Alternatives to Oak Barrel Maturation: Influence on Composition, Sensory Properties and Consumer Acceptance of Wine

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Abstract

The aim of this research was to investigate the use of oak alternatives by the wine industry, and specifically, to explore their influence on the composition, sensory properties, and consumer acceptability of wine. A detailed online consumer questionnaire was administered nationally to wine consumers to explore their knowledge of and attitudes towards the use of oak during winemaking. Within the sample population surveyed (n=1015), four distinct consumer segments were identified, each with significantly different attitudes towards the use of oak alternatives for wine maturation. A segment of more knowledgeable consumers, who appreciate and value traditional barrel maturation, held slightly negative views of wines produced with oak alternatives. However, a second cluster comprising less knowledgeable consumers were accepting of the use of oak alternatives, provided wine quality was not affected. The results from this study have advanced our understanding of consumer attitudes towards innovative oak technologies and provide justification for the use of oak alternatives by winemakers, who can now tailor their wines to better meet the expectations of specific segments of their target market.

A maturation trial was subsequently established to examine the effect of different storage vessels on wine composition and sensory properties. The specific objective of the trial was to compare vessels comprising three different panel types, i.e. stainless steel, plastic and oak wood panels, and the potential for each to produce wines of comparable composition and quality to those aged traditionally in barrels. The outcomes of the 12 month trial demonstrated that the different storage vessels each imparted oak characters to wine and therefore afford winemakers an alternative method for the oak maturation of wine. In a second maturation trial, traditional and alternative oak maturation regimes were employed to age Cabernet Sauvignon wines and the effect of each treatment on the composition, sensory properties and consumer acceptance of wine investigated. Acceptability scores from 116 consumers revealed no significant differences in consumers' overall liking of each wine, but

segmentation based on individual liking scores identified three distinct clusters comprising consumers with significantly different wine preferences. Multivariate data analysis revealed the sensory attributes driving wine preference for each consumer segment. These results further justify wine producers' use of alternative oak maturation regimes to reduce production costs and achieve wine styles that appeal to different segments of the consumer market.

To gain a holistic view of consumer preferences for oak attributes in wine, a third consumer study was undertaken to investigate the effect of key oak aromas on consumer emotions. Consumers (n=116) were asked to rate their liking of eight oak-derived aromas and to then consider how different oak aromas make them feel. Consumers scored each aroma favourably, but spice and chocolate were liked most, and smoky and coconut aromas were liked least. Segmentation of consumer liking scores revealed three segments which differed in their liking of and emotional response to different oak aromas, but differences were subtle. This study showed oak aromas generally elicited positive emotions, but broader classes of aromas, e.g. oak derived aromas versus fruit aromas (e.g. citrus, berry or tropical fruit), may elicit stronger emotional responses than individual oak aromas.

An additional study reporting the convenient, low-cost preparation of isotopically labelled volatile wine phenols, using microwave-assisted deuterium exchange, was also conducted. The development of a method that incorporates deuterium atoms on the aromatic ring offers significant benefits for quantitative GC-MS analysis by stable isotope dilution analysis, i.e. improved accuracy and reproducibility, and this study complemented the aforementioned consumer research.

Declaration

I certify that this work contains no material which has been accepted for the award of any other degree or diploma in my name, in any university or other tertiary institution and, to the best of my knowledge and belief, contains no material previously published or written by another person, except where due reference has been made in the text. In addition, I certify that no part of this work will, in the future, be used in a submission in my name, for any other degree or diploma in any university or other tertiary institution without the prior approval of the University of Adelaide and where applicable, any partner institution responsible for the joint-award of this degree.

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Anna Crump Date

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Chapter 1: Introduction

Oak barrels have long been the preferred vessel for the maturation of wine, with wines aged in oak wood generally associated with higher price points and perceived quality (Lockshin *et al.*, 1993). Full-bodied white wines, for example Chardonnay, often undergo oak fermentation and/or maturation. However, it is red varieties such as Shiraz, Cabernet Sauvignon and Merlot for which oak treatment is generally employed. Wines aged in barrels are continually exposed to small quantities of oxygen, which slowly permeates through the oak staves. This enables oxidation of certain phenolic compounds, thereby enhancing wine sensory characteristics, such as the softening of tannins (Singleton, 1974). Through condensation and polymerisation reactions between tannins and anthocyanins, the stability, palatability and structure of wines are also improved (Heras *et al.*, 2004; Heras *et al.*, 2008), which leads to wines with better colour stability and decreased astringency. During barrel maturation, volatile compounds are extracted into the wine and impart desirable oak aromas and flavours. However, the initial pool of extractable compounds within oak staves is limited, with their availability and rate of diffusion into wine largely dependent on the oak species, seasoning conditions, number of successive uses and wine contact time (Towey *et al.*, 1996).

The strength, pliability and composition of oak make it ideal for barrel manufacture (Singleton, 1974). The strength of oak wood has led it to become the standard against which other timber is compared (Singleton, 1974). A number of aspects can affect the quality and composition of cooperage oak including geographical origin, species, seasoning conditions, barrel assembly and toasting conditions (Matricardi *et al.*, 1999). French oak barrels are generally more expensive than American oak barrels, largely as a result of greater losses associated with coopering French oak (Perez-Prieto *et al.*, 2002). French oak must be split instead of sawn due to its irregular grain structure and higher porosity compared to American oak (Perez-Prieto *et al.*, 2002). A primary difference between the composition of

French and American oak is the higher concentration of volatile compounds typically found in American oak, in particular, the *cis*-isomer of oak lactone (Bozalongo *et al.*, 2007).

The most important oak-derived volatile compound is *cis*-oak lactone, which can directly impart coconut and woody aromas to wine (Mosedale *et al.*, 1999). Previous studies have reported a correlation between the levels of oak lactone and the quality of and preference for certain wines (Mosedale *et al.*, 1999; Pollnitz *et al.*, 1999). Other important oak volatiles include guaiacol (smoky), eugenol (cloves), and vanillin (vanilla) (Pollnitz *et al.*, 2004). As such, oak maturation can contribute a variety of sensory attributes to a wine's flavour profile, increasing its complexity.

Despite the positive contribution of oak to wine flavour, the use of oak as a maturation vessel significantly impacts on winery production costs. After grapes, oak is the second largest input cost associated with wine production (excluding labour). The Winemakers' Federation of Australia (2007) calculated the impact of oak maturation on the cost of wine production and the retail prices required to subsequently achieve a 50% gross margin (Table 1). Maturation, both in oak and in the bottle, significantly increases the cost of production, such that the retail price needs to: double to meet the 104.5% cost increase associated with 2 years maturation in French oak; and triple to meet the 174.5% increase associated with 2 years maturation in French oak, followed by 2 years of bottle maturation.

Table 1. Cost of maturation.

Description	Cost to make ($/case)	Cost increase compared to no oak (%)	Retail price to achieve 50% gross margin ($/750 mL bottle)
No oak	99.97	-	40.00
One year in new French oak	160.32	60.4	64.00
Two years in new French oak	204.43	104.5	82.00
Two years in new French oak and 1 year in bottle	237.74	137.8	95.00
Two years in new French oak and 2 years in bottle	274.39	174.5	110.00

Source: Winemakers' Federation of Australia (2007).

As such, there has been increasing demand within the wine industry for alternative methods of oak maturation (Perez-Magarino *et al.*, 2011). Winemakers have increased their use of oak alternatives, i.e. oak chips, shavings and powders, as rapid methods of introducing oak characteristics to wine, particularly for less expensive wines (Perez-Coello *et al.*, 1999; Heras *et al.*, 2008; Hernandez-Orte *et al.*, 2009). While previous studies have investigated the impact of oak alternatives on the sensory properties of wine (Cano-Lopez *et al.*, 2008; Arfelli *et al.*, 2011; Cejudo-Bastante *et al.*, 2011), few studies have examined consumer attitudes towards and acceptance of these products.

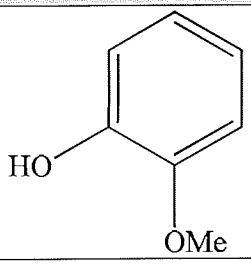
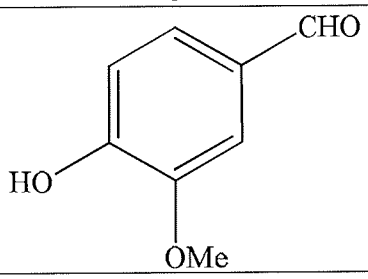
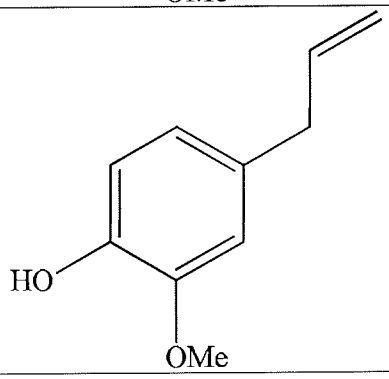
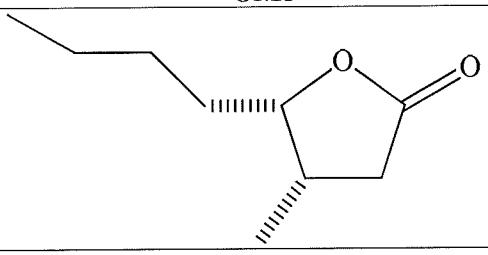
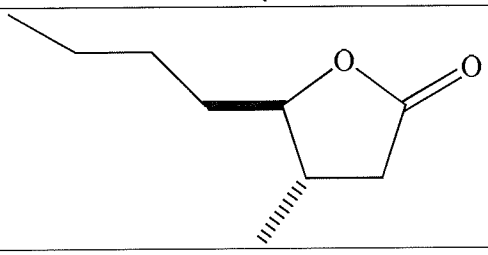
1.1 Oak volatiles

The evolution of oak-derived aroma and flavour during barrel maturation has been the subject of much research. While more than 200 volatile compounds have been identified in wine (Sefton *et al.*, 1990; Guth, 1997; Barbe *et al.*, 2008; Marco *et al.*, 2008; Polaskova *et al.*, 2008), only a relatively small number of these occur at concentrations that directly impact the aroma of the wine. There are differing opinions as to the relative importance of various oak-derived volatile compounds found in wine (which may in part be due to different analysis techniques), but there is general consensus regarding the importance of the *cis*-isomer of oak lactone (Mosedale *et al.*, 1999; Pollnitz *et al.*, 1999). The *cis*- and *trans*-isomers have detection thresholds of 20 and 140 µg/L respectively, in white wine (Brown *et al.*, 2006), and typically exhibit coconut and woody aromas in wine.

Other important oak-derived volatile compounds that can contribute to wine aroma include furfural (almond), guaiacol (smoky/burnt), eugenol (spices, cloves and smoke character), and vanillin (vanilla character) (Perez-Prieto *et al.*, 2002; Spillman *et al.*, 2004). Table 2 shows the structures, detection thresholds and sensory descriptors of several oak-derived volatile compounds. The compounds 5-methylfurfural and 4-methylguaiacol can also be extracted from oak wood into wine. The concentration of oak-derived volatiles in wines treated with different oak species has been previously studied (Garcia-Romero *et al.*, 1998; Garde Cerdan *et al.*, 2002; Perez-Prieto *et al.*, 2002; Consuelo

Diaz-Maroto *et al.*, 2004; Diaz-Maroto *et al.*, 2008). Diaz-Maroto and colleagues analysed oak samples from America, Russia, Hungary and France to develop a rapid analytical method screening for oak volatiles (Diaz-Maroto *et al.*, 2004). Their research identified higher concentrations of eugenol in American oak samples compared to those of European origin, and a significant decrease in oak-lactone concentrations in all samples after toasting. A subsequent study undertaken by Diaz-Maroto and colleagues identified significantly higher levels of guaiacol in Hungarian oak compared to samples from America and the other European locations (Diaz-Maroto *et al.*, 2008). Perez-Prieto and colleagues concluded that the greatest compositional difference between American and French oak was the concentration of *cis*-oak lactone, which was observed at up to four-fold higher concentrations in American oak than in French oak (Perez-Prieto *et al.*, 2002). This study also compared the compositional profiles of new and used barrels, and significant decreases in oak lactone and vanillin concentrations were observed in used barrels. Oak volatile concentrations in extracts of oak wood or wine can be measured by gas chromatography-mass spectrometry (GC-MS) using liquid-liquid or headspace extraction techniques (Carillo *et al.*, 2006). However, it is also important to relate compositional data with sensory data to understand the impact of oak on wine aroma and flavour. This can be achieved by descriptive sensory analysis and involves trained panellists rating the intensity of aroma and flavour attributes, including those derived from oak.

Table 2. Compounds derived from oak wood important to wine sensory characteristics.

Chemical name	Structure	Detection threshold (µg/L) [References]	Aroma descriptor [References]
guaiacol		7.5[a] [8]	smoky, burnt [1, 2, 3, 4, 6]
vanillin		320[b] [3]	vanilla [2]
eugenol		6[a] [8]	clove, spicy [1,2,3,4]
cis-oak lactone		20[c] [7]	coconut, woody [1,2,5]
trans-oak lactone		140[c] [7]	coconut, woody [1,4,5]

Sensory threshold concentrations determined in: [a] model wine; [b] red wine; [c] white wine. References: 1(Spillman *et al.*, 1998), 2(Marin *et al.*, 2005), 3(Carillo *et al.*, 2006), 4(Diaz-Maroto *et al.*, 2008), 5(Wilkinson *et al.*, 2004), 6(Pollnitz *et al.*, 2004) 7(Brown *et al.*, 2006), 8(Ferreira *et al.*, 2000).

During barrel cooperage, the toasting process is particularly important in determining the availability of volatile compounds for extraction during maturation. Toasting involves heating the barrel interior over an open flame to achieve a light, medium or heavy toast. However, the exact toasting conditions are usually a tightly guarded secret within the cooperage. The duration and flame temperature are monitored and controlled according to outside temperature and humidity. The thermal degradation of wood macromolecules (e.g. lignin, cellulose and hemicellulose) during toasting leads to the development of volatile compounds through pyrolysis and hydrothermolysis (Matricardi *et al.*, 1999). More specifically, the heat applied during the toasting process causes degradation of lignin and cellulose present in oak wood to release a range of volatile phenols, aldehydes, and furfurals (Carillo *et al.*, 2006; Bozalongo *et al.*, 2007). As lignin undergoes thermal degradation to ferulic acid, a subsequent decarboxylation step leads to the formation of 4-vinylguaiacol (Maga *et al.*, 2005). After oxidation, vanillin and vanillic acid are produced, which can be further decarboxylated to give guaiacol (Maga *et al.*, 2005). The pyrolysis of cellulose and hemicellulose produces compounds such as furfural and 5-methylfurfural (Carillo *et al.*, 2006; Bozalongo *et al.*, 2007). Therefore, depending on the degree of toasting, a wine's flavour profile may exhibit lighter vanilla, caramel or toasty notes (light toast) or heavier roasted coffee, chocolate or smoky notes (medium/heavy toast).

Thermal degradation of the oak macromolecules is largely responsible for the formation of oak derived volatile compounds that can be extracted into wine during maturation, although the formation and accumulation of volatile oak compounds can be attributed to other factors as well. The composition of oak depends on the initial pool of volatiles and their precursors present in the wood matrix (which in turn depends on the oak species, geographical origin, seasoning of the staves, toasting conditions and age of the barrel) (Spillman *et al.*, 1998). The concentration of oak volatiles in wine depends on the size and age of the barrel and rate of release of compounds from the wood, the duration of maturation, the temperature and storage conditions and the rate at which volatile compounds are utilised in further

chemical or biochemical transformations (Spillman *et al.*, 1998; Perez-Coello *et al.*, 1999; Garde Cerdan *et al.*, 2002; Diaz-Maroto *et al.*, 2004; Garde Cerdan *et al.*, 2006; Garde Cerdan *et al.*, 2006; Bozalongo *et al.*, 2007). The oak volatiles can also have an effect on the flavour of wine indirectly through physical or chemical interactions with other components of wine, affecting solubility of other compounds and oxygen availability (Mosedale *et al.*, 1999).

