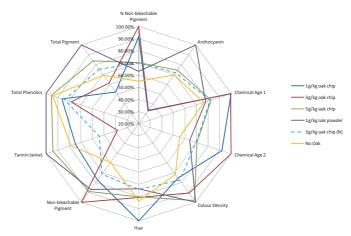


Figure 2. Comparison of colour and phenolic components after seven months post-ferment for all five treatments relative to the control treatment (no oak).

The wines treated with oak powder exhibited different characteristics from those treated with oak chips (at all dose levels), with higher free anthocyanins, total pigments, colour density, tannins and total phenolics, but lower non-bleachable pigments, hue and chemical age. This mirrors trends seen in previous studies (AWRI publication #961) that reported increased extraction of oak-derived volatile compounds with oak powder additions, in relation to oak chips.

As the amount of chips added (and contact surface area) increases, a tendency towards increased colour is seen, with higher pigment levels and higher colour density reflecting a higher degree of monomeric anthocyanins. The degree of stable colour (as measured through the proportion of non-bleachable pigments and chemical age indices) achieved after seven months tends to decrease with increasing oak addition.

Total phenolics and tannin levels appear to increase as the amount of chips added increases. These trends are exacerbated with the 1 g oak powder addition, with levels appearing higher than with the 5 g oak chip addition, probably due to the higher contact surface area.

# Study 2: Evaluating the impact of new vs old oak

The impact of oak source, seasoning process and toasting methods have been well documented over recent years, but the age of the barrel and its impact on red wine maturation remains an area of active study. Recent studies have highlighted that the pool of oak extractives that barrels contain is finite and that the quantity of these compounds and the rate of extraction diminish with reuse (Cerdán et al. 2002).

## Methodology

A block of Hunter Valley Shiraz was selected to provide the total volume requirement for the trial (approx. 4.5 T) and fermented as a single homogenous batch. The must was pressed and transferred to:

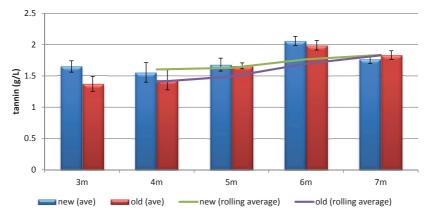
- Six new Demptos TG (tight grain) medium toast barrels, supplied by Classic Oak
- Six 4-year-old Demptos TG (tight grain) medium toast barrels (with an identical usage history), supplied by Brokenwood Wines
- Stainless steel kegs, to be used as the control wine and to be used as the source of topping wine

The topping regime was kept the same across all twelve barrels during storage. Measurements of tannins, phenolics and colour (total pigments) were taken at the following points, again using the AWRI WineCloud<sup>™</sup> method:

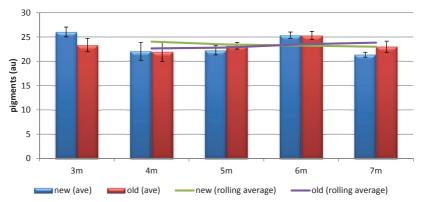
- Three times as part of maturity testing prior to harvest
- Daily during fermentation
- After pressing
- Once during MLF (dependent on duration)
- At post MLF sulfur addition
- Monthly during maturation over a 7-month period

#### Results

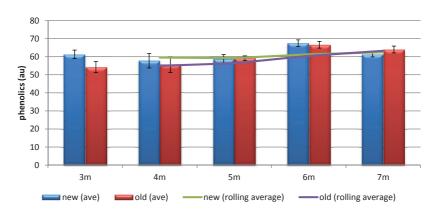
The average tannin, colour (pigments) and phenolics levels observed in the old and new oak barrels appear to be closely matched throughout maturation (see Figures 3, 4 and 5), with none of the wine attributes appearing to be particularly influenced by the age of the oak



**Figure 3.** Average tannin levels in the wines across six replicates for old and new oak between three and seven months post-ferment. A rolling average of tannin levels for both new and old oak treatments is also shown.



**Figure 4.** Average pigment levels in the wines across six replicates for old and new oak between three and seven months post-ferment. A rolling average of pigment levels for both new and old oak treatments is also shown.