1.2 Oak alternatives.

While the benefits of oak barrels for winemaking are numerous, their use has a significant impact on winery production costs (Table 1). Factors such as the geographic origin of oak (i.e. French vs. American), barrel volume, duration of maturation and barrel usage influence not only the composition and sensory properties of wine, but also winery expenditure on oak. For over 30 years the wine industry has increasingly adopted alternative maturation and storage vessels for economic and environmental reasons (Singleton, 1974; Perez-Coello *et al.*, 1999; Heras *et al.*, 2008; Hernandez-Orte *et al.*, 2009). More recently, the use of oak chips combined with micro-oxygenation (MOX) has been proposed as an alternative regime for aging wine, so as to reduce production costs whilst retaining extraction of oak aroma and flavour (Bozalongo *et al.*, 2007). The increased surface area of oak alternatives, compared to barrels, results in greater rates of flavour extraction, so both the quantity of oak and the duration of contact with wine are greatly reduced. Additionally, less oak is rejected for alternatives than for barrel cooperage, since oak with structural defects, i.e. knots, cracks or poor grain quality, is acceptable for the preparation of alternative oak products. The combined use of MOX and oak chips could potentially accelerate maturation and reduce the cost of producing oak-aged wines, whilst still achieving comparable quality to those aged traditionally in oak barrels.

Developed at the beginning of the 1990's in France, MOX involves the addition of small, controlled amounts of oxygen to wine at different stages of the winemaking process (Heras *et al.*, 2008; Hernandez-Orte *et al.*, 2009). Its application to aging wine is gaining popularity around the world,

mainly for red wine, and it has been widely used in countries including France, Italy, the U.S.A, Australia and New Zealand. MOX could prove to be an alternative or complementary method to traditional oak barrels for wine aging, and when combined with the addition of oak chips, this 'industrial aging' regime could imitate the modification of phenolic compounds and colour stabilisation achieved with traditional maturation in oak barrels (Cejudo-Bastante *et al.*, 2011). To date, few papers have examined the effect of MOX on wine volatiles (Hernandez-Orte *et al.*, 2009).

With repeated or extended use of barrels, the volatile compounds available for extraction into wine decreases (Garde Cerdan *et al.*, 2002). Furthermore, as a barrel ages it can become populated with undesirable microbes such as *Brettanomyces*, whose growth can lead to the development of off-flavours through the formation of 4-ethylphenol and 4-ethylguaiacol, which impart medicinal and horsey aromas (Chatonnet *et al.*, 1993). The growth of *Brettanomyces* is stimulated in barrels through inadequate sanitation, usually due to low SO_2 levels and/or high pH (Chatonnet *et al.*, 1993). Current processes which are used to prolong the use of a barrel include scraping out the inner surface to expose new timber and subsequent reheating of barrels (Mosedale *et al.*, 1999). Older barrels can be used for some specific wine styles e.g. Grenache, fortified wines or where lees aging is required.

1.3 Consumer research

Consumer research is often used to gain insight into consumers' acceptance, preference and perception of different foods and beverages, particularly to investigate the relative influence of extrinsic and intrinsic cues, such as region of origin, packaging, branding and sensory properties, on consumer liking and purchase intent (Mueller and Szolnoki, 2010). Wine consumer research has played a pivotal role in the past, providing insight into consumer perceptions and expectations surrounding innovative technologies such as cork vs. screw cap (Murray *et al.*, 1997; Marin *et al.*, 2007; Marin *et al.*, 2007). For example, a study into the influence of bottle closures on perceptions of wine quality found North American wine consumers considered wine bottled under screw cap to be of lower quality than wine

bottled under natural cork (Marin *et al.* 2007). A more recent study by Saliba and colleagues, involving 851 Australian wine consumers' perceptions of low alcohol wines identified female consumers and those who drink wine with food as the most likely target market for wines containing lower alcohol levels (Saliba *et al.*, 2013). The situational dependency of wine selection has also been investigated, with grape variety, geographical region of origin and food matching found to be important to consumers with high levels of wine involvement (Johnson and Bastian, 2007; Jaeger *et al.* 2009). The knowledge gained from consumer studies can be used by wine producers to both inform and justify their production methods, in particular for the production of wines at certain price points and/or targeted towards specific segments of the wine market.

Surprisingly, despite the importance of oak to wine production, few studies have considered consumers' knowledge of the role of oak in winemaking or even their preference for oak-aged wines. Instead, wine-related consumer studies have tended to focus on purchase drivers, product involvement and wine expertise (Johnson and Bruwer, 2003; Bruwer and Li, 2007; Johnson and Bastian. 2007; Chrea *et al.* 2011). Lockshin and Rhodus investigated the influence of price and oak flavour on the perception of wine quality and found consumers had no real preference for oak, whereas wine experts (i.e. wholesalers) held oak maturation in much higher regard, particularly with respect to the marketability of wine (Lockshin and Rhodus, 1993). A more recent study evaluated consumer preferences for wines aged in either oak barrels or with oak chips (Pérez-Magariño *et al.* 2011). The authors observed considerable disparity in consumers' wine preferences, but since consumers did not significantly reject wines made with oak chips, they concluded markets exist for wines made using both oak maturation regimes. However, a limitation of this study was the low sample population, i.e. only 65 consumers. A 2011 study found that, after fruity notes in wine, oak attributes were the most important sensory aroma and flavour characteristic for both males and females (Bruwer *et al.*, 2011). The authors

reported that of the various wood aromas, males reported liking 'oak and wood' characters, while females primarily indicated a liking of 'vanilla'.

1.4 Research objectives

Compared with traditional barrel maturation, some wine consumers might consider the use of oak alternatives to be 'industrial', which could negatively impact their purchasing decisions. While there is considerable literature describing the use of oak alternatives in the wine industry and their impact on wine composition and sensory properties, consumer acceptance of wines made using different oak maturation regimes has received little attention. To date, consumers' knowledge of the role of oak in winemaking and their preferences for oaked wines has not been investigated. The majority of wine consumer research instead relates to purchase intent and the influence of extrinsic cues, with little focus on preference for specific aroma and flavour characteristics. Therefore, a greater understanding of consumer acceptance for wines made using oak alternatives is required to ascertain their viability in the market.

Since 2009 there has been a shift in the focus of sensory and consumer research towards understanding consumers' emotional experiences when consuming products. This has become an increasingly important aspect of product development. As an emerging field within sensory science, few studies have been published in relation to wine and emotions, and to date, no studies have been identified in the literature describing how specific wine aromas affect consumer emotions. A recent study developed a list of adjectives that described the emotions of Italian wine consumers, with 16 words considered to adequately describe consumers' feelings during wine consumption (Ferrarini *et al.*, 2010). The study found that for the most part, consuming wine was associated with pleasurable emotions, rather than those linked to unpleasantness. However, the study was limited in that the chosen terms were specific to Italian consumers, which may not be relevant in other geographic locations. The limited research in this field therefore presented an opportunity for further work to be

undertaken to investigate the influence of oak-derived aromas on consumer emotions during wine consumption.

To better understand the influence of alternative oak maturation regimes on the composition, sensory properties, quality and consumer acceptance of wine, the work described in this thesis sought to address three key objectives:

Objective 1: To understand consumer knowledge of oak maturation in winemaking. Consumers are increasingly interested in food and beverage production, and often expect greater transparency and traceability in the use of additives and/or other methods of production. The main objectives of the first study were therefore to determine Australian wine consumers' knowledge of and attitudes towards oak use by the wine industry, and the relative importance of oak maturation as a factor influencing purchase decisions (see Chapter 2).

Objective 2: To investigate the influence of oak maturation methods on the composition, sensory profile and consumer acceptance of wine. The combination of chemical, sensory and consumer hedonic measurements will provide insight into the effect of different oak maturation regimes on wine characteristics (see Chapter 3).

Objective 3: To understand the influence of oak-related aromas and flavours on consumer wellbeing. Consumers' perceptions of how a product will make them feel will strongly influence their purchase intent. Therefore, if winemakers understand the influence of specific aromas on consumer wellbeing, they can tailor their winemaking practices to enhance aromas associated with positive emotions, so as to gain a competitive marketing edge (see Chapter 4).

Chapter 2: Paper 1 - Consumers' knowledge of and attitudes toward the role of oak in winemaking

Despite the importance of oak to wine production and the considerable cost associated with barrel maturation, few studies have explored consumer preferences for wines which have received oak treatment. Many wine-related consumer studies have investigated purchase drivers, product involvement and wine expertise without considering the importance of production information, such as oak maturation. While there is considerable literature describing the use of oak alternatives by the wine industry and their impact on wine composition and sensory properties, consumer acceptance of these wines has received comparatively little attention. Given the increasing use of oak alternatives by Australian wine producers, a greater understanding of consumer acceptance for wines made with oak alternatives is required, to ascertain their viability in the market.

This paper reports the results from an extensive online survey (1015 responses) in which the objective was to gain insight into Australian wine consumers' knowledge of and attitudes toward different oak maturation regimes, in particular, consumers' perceptions and acceptance of wines made using alternative oak products.

2.1 Statement of Authorship

Anna M. Crump, Trent E. Johnson, Susan E.P. Bastian, Johan Bruwer, Kerry, L. Wilkinson (2014). Consumers' knowledge of and attitudes towards the role of oak in winemaking. International Journal of Wine Research, **6**: 1-10.

Anna Crump

Designed and conducted consumer experiments (focus groups and online survey), analysed and interpreted consumer data, drafted and revised manuscript.

I hereby certify that the statement of contribution is accurate and I give permission for inclusion of the paper in this thesis.

Signed Date 18/2/15

Trent Johnson

Analysed and interpreted consumer data, assisted with revising the manuscript.

I hereby certify that the statement of contribution is accurate and I give permission for inclusion of the paper in this thesis.

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Sue Bastian

Analysed and interpreted consumer data, assisted with revising the manuscript.

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Johan Bruwer

Provided advice on methodology, analysed and interpreted consumer data, assisted with revising the manuscript.

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Signed Date 17/2/15

Kerry Wilkinson

Assisted with consumer experiments (focus groups and online survey), analysed and interpreted consumer data, assisted with revising the manuscript.

I hereby certify that the statement of contribution is accurate and I give permission for inclusion of the paper in this thesis.

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Date 18/2/15

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ORIGINAL RESEARCH

Consumers' knowledge of and attitudes toward the role of oak in winemaking

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Abstract: Oak plays an important role in the production of some white wines and most red wines. Yet, consumers' knowledge of the use of oak in winemaking and their preference for oak-related sensory attributes remains unclear. This study examined the knowledge and attitudes of 1,015 Australian wine consumers toward the use of oak in winemaking. Consumers who indicated a liking of oak-aged wines (n=847) were segmented according to their knowledge of the role of oak in wine production. Four distinct consumer clusters were identified, with significantly different preferences for wine sensory attributes and opinions regarding the use of oak alternatives for wine maturation. One segment comprised more knowledgeable consumers, who appreciate and value traditional oak maturation regimes, for which they are willing to pay a premium price. However, a segment comprising less knowledgeable wine consumers was accepting of the use of oak chips, provided wine quality was not compromised. Winemakers can therefore justify the use of oak alternatives to achieve oak-aged wines at lower price points. The outcomes of this study can be used by winemakers to better tailor their wines to the specific needs and expectations of consumers within different segments of the market.

Keywords: maturation, segmentation, wine, wine consumers

Introduction

Oak plays an important role in the production of some white wines and most red wines, affecting both physical attributes and sensory properties. The volatile compounds extracted from oak wood[1–4] can contribute to a wine's overall aroma, flavor, and complexity,[5,6] while the maturation process leads to increased color and stability[7] and reduced astringency.[8] However, oak is an expensive raw material and barrels contribute significantly to production costs. Therefore, while barrel maturation is still preferred for the production of premium wines, the range and application of alternative oak products (eg, oak battens, chips, shavings, and powder), as more rapid and economical methods of oak treatment, has increased.[9,10] The increased surface area of oak alternatives, compared to barrels, results in greater rates of flavor extraction, so both the quantity of oak and the duration of contact with wine are greatly reduced. Additionally, less oak is rejected for alternatives than for barrel cooperage, since oak with structural defects, ie, knots, cracks, or poor grain quality, is perfectly acceptable for the preparation of alternative oak products. Furthermore, the use of micro-oxygenation techniques, in conjunction with oak alternatives, also enables the introduction of small quantities of oxygen, thereby more closely replicating traditional maturation in barrels.[11]

Compared with traditional barrel maturation, some wine consumers may consider the use of oak alternatives to be "industrial", which might negatively impact their

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perceptions of quality. A greater understanding of consumers' knowledge of and attitudes toward different oak maturation regimes is therefore required to ascertain the acceptability of wines aged using alternative oak products.

Consumer research is often used to gain insight into consumers' acceptance, preference, and perception of different foods and beverages; in particular, the relative influence of extrinsic and intrinsic cues, such as region of origin, packaging, branding, and sensory properties, on consumer liking and purchase intent.[12] For example, a study into the effect of bottle closures on the perception of wine quality found North American wine consumers considered wine bottled under screw cap closures to be of lower quality than wine bottled under natural cork.[13] The situational dependency of wine selection influences has also been investigated, with grape variety, geographical region of origin, and food matching found to be important to consumers with high levels of wine involvement.[14,15] This information can be applied by industry to better tailor products to the specific needs and expectations of consumers; ie, to provide a basis for production and/or marketing strategies which target specific consumer segments.

Surprisingly, despite the importance of oak to wine production, few studies have considered consumers' knowledge of the role of oak in winemaking or even their preference for oak-aged wines. Instead, wine-related consumer studies have tended to focus on purchase drivers, product involvement, and wine expertise.[14,16–18] Lockshin and Rhodus investigated the influence of price and oak flavor on the perception of wine quality and found that consumers had no real preference for oak, whereas wine wholesalers held oak maturation in much higher regard, particularly with respect to the marketability of wine.[19] A more recent study evaluated consumer preferences for wines aged in oak barrels or with oak chips.[20] The authors observed considerable disparity in consumers' wine preferences, but since consumers did not significantly reject wines made with oak chips, they concluded that markets exist for wines using both oak maturation regimes.

Winemakers are receptive to the adoption of innovative winemaking processes, including the use of oak alternatives, provided economic benefit can be demonstrated and neither wine quality nor consumer acceptability will be compromised. The objective of this study was therefore to gain insight into wine consumers' knowledge of and attitudes toward different oak maturation regimes, in particular, consumers' perceptions and acceptance of wines made using alternative oak products; ie, to investigate whether or not knowledgeable consumers hold traditional oak barrel maturation of wine in high regard, while less knowledgeable consumers are accepting of oak alternatives, justifying their use by winemakers for wines at lower price points.

Materials and methods

Focus groups

Focus groups were conducted to gain preliminary insight into wine consumers' knowledge of the role of oak in winemaking, in order to inform the structure and content of a larger, more detailed consumer survey. Participants were recruited by distributing fliers to residential letterboxes in suburbs located within an 8 km radius to the west, south, and southeast of the Adelaide (SA, Australia) central business district. These suburbs were specifically chosen for their proximity to the focus group venue, and therefore increased likelihood of participation. Participants were screened against inclusion criteria comprising wine consumption at least once a month and being of legal drinking age (ie, at least 18 years of age) and exclusion criteria precluding participation by wine industry professionals and university staff and students.

Participants (12 females and 18 males, aged between 18 and 65 years) were assigned to one of three focus groups, held during May and June 2010, according to their availability. Two researchers attended each focus group, ie, a moderator and an assistant. The moderator led the focus group activities, which comprised: 1) a triangle test[21] to investigate differentiation of oaked and unoaked wines (using a commercial 10 Australian dollar [AUD] Chardonnay, with and without the addition of 10 g/L toasted oak); 2) an evaluation of wine bottle labels and the relative importance of label content; and 3) a series of preprepared questions pertaining to winemaking, the role of oak in wine production, and wine purchasing behavior. Group discussions were transcribed by the assistant, with each focus group also being recorded to ensure all responses were captured. The moderator remained neutral and did not try to influence the participants or bias their responses in any way. The duration of each focus group was ~90 minutes and participants were compensated with a bottle of wine.

Consumer survey

Themes identified during the focus groups were used to develop an online consumer survey, which was administered nationally using SurveyMonkey™. Participants were recruited using a variety of methods, including social networking sites, wine blogs, local and interstate media, and national distribution of a flier. Screening was performed using the same inclusion criteria applied to focus group participants.

The survey took ~10–15 minutes to complete and data were collected over an 8-month period. A convenience sample of 1,447 consumers was achieved, with a completion rate of 78% (ie, 1,128 responses). Participants residing outside Australia (n=113) were excluded, resulting in a total of 1,015 responses from Australian wine consumers.

The survey was divided into three sections. The first section comprised sociodemographic questions (Table 1), while the second and third sections explored consumers' wine involvement and knowledge of the use of oak in winemaking.

The second section comprised questions relating to participants' wine purchasing and consumption behavior, incorporating questions from previous research.[14] These questions were intended to ascertain how much consumers typically spend on a bottle of wine, the factors that most strongly influence their selection of wine for consumption at home, how often they read wine bottle labels, and their

Table 1 Consumer demographic, consumption, and knowledge characteristics

	Total sample (%) n=1,015	Oak knowledge segments (%)			
		C1 n=461	C2 n=133	C3 n=141	C4 n=112
Sex					
Male	37.0	33.8	38.3	44.7	64.3
Female	63.0	66.2	61.7	55.3	35.7
Age (years)					
18–34	35.8	33.2	41.4	29.8	41.1
35–54	46.5	48.4	44.4	48.9	47.3
55+	17.7	18.4	14.3	21.3	11.6
Highest level of education achieved					
Secondary qualifications	8.5	9.8	8.3	9.2	6.3
Technical/trade certificate	14.7	15.6	17.3	13.5	7.1
Undergraduate	27.5	27.8	27.1	22.0	33.9
Postgraduate	49.3	46.9	47.4	55.3	52.6
Average household income (AUD)					
≤$25,000	4.7	5.9	6.8	3.5	1.8
$25,001–$50,000	10.2	10.0	12.0	8.5	6.3
$50,001–$75,000	17.6	18.2	17.3	18.4	10.7
$75,001–$100,000	19.0	20.2	18.8	18.4	17.9
$100,001–$150,000	27.5	26.2	29.3	27.7	33.9
$150,001–$200,000	13.4	13.4	12.0	14.9	16.1
>$200,000	7.5	6.1	3.8	8.5	13.4
Frequency of wine consumption					
Frequent (≥4 times per week)	32.3	30.2	24.1	38.3	52.7
Moderate (1–3 times per week)	55.0	55.5	63.2	51.8	45.5
Occasional (≤ once per week)	12.7	14.3	12.8	9.9	1.8
Average spend on a bottle of wine (AUD)					
≤$10	12.2	14.1	9.0	10.6	2.7
$11–$15	38.3	43.4	30.8	38.3	20.5
$16–$20	33.1	30.4	43.6	36.2	34.8
$21–$30	13.8	10.4	12.8	13.5	33.0
$31–$50	2.3	1.7	3.0	0.7	8.0
>$50	0.3	0.0	0.8	0.7	0.9
Likelihood of reading back label information					
Unlikely	21.5	21.3	12.0	19.1	17.0
Neither likely nor unlikely	14.2	14.3	10.5	14.2	13.4
Likely	64.3	64.4	77.4	66.7	69.6
Self-assessed wine knowledge					
Limited/novice	10.5	13.2	6.0	5.7	1.8
Basic	43.1	51.0	45.1	26.2	14.3
Intermediate	38.6	34.3	44.4	56.7	41.1
Expert	4.2	1.3	2.3	5.0	24.1
Professional	3.5	0.2	2.3	6.4	18.8

Abbreviations: C1, cluster 1; C2, cluster 2; C3, cluster 3; C4, cluster 4.

tendency to cellar wines. Section 3 comprised questions relating to consumers' knowledge and opinions regarding the use of oak during winemaking (Table 2), and their liking of wine sensory attributes, including descriptors associated with oak maturation (Table 3). Questions also sought to determine consumers' preferences for oak-aged wines and French versus American oak (Table 4). The survey required participants to give either "yes" or "no" responses or to indicate agreement or disagreement to statements using 5-point category scales, as used elsewhere.[15,22] For example, the section concerning purchasing behavior required participants to indicate how strongly different factors influenced their wine purchasing decisions, again using 5-point category scales, ranging from 1, "very unlikely" to 5, "very likely" (Table 5).

Data analysis

Consumer data were analyzed using a combination of descriptive techniques, including analysis of variance (ANOVA) with post hoc Fisher's test, correlation analysis, and principal component analysis. For those participants who indicated they liked oaked wines (n=847), data were also analyzed by factor and cluster analysis to allow segmentation on the basis of oak knowledge. Statistical analyses were performed using SPSS Statistics (v 19; IBM Corporation, Armonk, NY, USA) and XLSTAT 2012.1.01 (Addinsoft, New York, NY, USA).

Results and discussion

The primary objective of the focus groups was to inform the structure and content of the online consumer survey.

Table 2 Consumers' knowledge of and attitudes toward the use of oak in wine production

	Oaked wine consumer segment n=847	Oak knowledge segments			
		C1 n=461	C2 n=133	C3 n=141	C4 n=112
Perceptions relating to wines aged in stainless steel tanks with oak chips, compared to wines aged in barrels ...					
Opinions					
Not as socially acceptable or impressive	3.0±0.79	2.93^{b}±0.61	3.30^{a}±0.87	2.55^{c}±0.91	3.49^{a}±0.83
Type of oak treatment should be on label	3.6±0.81	3.57^{b}±0.69	3.90^{a}±0.77	3.48^{b}±0.94	3.74^{a,b}±1.02
Don't care how it's made as long as it tastes good	3.6±0.98	3.77^{a}±0.86	3.47^{b,c}±0.99	3.74^{a,b}±1.09	3.21^{c}±1.14
Oak alternatives are the way of the future	3.0±0.74	3.12^{a}±0.52	3.12^{a}±0.75	2.97^{a}±0.88	2.25^{b}±0.90
Production method has no influence on purchase decision	3.3±1.00	3.51^{a}±0.80	3.06^{b}±1.00	3.42^{a}±1.08	2.33^{c}±1.02
Wines made with oak alternatives are "cheap"	3.1±0.72	2.97^{c}±0.40	3.43^{b}±0.82	2.62^{d}±0.78	3.77^{a}±0.85
Doesn't sound romantic	3.6±0.67	3.49^{b}±0.67	3.91^{a}±0.69	3.36^{b}±0.96	3.92^{a}±0.74
Knowledge					
Are young, "drink now" wines	3.3±0.68	3.18^{b}±0.48	3.90^{a}±0.54	2.98^{c}±0.81	3.73^{a}±0.74
Wine is always aged in a barrel	2.5±0.93	2.89^{a}±0.63	2.92^{a}±1.00	1.72^{b}±0.70	1.66^{b}±0.90
Have a crisper taste	3.0±0.57	3.02^{b}±0.29	3.27^{a}±0.62	2.90^{b}±0.69	2.54^{c}±0.89
This production method is used to produce large volumes of wine	3.5±0.69	3.18^{c}±0.44	3.94^{b}±0.55	3.30^{c}±0.78	4.24^{a}±0.68
Are not good enough to be aged in a barrel	2.9±0.67	2.83^{b}±0.40	3.33^{a}±0.66	2.22^{c}±0.61	3.48^{a}±0.75
Lower quality	3.0±0.76	2.83^{c}±0.46	3.56^{b}±0.67	2.30^{d}±0.68	3.97^{a}±0.60
Have less depth and complexity	3.2±0.70	3.00^{c}±0.38	3.72^{b}±0.60	2.93^{c}±0.82	4.13^{a}±0.67
Would be less likely to show signs of spoilage	3.0±0.64	3.07^{b}±0.41	3.42^{a}±0.68	2.84^{c}±0.83	2.77^{c}±0.86
Won't age as well/less cellaring potential	3.1±0.66	2.99^{c}±0.36	3.56^{b}±0.62	2.61^{d}±0.72	3.86^{a}±0.72
Are better than wines matured in old oak barrels	2.6±0.70	2.88^{a}±0.38	2.61^{b}±0.80	2.34^{c}±0.65	1.67^{d}±0.76
Top brands/wineries always use barrels	3.0±0.74	3.12^{b}±0.41	3.42^{a}±0.84	2.33^{d}±0.70	2.82^{c}±1.05
There would be no difference in quality between the two wines	2.7±0.74	2.97^{a}±0.47	2.60^{b}±0.77	2.78^{b}±0.80	1.84^{c}±0.80
Display better oak/aroma flavor	2.8±0.67	3.02^{a}±0.34	2.92^{a}±0.76	2.58^{b}±0.66	1.84^{c}±0.72

Notes: Data are mean ± standard deviation, where 1= strongly disagree and 5= strongly agree. Different letters within a row indicate a statistically significant difference ($P<0.05$, one-way ANOVA, Fisher's least significant difference post hoc).
Abbreviations: ANOVA, analysis of variance; C1, cluster 1; C2, cluster 2; C3, cluster 3; C4, cluster 4.

Table 3 Consumers' liking of wine sensory attributes

Sensory attributes	Total sample n=1,015	Oak knowledge segments			
		C1 n=461	C2 n=133	C3 n=141	C4 n=112
Oak					
Chocolate, caramel, butterscotch, honey	3.42±0.96	3.43±0.96	3.54±0.96	3.44±0.93	3.57±0.95
Vanilla, coconut, woody, oak	3.42±0.94	3.54±0.84	3.55±0.94	3.62±0.89	3.65±0.96
Red					
Spicy, black pepper, licorice	3.46±1.12	3.45^{b}±1.04	3.64^{b}±1.08	3.67^{b}±1.09	4.04^{a}±0.93
Herbaceous, earthy, tobacco, cigar	3.02±1.13	2.94^{c}±1.04	3.25^{b}±1.09	3.27^{b}±1.17	3.73^{a}±1.11
Prune, raisin, fig	3.36±0.89	3.40±0.83	3.46±0.95	3.35±0.96	3.45±0.88
Blackcurrant, blackberry, strawberry, cherry	3.87±0.88	3.82^{b}±0.87	$3.96^{a,b}$±0.90	$3.92^{a,b}$±0.88	4.16^{a}±0.71
White					
Floral, rose, geranium	3.00±0.95	2.92^{b}±0.91	3.00^{b}±0.99	2.91^{b}±0.99	3.37^{a}±0.96
Citrus, lemon, lime	3.65±0.94	3.59^{b}±0.93	3.40^{b}±0.99	3.63^{b}±0.97	4.04^{a}±0.87
Capsicum, cut grass, eucalyptus	2.78±0.97	2.81±0.93	2.77±0.95	2.75±1.02	3.06±1.09
Apricot, peach, apple	3.43±0.91	3.36^{b}±0.89	3.36^{b}±0.94	3.43^{b}±0.91	3.73^{a}±0.80
Melon, passion fruit, pineapple, tropical fruit	3.41±1.02	3.38±0.97	3.38±1.07	3.39±1.03	3.46±1.04

Notes: Data are means ± standard deviation, where 1= strongly dislike and 5= strongly like. Different letters within a row indicate a statistically significant difference ($P<0.05$, one-way ANOVA, Fisher's least significant difference post hoc).
Abbreviations: ANOVA, analysis of variance; C1, cluster 1; C2, cluster 2; C3, cluster 3; C4, cluster 4.

Participants' responses to questions pertaining to wine production, the role of oak in winemaking, and wine purchasing behavior, including the importance of information presented on wine bottle labels, varied considerably depending on factors such as sex, wine involvement, and frequency of wine consumption (data not shown), highlighting a need for segmentation of consumer data from the online survey. Participants also completed a triangle test, with oaked and unoaked Chardonnay wines presented in a balanced, randomized presentation order.[21] Twenty (out of 30) consumers correctly identified the different sample ($P<0.001$), but only four participants attributed oak-related sensory attributes as the basis for their difference. Most participants instead used basic sensory descriptors such as bitterness, sweetness, and/or fruit intensity to describe differences between the wines. This suggests consumers readily perceived the sensory attributes associated with oak maturation, but could not adequately describe them, in agreement with an earlier study.[23]

Demographic characteristics of consumers

One thousand and fifteen respondents completed the online survey. A higher proportion of participants were female, ie, 63% female and 37% male (Table 1), which was consistent

Table 4 Consumers' preferences for oak maturation of wine

	Total sample (%) n=1,015	Oaked wine consumer segment (%) n=847	Oak knowledge segments (%)			
			C1 n=461	C2 n=133	C3 n=141	C4 n=112
Enjoy drinking oaked wines						
No	16.6					
Yes	44.4		47.3	55.6	58.2	68.8
Sometimes	39.0		52.7	44.4	41.8	31.3
Prefer French or American oak						
Not sure/no preference		81.0	92.6	75.9	79.5	41.1
French		17.1	6.5	21.1	17.0	56.3
American		1.9	0.9	3.0	3.5	2.7
Able to tell the difference between wine made in barrel or with alternatives						
No		78.3	90.9	78.9	78.0	25.9
Yes		21.7	9.1	21.1	22.0	74.1

Abbreviations: C1, cluster 1; C2, cluster 2; C3, cluster 3; C4, cluster 4.