**Figure 5.** Average phenolics levels in the wines across six replicates for old and new oak between three and seven months post-ferment. A rolling average of phenolic levels for both new and old oak treatments is also shown.

barrels after seven months post-ferment. Both data sets show (on average) a modest increase over time for both phenolics and tannins, with total colour (pigment) levels remaining relatively consistent during maturation, as expected. There appears to be little difference in the variability of data obtained from the old and new oak barrels, with both data sets showing comparable standard deviations across the six replicates.

Initial data collected after three months post-ferment, when the wines had received approximately six weeks oak contact, suggests that the new oak barrels have an immediate impact on the colour and phenolic profile. During the early stages of (new) barrel use, higher proportions of the wine will be absorbed into the surface of the wood. This is likely to be accompanied by increased exposure to oxygen entrained in the oak matrix itself; a common driver for increased consumption of sulfur dioxide (SO<sub>2</sub>) in wines matured in new oak barrels (AWRI publication #1554).

Tannins, phenolics and pigment levels are all significantly higher in the new oak treatments at three months post-ferment. However, these apparent impacts have disappeared by the time the wines have been in barrel for six months, suggesting that a 'conditioning' period is experienced by the wines matured in new oak barrels. Studies carried out by Cerdán and Ancin-Azpilicueta (2006) and del Alamo-Sanza et al (2000) have suggested that there is a difference in the concentration of oak-extracted volatiles between new and used oak for short-term ageing (e.g. 6–9 months), but that this impact is less evident for longer-term ageing (12–18 months).

These initial differences are reflected in the principal component analysis (PCA) in Figure 6. Distinct clustering of data is seen for the old and new oak treatments at three months post-ferment, but these differences quickly become less apparent over time.

Colour (pigment) levels seen after seven months are comparable for the new and old oak treatments, even though the new oak treatment appeared to initially enhance colour levels, after approximately one month in barrel (three month time point). The period between three and four months coincides with an apparent drop in colour for the wines matured in the new oak barrels. This suggests that monomeric pigments are influencing this short-term effect and that colour components are stabilised earlier in the new barrels than they are in the old barrels. Over time, the free anthocyanin (bleachable) component of the total pigments measured in the new and old oak treatments are likely to decrease as they react with the tannins present to form stable pigmented tannins.

Perez-Prieto et al. (2003) showed that colour evolution is faster in small, new oak barrels than in large, used barrels. This evolution towards more stable red colour appeared to be confirmed by a higher content of polymeric anthocyanins, hue and lower percentage of red colour with new oak.

Feuillat et al. (2000) found that the redox potential is higher in new oak barrels than with used barrels. As anthocyanin polymerisation is enhanced by the presence of oxygen, we would expect that wines stored in new oak barrels exhibit decreases in free anthocyanins and

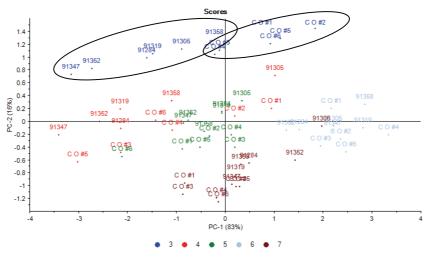


Figure 6. Principal component analysis of colour and phenolic data between three and seven months post-ferment for wines stored in old and new oak barrels.

increased polymeric anthocyanins in comparison with wines stored in old oak. However, it is unclear whether these anthocyanins are interacting with the grape-derived tannins present in the wine or with the reactive components released from wood, such as ellagitannins (Gambuti et al. 2010).

Over the five months that the wines were in barrel, tannin and phenolic levels appeared to increase, as the oak contact time increased, suggesting that the increase may be due to extraction of oak-derived tannins from the barrels. Although the rapid spectral measurement method employed is based on grape-derived tannin compounds, it is likely that it will be influenced by the presence of oak-derived tannin compounds. The comparative degree of sensitivity of the method to grape and oak-derived tannins is not known at this stage, but it is likely that the spectral response will be different, due to different extinction coefficients. In this case, the level of oak-derived tannins is likely to be small, in comparison to the level of grape-derived tannins.

#### Conclusions

These studies have shown that the colour and phenolic profile in red wines can be influenced by different oak treatments, both during fermentation and during maturation.