Table 5 Relative importance of factors influencing wine consumers' purchasing decisions

	Total sample n=1,015	Oak knowledge segments			
		C1 n=461	C2 n=133	C3 n=141	C4 n=112
Oak treatment	2.67±1.10	2.51^{c}±1.02	2.94^{a,b}±1.11	2.84^{b}±1.11	3.25^{a}±0.94
Production method	2.44±1.00	2.35^{c}±0.95	2.67^{a,b}±1.04	2.51^{b,c}±1.03	2.92^{a}±1.05
Previous consumption	4.46±0.64	4.48±0.61	4.42±0.63	4.48±0.68	4.33±0.66
Price	4.13±0.73	4.11±0.71	4.20±0.64	4.04±0.86	3.96±0.77
Wine style	4.05±0.90	3.97^{b}±0.93	4.14^{a,b}±0.80	4.12^{a,b}±0.96	4.26^{a}±0.68
Wine grape variety	4.00±1.03	3.87^{b}±1.04	4.17^{a}±0.91	4.22^{a}±0.92	4.38^{a}±0.77
Occasion	4.00±0.86	3.97±0.85	4.16±0.68	3.96±0.91	3.92±0.92
Try something different	3.54±0.90	3.49^{b}±0.87	3.58^{a,b}±0.86	3.61^{a,b}±0.85	3.83^{a}±0.95
Reputation of winemaker	3.71±0.90	3.67^{b}±0.87	3.83^{a,b}±0.91	3.70^{b}±0.90	4.00^{a}±0.81
Wine region	3.95±0.92	3.90^{b}±0.92	4.16^{a}±0.77	3.97^{a,b}±0.83	4.17^{a}±0.85
Environmental issues	2.63±1.11	2.65±1.13	2.78±1.15	2.53±1.09	2.49±1.04
Recommendation by wine writers	3.05±1.12	2.96^{c}±1.10	3.28^{a,b}±1.14	3.02^{b,c}±1.07	3.62^{a}±0.96
Cellaring potential	2.50±1.14	2.37^{c}±1.10	2.73^{b}±1.17	2.68^{b}±1.16	3.26^{a}±1.03
Wine brand	3.75±0.49	3.83^{a}±0.43	3.82^{a,b}±0.48	3.68^{b}±0.53	3.48^{c}±0.54
Food pairing	3.28±1.06	3.26^{b}±1.02	3.47^{a,b}±1.03	3.34^{a,b}±1.06	3.66^{a}±0.90
Year of vintage	3.34±1.02	3.29^{c}±0.95	3.56^{b}±0.95	3.53^{b}±0.99	3.92^{a}±0.86
Advertising	3.45±1.04	3.55^{a}±1.00	3.47^{a,b}±0.99	3.26^{b,c}±1.01	3.05^{c}±1.13
Awards or medals	3.35±0.97	3.47^{a}±0.87	3.50^{a}±0.97	3.12^{b}±0.98	2.96^{b}±1.09
Alcohol level	2.35±1.05	2.38^{a,b}±1.04	2.22^{b}±1.02	2.24^{b}±0.94	2.59^{a}±1.10
Packaging	3.13±1.03	3.18^{a}±1.00	3.25^{a}±1.02	3.06^{a}±1.01	2.69^{b}±1.08
Recommendation from friends/family	3.91±0.84	3.98^{a}±0.77	3.97^{a}±0.83	3.83^{a,b}±0.86	3.63^{b}±0.96
Cellar door visit	3.30±1.21	3.22^{b}±1.24	3.44^{a,b}±1.11	3.51^{a,b}±1.06	3.63^{a}±1.11
Additives	2.64±1.13	2.72^{a}±1.11	2.78^{a}±1.18	2.58^{a,b}±1.20	2.32^{b}±1.03
Hot or cold weather	3.45±1.14	3.44±1.09	3.66±1.14	3.60±1.12	3.55±1.19

Notes: Data are means ± standard deviation, where 1= very unlikely and 5= very likely. Different letters within a row indicate a statistically significant difference ($P<0.05$, one-way ANOVA, Fisher's least significant difference post hoc).
Abbreviations: ANOVA, analysis of variance; C1, cluster 1; C2, cluster 2; C3, cluster 3; C4, cluster 4.

with previous wine consumer research.[22] A relatively even distribution of consumers was obtained across the different age groups. The number of respondents with tertiary qualifications (76.8%) was greater than in the general population,[24] but again consistent with other studies that demonstrated wine consumers are more likely to hold tertiary qualifications.[16,25,26] This was also reflected in respondents' household incomes; ~50% of respondents had an average household income above 100,000 AUD, with the majority of participants' household earnings exceeding the 64,168 AUD per annum Australian median household income.[27]

Segmentation of wine consumers according to their oak knowledge

The wine industry has long understood the benefits of market segmentation. A number of segmentation studies involving Australian wine consumers have been reported in the literature, in which variables such as lifestyle, wine knowledge and involvement, and wine expertise were used to segment the market.[14,17,18,25,28]

In the current study, wine consumers' knowledge regarding the use of oak in wine production was used to identify four distinct market segments. Consumers' responses to 13 oak knowledge statements (Table 2) were subjected to correlation analysis, which revealed multiple coefficients >0.3. The Kaiser–Meyer–Olkin value was 0.81 and Bartlett's test of sphericity was statistically significant. Principal component analysis identified three factors with eigenvalues exceeding 1, which explained 28.9%, 15.2%, and 9.1% of the variance, respectively. Parallel analysis supported retention of the three-factor solution and oblimin rotation showed strong loadings with all but one of the variables from one factor (data not shown). The variable "no difference in quality" (Table 2) was excluded from further analysis as it did not load positively against any factor. Factor 1 related to the effect of oak on wine quality; factor 2 comprised consumers' opinions toward the use of oak alternatives; and factor 3 related to the contribution of oak to wine flavor. Hierarchal cluster analysis followed by k-means cluster analysis was performed on these three factors and yielded a four-cluster segmentation solution,

with the final cluster centers reported in Table 6. Subsequent discriminant analysis revealed that this solution provided a 93% accurate fit to the data.

Cluster 1 (C1), n=461

These predominantly female consumers made up the largest segment; C1 consumers indicated they "don't care how it's made as long as it tastes good", but otherwise did not have strong opinions (positive or negative) to statements concerning oak quality or the use of oak alternatives. This cluster comprised the highest proportion of occasional drinkers, who typically spend the least on wine for home consumption (ie, 57.5% spend less than 15 AUD/bottle). The majority of C1 considered their wine knowledge to be limited to basic, and constituents were least likely to read the back label of a wine bottle.

Cluster 2 (C2), n=133

These consumers did not have an opinion, either positive or negative, regarding the use of oak alternatives, but they did agree that oak has an impact on the taste and quality of wine. This cluster comprised a high proportion of young consumers (with 41.4% aged 18–34 years), with moderate wine consumption and an average spend of 16–20 AUD per bottle. C2 were also the cluster most likely to read wine bottle labels.

Cluster 3 (C3), n=141

The consumers in this segment neither agreed nor disagreed that oak influences the taste or quality of wine, but they did have a moderately strong, negative opinion regarding the use of oak alternatives. This cluster was not weighted toward one sex or the other, comprised the highest percentage of older consumers (21.3% over 55 years), and were well educated, with 55.3% holding postgraduate qualifications.

Cluster 4 (C4), n=112

The consumers within this segment were predominantly male (64.3%), and considered themselves more knowledgeable about wine than consumers from other clusters, with 42.9% rating their knowledge as expert or professional. These consumers were largely frequent wine drinkers, with strong opinions regarding the impact of oak on wine quality and strong negative views on the use of oak alternatives for wine maturation. Interestingly, this cluster had no real opinion on the effect of oak on wine taste. These consumers had high average household incomes and more than one-third spend 21–30 AUD/bottle of wine for home consumption.

Table 6 Final cluster centers for oak knowledge segmentation

	C1 n=461	C2 n=133	C3 n=141	C4 n=112
Oak impacts taste	3.09[b]	3.53[a]	2.91[d]	3.02[c]
Opinion toward oak alternatives	2.98[a]	2.97[a]	2.25[b]	2.00[c]
Oak impacts quality	2.97[c]	3.63[b]	2.68[d]	3.94[a]

Notes: Data are means. Different letters within a row indicate a statistically significant difference ($P<0.05$, one-way ANOVA, Fisher's least significant difference post hoc).
Abbreviations: ANOVA, analysis of variance; C1, cluster 1; C2, cluster 2; C3, cluster 3; C4, cluster 4.

Consumers' knowledge of and attitudes toward different oak maturation regimes

Within the segment of consumers who enjoy oaked wines (n=847), a large proportion of respondents answered "neither agree nor disagree" to the majority of statements relating to the use of oak in winemaking. Participants' mean responses ranged from 2.6 to 3.5 for oak knowledge statements and from 3.0 to 3.6 for opinions concerning oak maturation (Table 2), suggesting a general lack of knowledge regarding the role of oak in wine production. Indeed, more than 10% of these consumers thought wine was always aged in oak barrels (data not shown). Sex largely did not influence participants' perceptions of oak alternatives, but females agreed that oak alternatives were "the way of the future" slightly more than males, possibly because males perceived wines made with oak alternatives to be "cheap" (data not shown).

By comparison, significant differences were observed between the four clusters' opinions regarding the maturation of wine using oak barrels or oak chips. C1 responses were similar to those of the total population; mean knowledge responses ranged from 2.83 to 3.18 and mean opinion responses ranged from 2.93 to 3.77, demonstrating the tendency of C1 to "neither agree nor disagree". In contrast, C2, C3, and C4 responses reflected broader use of the category scale. C2 and C4 shared similar attitudes toward oak alternatives. Both clusters agreed that wines aged with oak chips are "not as socially acceptable or impressive", are "cheap", and "don't sound romantic", compared to barrel-aged wine; but C4 disagreed that "oak alternatives are the way of the future" and indicated that "production method has no influence on purchase decision". Like C1, C3 constituents indicated they "don't care how it's made as long as it tastes good". C3 generally considered oak alternatives more favorably than the other clusters; ie, they disagreed that wines made with oak chips were "cheap" and "less socially acceptable". Interestingly, all clusters agreed the method of oak treatment should be specified on the label. This is consistent with recent work that suggests modern consumers are increasingly interested in the production, traceability, and labeling of foods and beverages.[29]

These results support our assertion that consumers with increased wine knowledge, ie, consumers within C4, are less accepting of alternate methods of oak maturation and hold traditional barrel maturation in higher regard. This segment comprised more knowledgeable wine consumers (42.9% rated their wine knowledge as expert or professional), with a higher disposable income (>60% have a household income above 100,000 AUD), who were willing to pay a higher premium for a quality product (>40% spend more than 20 AUD/bottle). In contrast, less knowledgeable consumers were more accepting of oak alternatives. More than 88% of consumers in C3 and 95% of consumers in C1 and C2 rated their knowledge as novice, basic, or intermediate. These clusters, in particular C1 and C3, were also more accepting of oak alternatives. C1, C2, and C3 generally purchase wines at lower price points (>80% spend less than 20/bottle AUD); ie, wines which are more likely to be matured using oak alternatives. These findings suggest winemakers are justified in using oak alternatives; ie, the target market does not consider these wines to be inferior.

Consumers' preferences for wine sensory attributes, including oak-derived sensory attributes

Consumers were asked to rate their preferences for eleven groups of sensory attributes commonly associated with wine aroma and flavor (Table 3). The majority of sensory attributes were scored favorably by participants, ie, mean scores were ≥3.4. The exceptions were "capsicum, cut grass, eucalyptus", "floral, rose, geranium", and "herbaceous, earthy, tobacco, cigar" attributes, which received mean ratings of 2.78, 3.00, and 3.02, respectively, from all participants. Berry fruit attributes, ie, "blackcurrant, blackberry, strawberry, cherry", were most preferred, with a 3.87 rating. These attributes were also preferred by each of the oak knowledge clusters (3.82–4.16). Citrus ("citrus, lemon, lime"), spice ("spicy, black pepper, licorice"), and oak ("vanilla, coconut, woody, oak") attributes were also highly rated, while green characters ("herbaceous, earthy, tobacco, cigar" and "capsicum, cut grass, eucalyptus") were least popular.

C1 ratings ranged from 2.81 to 3.59, ie, somewhat lower and with a smaller range than for other clusters. There was no significant difference between C2 and C3 responses, while average ratings for C4 tended to be higher than other clusters – in some cases, significantly higher. Oak attributes were favorably rated by all clusters (3.43–3.65); C4 gave oak attributes the highest scores, although scores were not significantly different between clusters. These findings are similar to those reported in a study that compared liking scores of wine consumers and wine experts, in which consumers indicated they liked "confectionary", "floral", "vanilla", "red berry", "coconut", and "caramel" attributes (of which vanilla, coconut, and caramel are generally considered to be oak-derived), but disliked "pepper", "smoky", and "woody" attributes.[28] In contrast, "woody" had a positive influence on wine experts' liking scores, but experts disliked "vegetal", "coffee", "smoky", and "leather" characters.

Previous studies have found that the descriptions given by novice wine drinkers usually comprise basic terms such as "sweet" or "fruity" and therefore do not enable identification or discrimination of different wines.[23,30] Additionally, since certain attributes are often associated with red and white wines, for example, red wines are typically described using dark attributes (pepper, blackberry) while white wines are described using white or yellow attributes (lemon, honey),[31] untrained consumers may associate these terms with specific wine styles. Thus, if they prefer white wines, for example, they may respond less favorably to those attributes typically associated with red wines.

Consumers' preferences for oak-aged wines

The vast majority (83.4%) of participants indicated they enjoy drinking oaked wines (Table 4). Of those consumers who enjoyed oaked wines, most had no preference for wines aged with French or American oak, but where a preference was given, it was overwhelmingly in favor of French oak, while most (78.3%) did not believe they would be capable of distinguishing between wine aged in a barrel and wine aged with oak alternatives. Segmentation of these consumers into their four oak knowledge clusters allowed interesting differences between clusters' responses to be observed. An increasing acceptance of oaked wines was observed across C1 to C4, with a higher proportion of participants responding "yes" than "sometimes"; ie, more confident responses.

The majority of participants from C1, C2, and C3 indicated no preference for French versus American oak (90.9, 78.9, and 78.0, respectively), whereas the segment most knowledgeable about oak and wine, ie, C4, indicated a strong preference for French oak (56.3%). The majority of this cluster (74%) also believed they would be able to differentiate wines based on oak maturation regimes.

The importance of oak as a purchase driver for wine consumers

Participants were asked to rate the importance of 24 intrinsic and extrinsic wine choice factors, to determine the relative influence

of oak maturation on Australian consumers' wine selection and purchasing behavior (Table 5). Prior consumption, wine style, grape variety, occasion, and price were identified as the five most important factors when selecting wine, in agreement with previous findings.[14] Production method and oak treatment (ie, the use of French or American oak, new or old oak, and the duration of oak treatment) were ranked 19th and 24th, respectively, and are therefore unlikely to have any real impact on wine purchasing decisions. When specified, this information would generally be presented to consumers via the wine back label. In the current study, the majority of respondents (ie, 64%) indicated they were likely to read back label information. While the inclusion of manufacturing statements on wine labels is of interest to consumers,[32] back label information has been shown to have considerably less influence on wine choice than price.[25]

Responses were also analyzed following segmentation according to oak knowledge. The four oak knowledge clusters also rated previous consumption, price, wine style, grape variety, and occasion as purchase drivers of considerable importance, as evidenced by mean scores ranging from 3.87 to 4.48. However, C4 rated the reputation of the winemaker (4.00) higher than price (3.96) and occasion (3.92). Wine region was an important consideration for C2, C3, and C4 (3.97–4.17), while C1 and C2 regarded recommendations from friends and family favorably (3.98 and 3.97, respectively). The segments' self-assessed wine knowledge was reflected in their responses. C1, the segment with the least wine knowledge (Table 1), attached significantly more importance to wine brand, advertising, awards or medals, and packaging than C4, the most knowledgeable segment, who instead regarded recommendations from wine writers and the year of vintage to be of greater importance. While oak treatment and production method were not considered to be especially important purchase drivers by any of the oak knowledge clusters (2.35 to 3.25), the relative importance of these factors differed significantly between clusters, with C4 being more likely (3.25 and 2.92) to be influenced by these factors than C1 (2.51 and 2.35).

Conclusion

This study has shown that the oak maturation regime employed during winemaking has little influence on the purchasing decisions of most, but not all, consumers. Within the Australian wine consumer population, there exists a segment comprising knowledgeable consumers who appreciate and value traditional oak maturation regimes, for which they are willing to pay a premium. However, less knowledgeable wine consumers were not deterred by the use of oak chips provided wine quality was not compromised, and so winemakers can therefore justify the use of oak alternatives to achieve oak-aged wines at certain price points. Significant cost savings, in terms of both capital investment (ie, barrels) and labor associated with cellar management can be realized through the use of oak alternatives. Consumers' responses confirmed their liking of oak-related sensory attributes, despite the fact that, in some cases, they may not have known such attributes originated from oak.

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Disclosure

The authors report no conflicts of interest in this work.

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Chapter 3: Maturation of wine using barrel alternatives

3.1 PART 1 - Introduction

In response to economic pressures, the wine industry has increased its use of oak alternatives, i.e. oak chips, shavings and powders, as rapid methods of oak maturation, particularly for less expensive wines (Perez-Coello *et al.*, 1999; Heras *et al.*, 2008; Hernandez-Orte *et al.*, 2009) with wines aged in oak barrels generally being associated with higher price points and increased perceptions of quality (Lockshin *et al.*, 1993). Several studies have described the addition of oak chips to wine, either during or following fermentation, and the resultant presence of oak-derived volatile compounds and/or oak-related sensory attributes in the resulting wine (Gutierrez Afonso, 2002; Bautista-Ortin *et al.*, 2008; Cano-Lopez *et al.*, 2008). A key difference between barrel maturation and maturation with oak alternatives is the opportunity for controlled oxidation, which occurs during barrel maturation due to the oxygen permeability of oak wood (Singleton, 1974). Therefore, development of a vessel which more closely mimics barrel maturation conditions, in order to produce wines of similar quality, could be highly desirable. Micro-oxygenation (MOX) involves the controlled addition of oxygen to wine during fermentation and/or maturation to improve the colour stability, tannin structure and other sensory attributes (Paola Parpinello *et al.*, 2012). Depending on wine type, oxygen is introduced at a rate of 2 to 90 mg/L per month, using a diffuser controlled by a computerised monitoring system (Gomez-Plaza *et al.*, 2011). Previous studies have combined MOX with oak alternatives to overcome some shortfalls associated with the use of oak staves or chips alone, whilst still achieving economic benefits (Perez-Magarino *et al.*, 2009; Del Alamo *et al.*, 2010; Arfelli *et al.*, 2011; Cejudo-Bastante *et al.*, 2011). Arfelli and colleagues found that wines produced using a combination of oak chips and MOX were significantly more preferred by sensory panellists than the control wine, which was not subjected to MOX (Arfelli *et al.* 2011); the authors primarily attributed this to the reduced bitterness and astringency exhibited in these wines.

This chapter describes the evaluation of a 'barrel alternative' developed by South Australian company, Ausvat Pty. Ltd., known as the Stakvat®. Stakvats are 900 L stainless steel vessels which can be used for fermentation, maturation and/or storage of wine. They feature internal temperature control (for heating or cooling wine), sloped bases to facilitate drainage, easy-open doors to facilitate cleaning, rounded corners to prevent the accumulation of bacteria, and fittings that allow Stakvats to be easily transported (e.g. by forklift) and stacked for efficient storage (Figure 1). Indeed, the Stakvat system improves wine storage efficiency by almost 60%; with 30 Stakvats (containing 27,000 L of wine) occupying approximately the same storage space as 76 oak barriques (containing only 17,100 L of wine (Warren, 2010). Additionally, these barrel alternatives enable cost-effective integration of oak and/or oxygen, by replacing two sides of the Stakvat (each of approximately 0.83 m^2) with oak or plastic (food-grade polyethylene) panels; with or without the addition of oak alternatives and/or MOX.

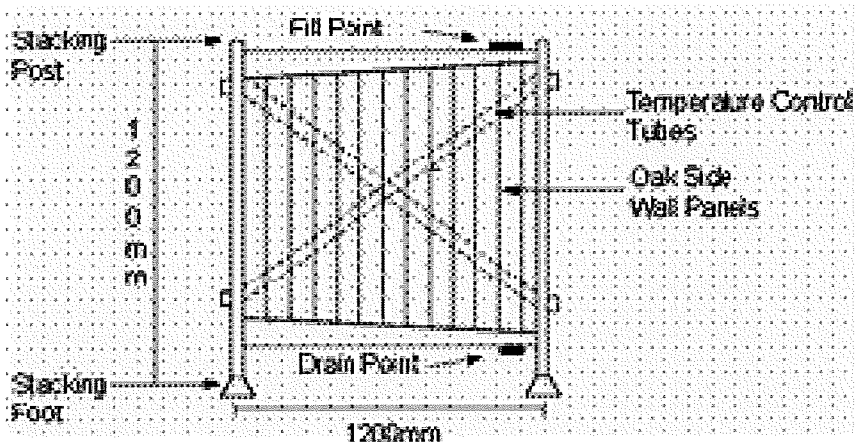

Figure 1. Schematic diagram of an oak-sided Stakvat.

A maturation trial was carried out to evaluate the aging potential of different types of Stakvats, using a combination of chemical and sensory analyses to determine the impact on wine composition and organoleptic properties. The specific objective of the trial was to evaluate three different panel types, i.e. stainless steel, plastic and oak wood, and the potential of each to produce wines of comparable composition and quality to those aged traditionally in barrels.

3.2 Materials and Methods

3.2.1 Wine maturation

Two wines, a 2010 McLaren Vale Shiraz and a 2010 McLaren Vale Cabernet Sauvignon, were each matured for 12 months (from June 2010 to June 2011) in three types of Stakvat (in triplicate) being: (i) stainless steel, (ii) stainless steel with two plastic panels, i.e. 4 mm high density polyethylene (HDPE), and (iii) stainless steel with two French oak wood panels (Figure 2, Figure 3). Prior to the trial commencing, samples of untreated (i.e. control) wine were bottled and cellared at 15°C. French oak staves were inserted in the Stakvats with stainless steel and plastic panels (but not oak-sided Stakvats). The same batch of French oak wood was used for the Stakvats with oak-sided panels (for maturation of both Shiraz and Cabernet Sauvignon wines) and the staves inserted into the stainless steel and plastic-sided Stakvats for maturation of Shiraz wine. However, the winery involved in the maturation trial insisted that the staves inserted into the stainless steel and plastic-sided Stakvats to be used for Cabernet Sauvignon maturation should be "medium plus" toasted French oak staves. Sulphur dioxide levels were monitored regularly (i.e. monthly) by the industry partner and maintained at approximately 20 and 53 ppm free and total SO_2, respectively. After filling, the Stakvats were stored at temperature conditions maintained between 15 and 18°C. Wine (100 mL per Stakvat) was collected for chemical analyses after 3, 6, 9 and 12 months maturation. Following maturation, i.e. at 12 months, wines (36 L per Stakvat) were bottled for sensory analysis. However, two replicates of the Shiraz wine matured in stainless steel sided Stakvats were found to be missing; unfortunately they had been bottled by the winery for commercial purposes two weeks prior to completion of the maturation trial. Bottled wines were cellared for approximately 3 months (at 15°C) until required for sensory analysis, at which time chemical analyses were repeated.

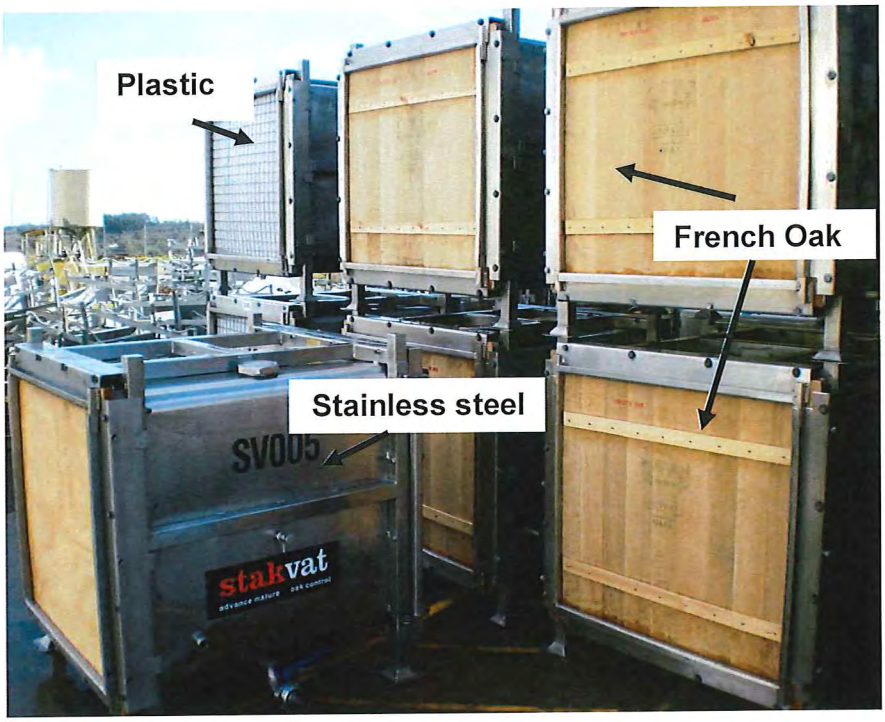

Figure 2. Stainless steel, oak and plastic-sided Stakvats.

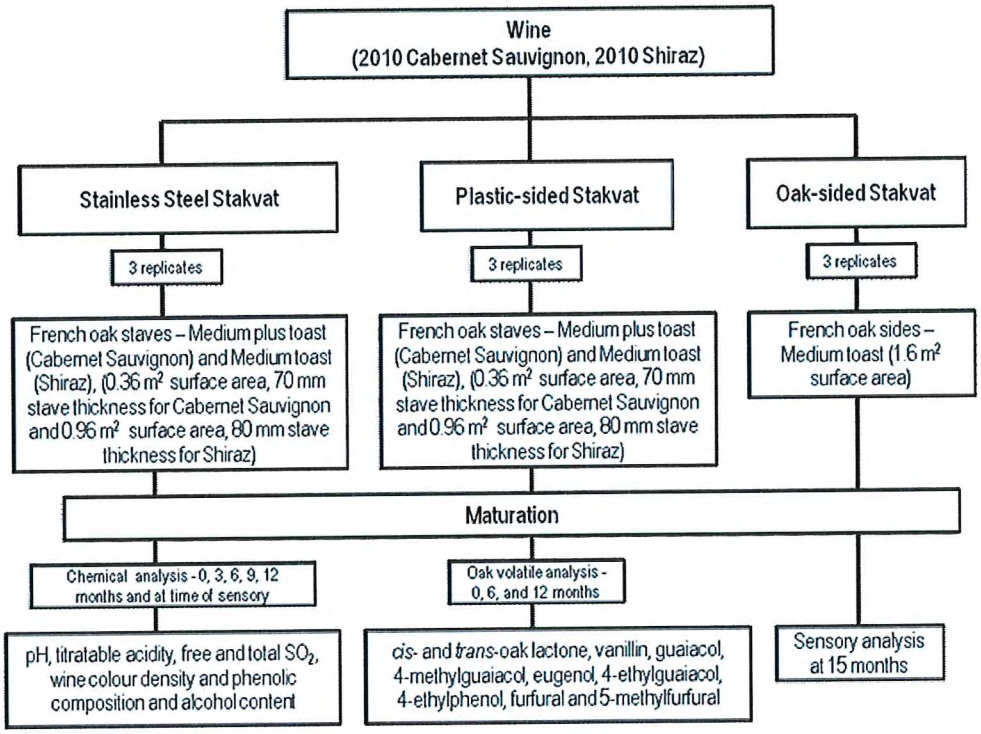

Figure 3. Flow diagram of the experimental design.

Treatments were initially established in triplicate, but only one sample of Shiraz wine matured in stainless steel-sided Stakvats remained at completion of the maturation trial.

3.2.2 *Compositional analysis of wines*

Wine samples were analysed (in duplicate) to determine pH, titratable acidity (as tartaric acid equivalents to an end point of pH 8.2), free and total SO_2 content (by the aspiration method), wine colour density and phenolic composition (Iland *et al.* 2004). Alcohol content (% v/v) was measured using an alcolyzer (Anton Paar, Graz, Austria). The concentrations of *cis*- and *trans*-oak lactone, vanillin, guaiacol, 4-methylguaiacol, 4-ethylguaiacol, 4-ethylphenol, eugenol, furfural and 5-methylfurfural were determined by the Australian Wine Research Institute's (AWRI) Commercial Services Laboratory (Adelaide, Australia), according to the SIDA methods reported previously (Pollnitz, 2000; Pollnitz *et al.*, 2000; Pollnitz *et al.*, 2004). These publications describe the preparation of internal standards, method validation and instrument operating conditions used herein. Analyses were performed using an Agilent 6890 Gas Chromatograph coupled to a 5973 Mass Selective Detector.

3.2.3 *Sensory analysis of wines*

Difference testing

Difference tests were conducted on all Cabernet Sauvignon wines using the triangle test method (Meilgaard *et al.* 2007) to establish whether or not there were perceivable differences between control (unoaked) wines, and wines matured for 12 months in the different types of Stakvat. Wines (30 mL) were served at 22-24°C, in covered 215 mL ISO tasting glasses. Samples were assigned random three digit codes and assessed in isolated, temperature-controlled booths, under red-light illumination. Wines were presented to a panel of 24 judges using a balanced presentation order, comprising all possible configurations (i.e. AAB, ABA, BAA, BBA, BAB and ABB, where A denotes the control wine and B denotes a Stakvat treated wine). Panellists were asked to smell and taste the samples, and to then identify the sample which was different.

Descriptive Analysis

Descriptive Analysis (DA) of all Cabernet Sauvignon and Shiraz wines was undertaken approximately three months after bottling, using a panel comprising ten University of Adelaide students, enrolled in postgraduate viticulture or oenology degrees. Prior to formal assessment, panellists underwent 20 hours of training (comprising 10 x 2 hour sessions held over 6 weeks) involving the detection, identification, evaluation and intensity rating of red wine aroma and palate (i.e. flavour, taste and mouth-feel) attributes. During the training sessions, the panel evaluated each wine at least twice, to generate appropriate aroma and palate attributes that discriminated the wines. The panel agreed upon 10 aroma attributes and 15 palate attributes, with accompanying definitions (Table 3). Panellists were unable to perceive any differences in wine colour, so colour was not evaluated. Wines (30 mL) were served at 22-24°C, in covered 215 mL ISO tasting glasses. Samples were assigned random three digit codes and assessed in isolated, temperature-controlled booths, under red-light illumination. Wines were presented to the panel in duplicate using a balanced presentation order. Panellists were asked to rate each attribute using a 15 cm unstructured line scale with anchor points of "low" and "high" placed at 10% and 90%, respectively. Sensory reference standards (Table 4) were prepared in 30 mL of red wine and were provided to panellists during training sessions and at each formal evaluation. Panellists were encouraged to re-acquaint themselves with the sensory reference standards and written definitions of attributes several times during each evaluation. Distilled water and crackers were provided as palate cleansers and panellists were required to have a 1 minute break between samples, and a 5 minute break every 3 samples. Sensory data was collected using FIZZ software (Version 2.40 E, Biosystemes, Couternon, France).

3.2.4 Statistical Analysis

Statistical and multivariate data analyses were conducted using Senpaq (Version 5.01, Qi Statistics, United Kingdom) and XLSTAT (Version 2013.4.03, Addinsoft). Analysis of Variance (ANOVA) was performed on the data, to assess whether the chemical, volatile and sensory mean scores were

significantly different from each other. Principal Components Analysis (PCA) was used to identify the most important sensory differences between samples and to explore the similarities and groupings in relation to the attributes.

Table 3. Aroma, palate and mouth-feel attributes used for descriptive analysis of wines.

Aroma attributes	Definition	Anchor
Red fruit	Aroma of fresh red fruits: raspberry, strawberry, red currant and plum	Low–High
Black fruit	Aroma of dark fruits: blackberry, black cherry, blackcurrant and black olive	Low–High
Vegetal green	Grassy, green bean, green capsicum, stalky, tomato leaf, asparagus	Low–High
Herbaceous green	Aroma of dried herbs	Low–High
'Other' green	Eucalypt, menthol	Low–High
Toasty oak	Aroma of toasted oak	Low–High
Sweet oak	Vanilla, coconut, mocha	Low–High
Woody oak	Pencil shavings	Low–High
Savoury	Aroma of smoked meat: bacon	Low–High
Spice	Mixed spice, black peppercorns and white pepper	Low-High
Palate attributes	**Definition**	**Anchor**
Red fruit	Fresh red fruits: raspberry, strawberry, red currant and plum	Low–High
Black fruit	Dark fruits: blackberry, black cherry, blackcurrant and black olive	Low–High
Green	Any green perceived, could be vegetal, herbaceous or 'other'	Low–High
Spice	Mixed spice, black peppercorns and white pepper	Low–High
Toasty oak	Aroma of toasted oak	Low–High
Sweet oak	Vanilla, coconut, mocha	Low–High
Woody oak	Pencil shavings	Low–High
Savoury	Smoked meat	Low–High
Astringency	Perception of drying or puckering sensation	Not drying–Drying
Bitter	Perception of bitterness	Low–High
Acid	Level of acid perceived	Low–High
Tannin quality	Mouth-feel perception of tannins	Silky/Smooth-Suede/Coarse
Body	Weight	Light bodied–Full bodied
Length of fruit	Length of time fruit characters are perceived after expectoration	0–20 secs
Length of 'other'	Length of time 'other' attributes are perceived after expectoration	0–20 secs

Table 4. Reference standards used for formal sensory analysis of wines.