It appears that the amount of oak added during ferment influences colour development and stabilisation, with the higher doses employed in this study resulting in higher levels of monomeric pigments (anthocyanins) and comparatively lower pigmented tannins. Oak powder additions have a bigger influence than oak chips (on a weight basis), due to the higher contact surface area, suggesting a pseudo-fining effect during the fermentation process. Results observed with the deoxygenated oak chips show that oxygen typically entrained in oak adjuncts may play a significant role in the profile of the final wine when additions are made during fermentation; the wines fermented with deoxygenated oak chips showed lower colour, tannin and phenolic levels than the other oak treatments.

Phenolic and tannin levels increase over time while red wine is matured in barrels. This increase is likely to be directly due to the extraction of oak-derived tannin compounds and their role in stabilising colour components through formation of pigmented tannins. New oak barrels appear to have an immediate impact on the colour and phenolic profile of the red wines, with tannins, phenolics and pigment levels significantly higher than those found in the old oak barrels after six weeks' maturation. It appears that the colour components are stabilised earlier in the new barrels and that a 'conditioning' period is required for the wines. After six months' maturation, however, the wines exhibited comparable levels of colour, tannins and phenolics.

#### References

- AWRI publication #961. Campbell, J.I., Pollnitz, A.P., Sefton, M.A., Herderich, M.J., Pretorius, I.S. (2006) Factors affecting the influence of oak chips on wine flavour. Aust. N.Z. Wine Ind. J. 21(4): 38–42.
- AWRI publication #1436. Dambergs, R.G., Mercurio, M.D., Kassara, S., Cozzolino, D., Smith, P.A. (2012) Rapid measurement of methyl cellulose precipitable tannins using ultraviolet spectroscopy with chemometrics: application to red wine and inter-laboratory calibration transfer. Appl. Spectrosc. 66(6): 656–664.
- AWRI publication #1554. Holdstock, M. (2013) Ask the AWRI: Snapshot of oak-related queries. Aust. N.Z. Grapegrower Winemaker 595: 68.
- del Alamo-Sanza, M., Bernal-Yague, J.L. Gomez-Cordoves de la Vega, C. (2000) Influence of the type of barrels and oak variety on the aging of a Ribera del Duero red wine. Food Sci. Technol. Int. 6: 483–493.
- Cerdán, T.G., Mozaz, S.R., Azpilicueta, C.A. (2002) Volatile composition of aged wine in used barrels of French oak and of American oak. Food Res. Int. 35: 603–610.
- Cerdán, T.G., Ancin-Azpilicueta, C. (2006) Effect of oak barrel type on the volatile composition of wine: storage time optimization. LWT 39(3): 199–205.
- Feuillat F, Naudin R., Peyron D. (2000) La crianza de los vinos en madera: experimentaciones en Bourgogne, Fundamentos de Enología. Dunod, Paris, pp 626-640.
- Gambuti, A., Capuano, R., Lisanti, M.T., Strollo, D., Moio, L. (2010) Effect of aging in new oak, one-year-used oak, chestnut barrels and bottle on color, phenolics and gustative profile of three monovarietal red wines. Eur. Food Res. Technol. 231(3): 455–465.
- Gomez-Garcia-Carpintero, E., Gomez-Gallego, M.A., Sanchez-Palomo, E., Gonzalez-Vinas, M.A. (2012) Impact of alternative technique to ageing using oak chips in alcoholic or in malolactic fermentation on volatile and sensory composition of red wines. Food Chem. 134(2): 851–863.
- Perez-Prieto, L.J., de la Hera-Orts, M.L., López-Roca, J.M., Fernandez-Fernandez, J.I., Gómez-Plaza, E. (2003) Oakmatured wines: influence of the characteristics of the barrel on wine colour and sensory characteristics. J. Sci. Food Agric. 83: 1445–1450.
- Rodriguez-Bencomo, J. J., Ortega-Heras, M., Perez-Magarino, S. (2010) Effect of alternative techniques to ageing on lees and use of non-toasted oak chips in alcoholic fermentation on the aromatic composition of red wine. Eur. Food Res. Technol. 230(3): 485–496.

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