Attribute	Standard
Vegetal green	6 cm of rachis (grape bunch stem) chopped in 1 cm lengths;1 tbsp fresh grass; 2 cm piece of green bean; 2 cm piece of asparagus; 1 cm^2 piece of green capsicum; 1 tomato leaf
Herbaceous green	¼ tsp mixed dried herbs, dill and thyme
'Other' green	1 drop of eucalypt solution (1 drop eucalypt oil in 2 L water). 1/8 tsp Vicks VapoRub
Toasty oak	0.25g heavy toasted American oak chips + 0.25g heavy toasted French oak chips in 100 mL wine
Sweet oak	1 drop coconut essence + 1 drop vanilla essence + ¼ tsp mocha (Moccona Mochaccino)
Woody oak	½ tsp pencil shavings
Red fruit	2 frozen raspberry, 2 frozen strawberry, 1 small piece red plum, 4 frozen red currants
Dark fruit	2 frozen blackberry, 2 frozen black cherry, 4 frozen black currants, 6 black olives + 5ml brine
Spice	¼ tsp mixed spice (McKenzie's) + 1 grind black peppercorn + 1/8 tsp white pepper
Savoury	1 tsp chopped bacon (uncooked)

Samples prepared in 30 mL red wine.

3.3 Results and Discussion

The maturation trial was originally intended to evaluate Stakvat treatments that would utilise oak from the same batch and source, to minimise differences between experimental treatments. However, the industry partner involved in the trial insisted that "medium plus" toasted French oak staves were to be utilised for the stainless steel and plastic-sided Stakvats used for maturation of Cabernet Sauvignon wine. The experimental rigour of the trial was further compromised when two replicates of Shiraz wine aged in stainless steel-sided Stakvats were bottled by the industry partner after 11 months maturation, leaving only one replicate of this treatment for chemical and sensory analysis at the conclusion of the trial. Nonetheless, the results obtained from this trial are presented because valid comparisons could still be obtained between the Cabernet Sauvignon plastic and stainless steel treatments and Shiraz plastic and oak treatments, albeit not between Cabernet Sauvignon and Shiraz oak treatments and Shiraz stainless steel treatments.

3.3.1 Compositional analysis of wines

Compositional analysis of wines was performed after 0, 3, 6, 9 and 12 months of maturation and at the time of sensory analysis, to determine the influence of the type of Stakvat and the duration of

maturation on basic wine chemistry parameters. No meaningful differences were observed between Stakvat treatments, such that the pH, TA, alcohol, colour, phenolic and sulphur dioxide content of Cabernet Sauvignon and Shiraz wines (respectively) were comparable after 12 months maturation (Table 5). Wine compositions were also similar to those reported for commercial wines (Lattey *et al.*, 2010), irrespective of the maturation method and duration. Similarly, no significant differences were observed between measurements taken at 3, 6 and 12 months (data not shown).

In contrast, differences were observed for measurements of total phenolics and total red pigments between the control and corresponding oak aged wines throughout the maturation period (data not shown), with the levels of each decreasing over time. This is most likely due to the tannins precipitating and/or the further reaction of phenolic compounds during the maturation period (Somers, 1971). As wine ages, anthocyanins are converted into more stable pigments which result in colour modifications (bright red transforms into brick-red hues) (Monagas *et al.*, 2006).

Table 5. Chemical composition of Cabernet Sauvignon and Shiraz wines after maturation in different types of Stakvat.

	Treatment	pH	TA (g/L)	Alcohol (% v/v)	Colour density	Total red pigments (mg/L)	Total phenolics (au)	Free SO_2 (mg/L)	Total SO_2 (mg/L)
Cabernet Sauvignon	Initial wine	3.6	8.2 ± 0.1	13.1	154 ± 5.5	41	69 ± 0.6	29 ± 1.5	46 ± 1.5
	Plastic (HDPE)	3.4	5.9 ± 0.1	13.8	125 ± 2.5	17	52 ± 0.6	17 ± 3.2	38 ± 3.5
	Stainless Steel	3.3	5.9 ± 0.2	13.8	120 ± 3.5	17	52 ± 1.0	13 ± 0.6	31 ± 4.6
	Oak Wood	3.4	5.8 ± 0.1	13.8	125 ± 6.2	16	51 ± 0.6	14 ± 1.5	35 ± 3.5
Shiraz	Initial wine	3.6	7.7 ± 0.2	14.4	164 ± 2.0	43	73 ± 2.0	31 ± 5.8	42 ± 5.8
	Plastic (HDPE)	3.3	5.5 ± 0.1	14.6	118 ± 3.5	18	52 ± 4.0	15 ± 1.2	35 ± 4.5
	Stainless Steel*	3.4	5.6	14.6	119	18	55	16	30
	Oak Wood	3.4	5.5 ± 0.3	14.6	124 ± 4.9	19	55 ± 0.6	17 ± 1.7	30 ± 3.5

Values are means of three replicates (± standard deviation), unless otherwise stated (i.e. * denotes one sample only). Data for initial wines were obtained from analyses performed prior to maturation (i.e. at t=0 months). Data for oak-aged wines were obtained from analyses performed at the time of sensory.

The concentrations of key oak volatiles were measured to determine the influence of different Stakvat treatments on wine composition (Table 6). Prior to maturation, the wines contained virtually none of the oak volatiles measured, as would be expected of an unoaked wine. Traces of guaiacol were detected in both the Cabernet Sauvignon and Shiraz varieties, in agreement with levels reported in previous studies (Wilkinson *et al.*, 2011). For Cabernet Sauvignon wines, statistically significant differences were observed between the concentrations of *cis*- and *trans*-oak lactone, vanillin, furfural and 5-methylfurfural; with higher concentrations observed in wines aged in oak-sided Stakvats, compared to the other treatments. This can be attributed at least in part to the different toasting intensities and increased surface area of oak wood, being 1.6 m^2 for oak-sided Stakvats, but only 0.36 m^2 for stainless steel and plastic-sided Stakvats. Increased oak volatile levels were observed in all Cabernet Sauvignon wines following 12 months maturation. The concentrations of oak lactone in Cabernet Sauvignon wines aged in stainless steel and plastic-sided Stakvats was at or below the analytical limit of quantification (i.e. 10 µg/L), so the levels observed are unreliable, but clearly different from those observed in wines aged in oak-sided Stakvats. This suggests the inserted staves contained only low levels of oak lactone and its precursors (Wilkinson *et al.*, 2004). The oak lactone content of oak wood has been shown to vary considerably; a 2002 study investigated oak lactone levels in 133 oak samples, and found concentrations ranged from 0.4 to 181 µg/g (Doussot *et al.*, 2002).

For Shiraz wines, similar levels of oak lactone, guaiacol and 4-methylguaiacol were observed after six months maturation. Higher concentrations of vanillin, furfural and 5-methylfurfural were observed in wines aged in oak-sided Stakvats, than in wines aged in stainless steel or plastic-sided Stakvats. Again, this is likely due to differences in the surface area of oak wood for the different types of Stakvat (Figure 3) and/or variation between the initial pool of volatiles available for extraction, although the exact reasons behind the compositional changes in aromatic aldehydes remains unclear.

Table 6. Concentrations of oak volatiles in Cabernet Sauvignon and Shiraz wines after 0, 6 and 12 months maturation in different types of Stakvat.

	Treatment	Duration of maturation (months)	Concentration (µg/L)									
			cis-oak lactone	*trans*-oak lactone	vanillin	guaiacol	4-methyl guaiacol	eugenol	4-ethyl guaiacol	4-ethyl phenol	furfural	5-methyl furfural
Cabernet Sauvignon	Control*	0	nd	17	nd	1	nd	nd	nd	nd	20	nd
	Plastic (HDPE)	6	nd	nd	47 ± 4 b	3 ± 0.57	4 ± 1 a	nd	nd	nd	211 ± 15 b	35 ± 7 b
	Stainless Steel	6	4 ± 6 b	10 ± 1 b	52 ± 1 b	4 ± 0.57	4 ± 1 a	nd	nd	nd	202 ± 22 b	30 ± 4 b
	Oak Wood	6	22 ± 2 a	25 ± 3 a	85 ± 3 a	3 ± 0.57	3 ± 1 b	nd	nd	nd	292 ± 27 a	90 ± 14 a
	P value		0.001	<0.0001	<0.0001	0.729	0.020				0.004	0.001
	Plastic (HDPE)	12	nd	nd	68 ± 6 b	6 ± 1	6 ± 1 a	nd	nd	nd	200 ± 30	53 ± 12 b
	Stainless Steel	12	9 ± 8 b	nd	72 ± 6 b	7 ± 1	7 ± 1 a	nd	nd	nd	241 ± 96	46 ± 9 b
	Oak Wood	12	51 ± 6 a	40 ± 6 a	143 ± 9 a	7 ± 1	4 ± 1 b	nd	nd	nd	304 ± 95	98 ± 17 a
	P value		< 0.0001	< 0.0001	< 0.0001	0.729	0.001				0.341	0.005
Shiraz	Control*	0	nd	nd	25	7	nd	nd	nd	nd	25	nd
	Plastic (HDPE)	6	22 ± 4	19 ± 5	56 ± 7 b	9 ± 1	2 ± 1 b	nd	nd	nd	129 ± 3 b	43 ± 5 b
	Stainless Steel	6	25 ± 7	25 ± 3	54 ± 0 b	9 ± 0	2 ± 1 b	nd	nd	nd	187 ± 60 b	60 ± 6 b
	Oak Wood	6	23 ± 11	23 ± 11	104 ± 8 a	10 ± 0	3 ± 1 a	nd	nd	nd	351 ± 43 a	99 ± 15 a
	P value		0.903	0.138	< 0.0001	0.125	0.079				0.002	0.001
	Plastic (HDPE)	12	37 ± 11	17 ± 5 b	65 ± 5 b	15 ± 1 b	2 ± 1 b	nd	21 ± 6 b	165 ± 51	29 ± 11	6 ± 10
	Stainless Steel*	12	33	47	74	15	2	nd	nd	99	105	35
	Oak Wood	12	53 ± 29	42 ± 8 a	171 ± 6 a	17 ± 1 a	3 ± 1 a	3 ± 6	62 ± 30 a	803 ± 767	24 ± 5	4 ± 8
	P value		0.120	0.174	0.001	0.061	0.013		0.015	0.156	0.935	0.800
	Wine Concentrations†		47-1285	14-587	26-770	11-72	3-23	6-114	1-69	3-680	14-2121	9-270

Values are means of three replicates (± standard deviation), unless otherwise stated (i.e. * denotes single sample only). Different letters within a column indicate a statistically significant difference (for each wine and for each time point) as determined by Fishers post hoc test; nd = not detected (i.e. <10 µg/L). †(Rodriguez-Rodriguez *et al.*, 359; Pollnitz, 2000; Garde Cerdan *et al.*, 2002; Garde Cerdan *et al.*, 2010; Castro-Vazquez *et al.*, 2011; Martinez-Gil *et al.*, 2012).

Following 12 months maturation, the levels of *cis*-oak lactone, vanillin and guaiacol increased in the plastic- and oak-sided treatments, but conclusions can't be drawn on with respect to the stainless steel treatment, due to only one sample being available. Again, wines aged in oak-sided Stakvats contained the highest concentrations of these oak volatiles. Similar levels of *trans*-oak lactone were obtained for wines aged in stainless steel and oak-sided Stakvats, albeit there was only a single sample available for the stainless steel treatment. The concentrations of furfural and 5-methylfurfural decreased over time; the largest reduction occurred in wines from the oak-sided treatment, for which concentrations decreased from 351 to 24 µg/L. However, this was not unexpected given these volatiles have previously been found to be readily converted to their corresponding alcohols as a consequence of microbiological activity (Spillman *et al.*, 1998). During the 6 to 12 month maturation period, significant levels of 4-ethylguaiacol and 4-ethylphenol were produced in the Shiraz wines, presumably due to a moderate level of *Brettanomyces* growth (Chatonnet *et al.*, 1992). That higher levels of 4-ethylguaiacol and 4-ethylphenol were observed in wines from the plastic and oak-sided Stakvats suggests the extent of *Brettanomyces* growth may have been influenced by oxygen ingress and/or depletion of sulphur dioxide; albeit total SO_2 levels were maintained at approximately 45 mg/L and 60 mg/L during maturation for the Shiraz and Cabernet Sauvignon wines respectively (data not shown). It is possible that *Brettanomyces* may have been present in the wine prior to treatment; alternatively the wine composition may have enabled *Brettanomyces* to flourish, e.g. due to low level differences in residual sugar concentrations. The standard deviation calculated for 4-ethylphenol in Shiraz wine aged in oak-sided Stakvats was exceptionally high due to only having one sample; but it is not unexpected to see random microbiological growth in individual vessels. Careful monitoring of sulphur dioxide levels remains an essential cellar management practice, when using Stakvats in lieu of oak barrels for maturation of wine. Trace (i.e. 3 µg/L) levels of eugenol were detected only in wines from the oak-sided treatment after 12 months maturation. However, at this level, eugenol is unlikely to have any perceivable impact on the organoleptic characteristics of these wines.

For most of the oak volatiles measured, concentrations increased during the maturation period, irrespective of wine variety (i.e. Cabernet Sauvignon vs. Shiraz) or the maturation treatment. The levels of furfural and 5-methylfurfural decreased in Shiraz wines with time, presumably due to their conversion into their respective alcohols. However, this was not observed in Cabernet Sauvignon wines, where the levels of these volatiles remained similar or increased during the 6 to 12 month maturation period. The depletion of these volatiles in the Shiraz wine could be due to *Brettanomyces* growth (or factors that allow *Brettanomyces* to flourish) which facilitated their reduction to their corresponding alcohols. The highest concentrations of oak volatiles were typically observed in wines matured in oak-sided Stakvats, again irrespective of variety. This likely reflects differences in the surface area of oak wood between treatments; i.e. 1.6 m^2 for oak-sided Stakvats, compared to 0.36 m^2 or 0.96 m^2 for the stainless steel and plastic-sided Stakvats used for Cabernet Sauvignon (0.36 m^2) and Shiraz (0.96 m^2) wines (Figure 3). The sensory effect of individual oak volatile compounds is difficult to ascertain due to the variation in detection thresholds reported in the literature. Earlier reported thresholds were quite high, whereas subsequent studies suggested thresholds were in fact considerably lower. For example, aroma detection thresholds of 92 and 95 µg/L were reported for *cis*-oak lactone (as a mixture of two diastereomers) and guaiacol (in white wine) respectively (Boidron *et al.*, 1988; Chatonnet *et al.*, 1990); but lower levels of 23 and 10 µg/L have subsequently been reported for the nature identical (4S,5S)-isomer of *cis*-oak lactone and guaiacol, respectively (Ferreira *et al.*, 2000; Wilkinson *et al.*, 2004). For this reason, sensory studies including descriptive analysis were undertaken to investigate the intensity of oak-related sensory attributes in Cabernet Sauvignon and Shiraz wines.

3.3.2 Sensory Analysis

Triangle tests were carried out on Cabernet Sauvignon wines to establish whether or not there were perceivable differences between control (i.e. unoaked) wines, and wines matured for 12 months in the different types of Stakvat; i.e. as a preliminary experiment to determine whether descriptive sensory analysis would be worthwhile. Panellists were unable to distinguish the Cabernet Sauvignon wine

matured in the plastic-sided Stakvat from the control wine, but 15 of 24 judges correctly differentiated wines aged in stainless steel or oak-sided Stakvats from the control wine (Table 7), indicating these wines were significantly different (at ≥99% confidence levels). Differences were apparent between control and treated Shiraz wines during bench top tasting, partly as a consequence of the *Brettanomyces* growth, thus difference tests were not conducted for this variety.

Table 7. Difference test scores for Cabernet Sauvignon wines matured for 12 months in different types of Stakvats against the control wine.

Treatment	Correctly identified 'different' sample (n=24)	Significance
Plastic (HDPE)	10	$P = 0.4$
Stainless Steel	15	$P = 0.01$
Oak Wood	16	$P = 0.001$

DA was performed in order to characterise differences in the aroma and palate attributes of Cabernet Sauvignon and Shiraz wines obtained following 12 months maturation in the different types of Stakvat. The primary objective of DA was to establish the nature and intensity of differences between wines from different maturation regimes using a trained panel.

During DA training sessions, the panel identified a range of key aroma and palate attributes, including red fruit, dark fruit and savoury aromas and flavours, as well as tannin quality, that defined the various wines. Some attributes were indicative of varietal expression, e.g. '*spice*' aroma and flavour (for Shiraz), and '*herbaceous*' and '*vegetal*' green attributes (for Cabernet Sauvignon). Aroma and flavour attributes that related specifically to oak treatment included: '*sweet oak*', '*woody oak*' and '*toasty oak*' (Table 3). Following formal assessments, ANOVA was performed on intensity rating data obtained for Cabernet Sauvignon and Shiraz wines. Data from two panellists was excluded on the basis of poor (inconsistent) performance and the large number of interactions that resulted.

For Cabernet Sauvignon wines, 9 attributes were found to have statistically significant differences between samples (i.e. at $P \leq 0.2$, significant differences were not detected at $P \leq 0.1$). These attributes were therefore retained for further analysis (Figure 4). Surprisingly, the control wine received similar intensity ratings for each of the oak-related sensory attributes as the wines aged in different Stakvat treatments. This may indicate the panel required further training to better differentiate these attributes, since the concentrations of oak volatiles observed in the oak-aged wines suggested perceptible differences might be expected.

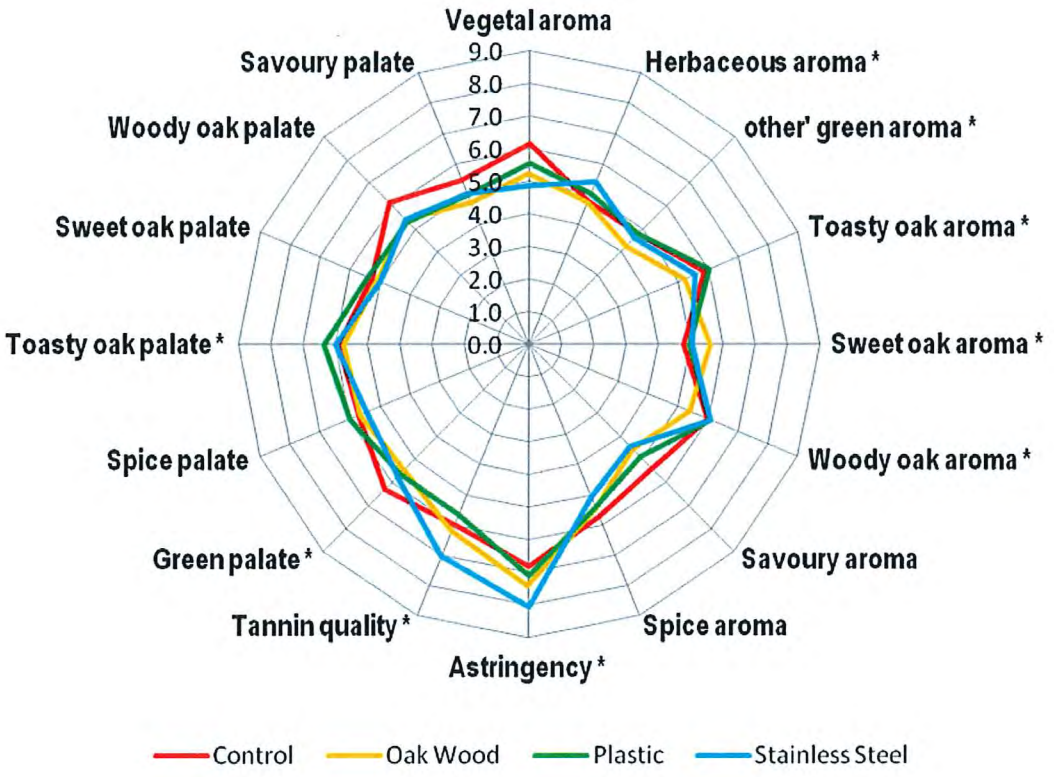

Figure 4. Mean ratings of sensory attributes in Cabernet Sauvignon wines matured in different types of Stakvat for 12 months.

Values are mean ratings for 3 wine replicates assessed by 8 judges during 3 replicate sensory evaluations.

* indicates a statistical significance at $P \leq 0.2$, as determined by Fisher's LSD for multiple comparisons.

The Cabernet Sauvignon wines aged in the stainless steel Stakvats were scored significantly higher in '*herbaceous green*' attributes than the corresponding wine aged in the oak-sided Stakvat; while the

control wine received significantly higher ratings for *'green'* aroma and flavour. Green characters can be considered varietal characteristics of Cabernet Sauvignon wines, so this result was not unexpected, and instead suggests the potential for green characters to be masked by the influence of oak. Similar results were reported by Aiken and Noble in a previous study involving the addition of oak chips to Cabernet Sauvignon during fermentation (Aiken *et al.*, 1984). Wines aged in the plastic-sided Stakvat exhibited *'woody oak'* aroma and *'toasty oak'* aroma and flavour, but received the lowest rating for *'sweet oak'* attributes. The stainless steel Stakvats yielded wines with significantly higher *astringency* and *tannin quality ratings*. Although the oak-aged Cabernet Sauvignon wines were not found to exhibit prominent oak-related sensory attributes, particularly compared to the corresponding control wine, the oak treatment appears to have reduced the intensity of green characters, suggesting some aroma and flavour suppression has occurred. The different sources and surface areas of oak wood used for the maturation of Cabernet Sauvignon undoubtedly confounds interpretation of chemical and sensory data, and therefore, the true impact of the different types of Stakvat on wine composition and sensory properties.

Principal Component Analysis (PCA) was performed on the mean intensity ratings obtained from the 8 judges, for the statistically significant Cabernet Sauvignon attributes; the first two PCs explained 74% of the variation in sensory data (Figure 5). The bi-plot shows clustering of the oak-related sensory attributes on the right, except for *'sweet oak'* which appeared on the left side. The wine aged in oak-sided Stakvats was best characterised by *'sweet oak'* aromas, while the wine aged in the plastic-sided Stakvat exhibited more *'toasty oak'* aromas and flavours. This is likely due to the different sources of oak used in these treatments, as described in section 1.3. The control wine and the wine aged in stainless steel Stakvats were grouped close to the various *'green'* attributes, which suggests the aroma and flavour of these wines was driven by varietal characteristics rather than any apparent influence of oak.

Compositional analysis of the oak volatile content of these wines (shown in Table 6) indicated the oak-sided Stakvat yielded significantly higher levels of oak lactone and vanillin, which could account for the resultant wine's correlation with the '*sweet oak*' descriptor. In contrast, the stainless steel Stakvat yielded lower levels of oak volatiles, which might explain the corresponding wine's close association with '*green*' *aromas*; i.e. attributes commonly associated with the varietal expression of Cabernet Sauvignon wines. Furthermore, the reduced oxygen ingress expected for this treatment may also explain the increased perception of astringency; albeit the DA panel didn't seem to associate enhanced astringency with reduced tannin quality.

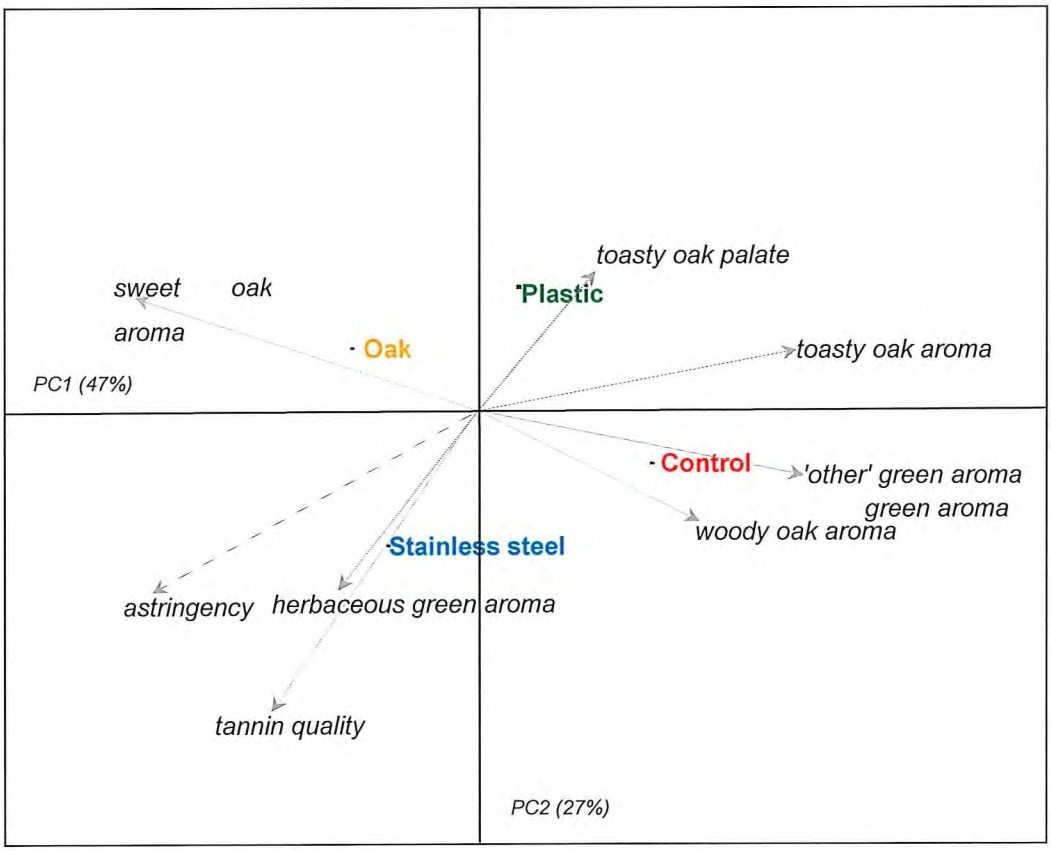

Figure 5. PCA bi-plot of the mean sensory data obtained for Cabernet Sauvignon wines aged in different types of Stakvat; PC1 versus PC2.

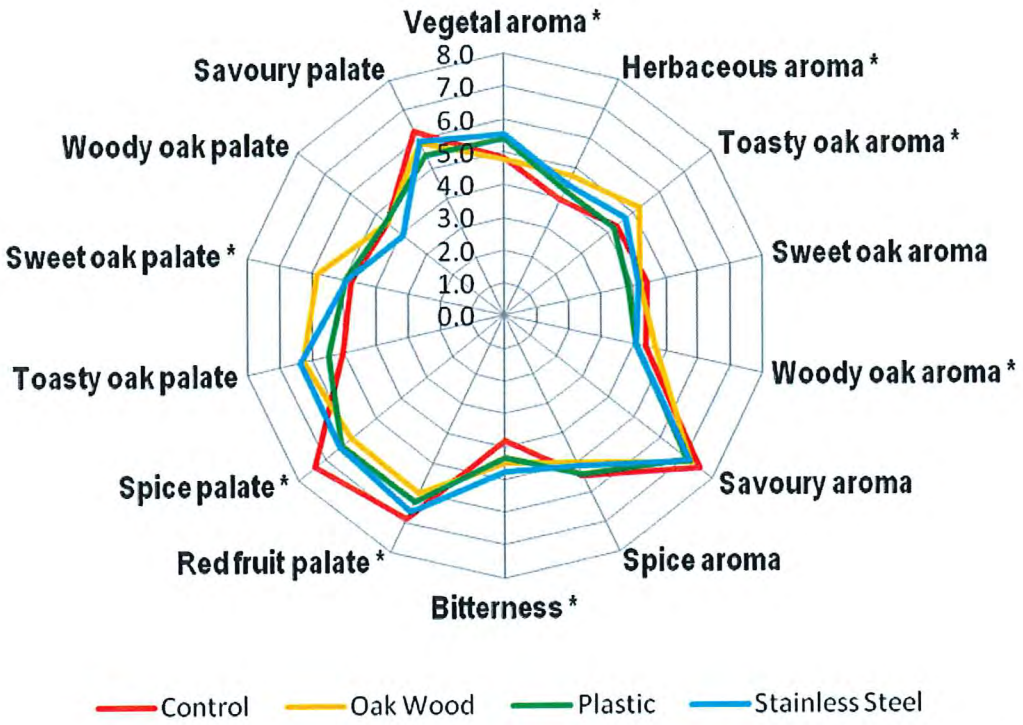

Figure 6. Mean ratings of sensory attributes in Shiraz wines matured in different types of Stakvat for 12 months.

Values are mean ratings for 3 wine replicates assessed by 8 judges during 3 replicate sensory evaluations.
* Indicates a statistical significance at $P \leq 0.2$, as determined by Fisher's LSD for multiple comparisons.

The DA panel also rated the intensity of 25 sensory attributes for each Shiraz wine, and 8 attributes were found to differ significantly ($P \leq 0.2$) between treatments (Figure 6). Again, the control wine received similar intensity scores for oak-related sensory attributes compared to the oak aged wines. As for the Cabernet wines, this could indicate the panel required further training to better differentiate these attributes. However, Shiraz wines also exhibited a notable bitterness, which may have influenced panellists' ability to discriminate samples. The DA panel generally considered the oak-aged Shiraz wines more intense in oak-related attributes, i.e. '*toasty oak*' and '*woody oak*' aromas and '*sweet oak*' flavour, compared to the control wine; albeit, the wine matured in the plastic-sided Stakvat received slightly lower '*toasty oak*' and '*woody oak*' scores and the wine aged in the stainless steel Stakvat was scored lower for '*woody oak*' aroma. The oak-sided Stakvat produced the wine with the most intense 'sweet oak' flavour. Again the stainless steel and plastic-sided Stakvats produced wines highest in

'*vegetal green*' aroma, while the oak-sided Stakvat gave wine with the highest '*herbaceous green*' aroma. The stainless steel Stakvat treatment resulted in the most bitter wine, relative to the control wine; albeit the intensity was not significantly different from the other Stakvat treatments. The control wine exhibited the highest '*red fruit*' and 'spice' attributes on the palate.

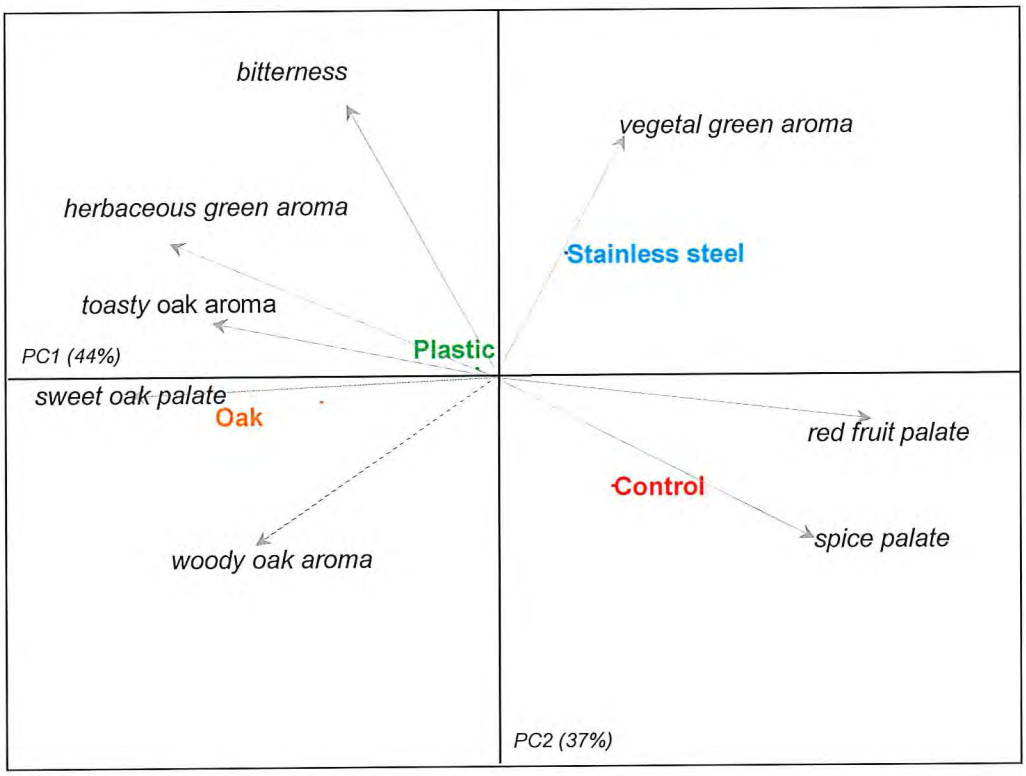

Figure 7. PCA bi-plot of the mean sensory data obtained for Shiraz wines aged in different types of Stakvat; PC1 versus PC2.

PCA was performed on the mean intensity ratings obtained from the 8 judges, for the statistically significant Shiraz attributes; the first two PCs explained 81% of the variation in sensory data (Figure 7). The bi-plot shows clustering of the oak attributes in the two left quadrants and the wines matured in plastic-sided and oak-sided Stakvats are situated in close proximity. This indicates the panel could readily discriminate oaked and un-oaked Shiraz wines, with PC1 displaying separation of wines on the basis of oak-derived vs. fruit-derived aromas and flavours. The wine aged in oak-sided Stakvats, and to a lesser extent, wine aged in plastic-sided Stakvats, were characterised by '*sweet*', '*toasty*' and '*woody*'

oak attributes (Figure 7); attributable to the higher concentrations of oak volatiles observed in these wines (Table 6), oak lactone and vanillin in particular. In contrast, the control wine was situated on the right side of the plot, in close proximity to *'red fruit'* and *'spice'* descriptors; while wines from the stainless steel Stakvat were closely correlated with *'vegetal green'* notes. These wines had comparatively lower concentrations of oak volatiles (at least compared with the wines from the oak-sided Stakvat), which might explain why panellists instead perceived more intense *'red fruit'*, *'spice'* and/or *'green'*, rather than oak characters.

From the DA results it can be concluded for both wine varieties, that the different Stakvat treatments were able to impart oak volatiles and oak-related sensory attributes to wines, with the influence of oak most evident in wines matured in the plastic-sided and oak-sided Stakvats. Control wines and wines aged in stainless steel Stakvats were instead driven by varietal characteristics, such as *'red fruit'*, *'green'* and *'spicy'* aromas and/or flavours. For control wines, this was simply due to the absence of oak treatment, but, for in the case of stainless steel treatment, this may reflect the lack of oxygen ingress for this vessel.

Although difference testing suggested two of the treatments produced wines that exhibited significantly different sensory properties from the control (Table 7), large differences were not observed by DA, evidenced by variation at a significance level of 0.2. Panellist performance may have improved following additional training, in particular with regards to differentiation of the oak-specific sensory attributes, but given the existing limitations imposed on the maturation trial, this was not pursued. Informal comments from participants from both the different tests and DA indicated the wines were considered to be quite bitter, which influenced the DA panels' ability to reproducibly rate palate attributes. In the case of Shiraz wines, GC-MS analysis indicated probable *Brettanomyces* growth, based on the presence of 4-ethylguaiacol and 4-ethylphenol.

3.4 Conclusions

The maturation trial demonstrated that the different types of Stakvat can impart oak characters to wine and therefore offer winemakers an alternative method for the oak maturation of wine. In this study, the oak type and surface area appeared to have a greater influence on the concentration of oak volatiles observed in wines, than the type of Stakvat used, despite methodological issues that resulted from conditions imposed by the industry partner.

In the case of the Shiraz wines, for which the same batch of oak was used for all treatments, the concentrations of several key oak volatiles were found to be significantly higher in wine from the oak-sided Stakvat than in wines aged in stainless steel or plastic-sided Stakvats. Compositional analyses reflected DA results; i.e. oak-related sensory attributes were more prominent in wines matured in oak-sided Stakvat. This is likely due to a combination of increased oak wood surface area as well as oxygen ingress for this Stakvat; especially compared to the stainless steel Stakvat, which would allow very little oxygen (if any) to permeate into the wine. It would be interesting to repeat the maturation trial with a treatment that involved both oak and MOX in the stainless steel Stakvat.

Ideally, wines matured with barrel alternatives would also be evaluated alongside wines aged traditionally in oak barrels, but again, the inherent variation in oak composition, especially due to different toasting regimes, complicates experimental design.

3.5 Future work

The unintended use of two different types of oak for the maturation of Cabernet Sauvignon wines, together with the loss of two replicates of Shiraz wine aged in stainless steel Stakvats, imposed significant limitations on this study. If such a trial were to be repeated, the following guidelines should be implemented to ensure experimental rigour and validity:

- Each experimental treatment should be conducted in triplicate (at a minimum)
- The same oak wood (i.e. oak species, as well as seasoning and toasting conditions) should be utilised for all treatments
- The oak surface area should be the same across all treatments
- The same wine should be matured in each vessel, with an appropriate control i.e. no oak
- Analysis of pH, TA, ethanol, SO_2, colour, phenolics, sugar and oak volatiles should be performed at 0, 3, 6, 9 and 12 months (for a 12 month maturation trial), and
- Cellar operations (i.e. 'topping up' of vessels) should be performed to monitor SO_2 depletion

In addition to the chemical and sensory analyses described above, consumer acceptance testing of wines could also be performed. The maturation trial could also be conducted over a longer period of time (e.g. 18 or 24 months) to ensure greater differences in wine composition and sensory properties. Compositional analysis of oak wood prior to maturation of wine might also be useful, to determine the flavour potential of oak, as well as the natural variation across staves from the same batch. A subsequent maturation study was performed (as follows), to address these limitations.

3.6 PART 2: Paper 2 - Consumer acceptance, sensory properties and chemical composition of Cabernet Sauvignon wines made using alternative oak maturation regimes

Despite their increased use by the wine industry, few studies have investigated the influence of alternative oak maturation regimes on consumer preferences and acceptability of wine. A recent study compared consumer preferences for wines aged either in oak barrels or with oak chips, and reported considerable disparity between individual consumers' wine preferences (Perez-Magarino *et al.*, 2011). The authors concluded that markets exist for wines made using both oak maturation regimes, since consumers did not significantly reject wines aged with oak chips. However, the outcomes reported in this study were limited due to the relatively small sample size (i.e. n=65) used.

The consumer survey described in Chapter 2 confirmed the majority of wine consumers have a very limited understanding of the role of oak in winemaking. Knowledgeable wine consumers were found to appreciate traditional barrel maturation and were willing to pay a premium for barrel-aged wines, whereas less knowledgeable consumers were accepting of the use of oak chips. A limitation of this study was that it didn't include wine tasting; this therefore presented an opportunity for further work, i.e. to examine whether or not consumer perceptions of oak maturation regimes match their wine preferences. The outcomes of the Stakvat trial (described earlier in Chapter 3), identified further opportunities for investigating the consumer acceptance and sensory properties of wines made using oak alternatives compared to those made traditionally.

To address the limitations of previous studies, this paper describes an investigation into the influence traditional and alternative oak maturation regimes on the composition, sensory properties, quality and consumer acceptance of wine. Through segmentation of the consumer sample and identification of the sensory attributes driving liking, the results of this work can be used by winemakers to tailor their oak

maturation regimes, and deliver marketing strategies which are targeted towards specific segments of the wine market.

3.7 Statement of Authorship

Anna M. Crump, Trent E. Johnson, Kerry, L. Wilkinson, Susan E.P. Bastian (2015). Influence of oak maturation regime on composition, sensory properties, quality and consumer acceptability of Cabernet Sauvignon wines. Journal of Agricultural and Food Chemistry, **63**: 1593-1600.

Anna Crump

Designed experiments, collected samples, conducted sensory, consumer and expert panel tastings, conducted wine chemical analysis, analysed and interpreted sensory, consumer and chemical data, drafted and revised the manuscript.

I hereby certify that the statement of contribution is accurate and I give permission for inclusion of the paper in this thesis.

Signed ..... Date 18/2/15

Trent Johnson

Assisted with experimental design and consumer tastings, analysed and interpreted sensory and consumer data, assisted with revising the manuscript.

I hereby certify that the statement of contribution is accurate and I give permission for inclusion of the paper in this thesis

Signed ..... Date 18/2/15

Kerry Wilkinson

Assisted with experimental design and consumer tastings, analysed and interpreted sensory, consumer and chemical data, assisted with revising the manuscript.

I hereby certify that the statement of contribution is accurate and I give permission for inclusion of the paper in this thesis

Signed ..... Date 18/2/15

Sue Bastian

Assisted with experimental design and consumer tastings, analysed and interpreted sensory and consumer data, assisted with revising the manuscript.

I hereby certify that the statement of contribution is accurate and I give permission for inclusion of the paper in this thesis.

Signed Date 18.2.15

Crump, A.M., Johnson, T.E., Wilkinson, K.L., & Bastian, S.E.P. (2015). Influence of Oak Maturation Regimen on Composition, Sensory Properties, Quality, and Consumer Acceptability of Cabernet Sauvignon Wines.
Journal of Agricultural and Food Chemistry, *63*(5), 1593-1600.

Chapter 4: Consumers' emotional response to oak aromas

4.1 Introduction

For many years, hedonic liking has been widely used by food and beverage industry practitioners to determine consumer preference and acceptability. It has been used to gauge the consumer appeal of new products, to benchmark within a given product category and to gain competitive advantage through tailored marketing campaigns based on product attributes (Lawless *et al.*, 2010). However, the focus of recent sensory and consumer research has seen a shift towards a more holistic approach, which investigates the emotional experiences elicited when products are being consumed (Ferrarini *et al.*, 2010; King *et al.*, 2010; Cardello *et al.*, 2012). The driver behind this research can be attributed to consumers' purchase intent being strongly influenced (either positively or negatively) by their perceptions of a products' impact on their emotional state and wellbeing (Cardello *et al.*, 2012).

Research concerning emotions and eating behaviour has a long history within the field of psychology, and the literature reports numerous studies involving the emotional and psychological drivers for eating (or overeating) a particular food (Kemp *et al.*, 2011; Peters *et al.*, 2011; Salerno *et al.*, 2014). However, measurement of emotions elicited when consuming a food or beverage product remains an emerging field of study for sensory and consumer researchers.

Since 2009, researchers have developed scales for measuring emotions elicited during food or beverage consumption, although as yet, there is no scale that can be confidently applied to all situations. The most well-known and widely used emotions scales for consumer testing include: The EsSense Profile™ (King *et al.*, 2010), ScentMove™ (Porcherot *et al.*, 2010) and The Geneva Emotion and Odor scale (Chrea *et al.*, 2009). More recently, consumer researchers have begun using The Positive and Negative Affect Schedule (PANAS), which is commonly used in psychology research (Kuesten *et al.*, 2014)

Few studies have considered the relationship between wine consumption and emotions, and to date, none have investigated whether specific wine aromas might affect consumer emotions. A recent study developed a list of adjectives that described the emotions of Italian wine consumers (Table 8), with 16 words considered to adequately describe wine consumers' feelings during wine consumption (Ferrarini *et al.*, 2010).

Table 8. Terms used in an Italian consumer study to describe emotions elicited during wine consumption

Pleasant (positive) adjectives	Unpleasant (negative) adjectives
Amusing	Aggressive
Happy	Overwhelming
Euphoric	Disgusting
Joyful	Bland
Keen	
Passionate	
Interesting	
Elegant	
Curious	
Desirable	
Peaceable	
Pleasant	

The study found that for the most part, consuming wine was associated with pleasurable emotions, rather than those emotions linked to unpleasantness. Ferrarini and colleagues concluded that their research into the consumer emotional response could be strengthened by undertaking sensory analysis, to allow for differentiation between wine varieties. While this study was limited in that the list of terms may only be relevant to the geographic origin in which the study was conducted, i.e. it may not be applicable in a broader sense, it is interesting nonetheless.

Given the diversity of oak-derived aromas i.e. from spice, to vanilla, chocolate and smoky, and the high proportion of wine styles aged in contact with oak, the aims of this study were to investigate the

influence of specific oak aromas on consumer emotions. This work follows on from the study described in Chapter 2, in which a large proportion (83%) of consumers (n=1015) indicated their liking of oak-aged wines, and sought to determine to what extent individual oak attributes may influence consumer emotions during consumption of red wine.

4.2 Materials and Methods

This study was conducted as part of the broader research described in Chapter 3. Consumers who participated in the trials reported in Chapter 3 Section 3.6, completed an additional survey to determine their emotional response to different oak aromas.

4.2.1 Consumer Survey

The consumer survey was carried out in a sensory laboratory, with each participant assigned a random 'taster' number and allocated to an individual booth. Participants were recruited using a variety of methods, including social networking sites (i.e. Facebook, LinkedIn, Twitter), distribution of a flyer (via a mailbox drop within a 6 km radius of the University of Adelaide's Waite campus and distribution at local wine retail outlets) and email, as described earlier in Chapter 3. Participants were screened against inclusion criteria that required consumption of red wine at least once a month and being of legal drinking age (i.e. ≥18 years of age); exclusion criteria precluded participation by wine industry professionals and University of Adelaide staff and students. A sample of 116 consumers was recruited to undertake the online survey (Survey Monkey™) in October 2012.

The survey took consumers approximately 20-30 minutes to complete and comprised four sections. The first and second sections explored consumers' wine consumption behaviour and level of wine knowledge, respectively; while the third section investigated the consumers' self-reported emotional response to names of oak aromas, and the fourth section comprised socio-demographic questions.

The first section used to explore the consumers' current wine consumption included questions relating to their average spend on wine for home consumption and how likely they would be to read the wine back label. The questions in the second section were used to ascertain the consumers' level of wine knowledge, using a scale developed previously (Flynn *et al.*, 1999), along with a set of 10 multiple choice questions relating specifically to the use of oak in winemaking. Section three of the survey was designed to evaluate the consumers' emotional response to a range of oak-derived aroma names (chocolate, coconut, coffee, smoky, spice, toasty, vanilla and woody), based on the Geneva Emotion and Odor Scale (Chrea *et al.*, 2009; Porcherot *et al.*, 2010). Consumers were first asked to rate their liking of each oak aroma, using a 9-point scale with anchors from 1, "dislike extremely" to 9, "like extremely". Consumers then indicated to what extent each oak aroma name elicited various emotions, again using a 9-point scale, anchored from 1, "not relevant at all" to 9, "extremely relevant". The emotions included in this study were grouped into six categories: happiness (well-being, pleasantly surprised), romantic (desire, in love), disgusted (irritated, unpleasantly surprised), relaxed (serene, reassured), nostalgic (amusement, mouthwatering), and energetic (invigorated, clean) (Chrea *et al.*, 2009; Porcherot *et al.*, 2010).

4.2.2 Data Analysis

Data were analysed using Senpaq v5.01 (Qi Statistics, 2012) and XLSTAT 2013.1.01 (Addinsoft, 2012). A combination of descriptive and multivariate techniques were used, including Analysis of Variance (ANOVA) with post-hoc Tukey's test and principal component analysis (PCA). Hedonic clusters were identified using factor and cluster analysis.

4.3 Results and Discussion

Favourable responses were recorded for each of the oak aromas, but spice (7.0) and chocolate (6.9) were liked significantly more than other aromas, whilst smoky (5.1) and coconut (4.8) were liked least (Table 9). The ANOVA performed on data linking emotions with oak aromas indicated that *chocolate*

was most relevant to all but one emotion, and received the highest overall mean score for happiness (5.6). Interestingly, *coffee* aroma received a relatively high score for energetic (3.6), albeit *spice* was scored highest for this emotion (4.1). In addition to *chocolate*, *spice* aroma also elicited feelings of happiness (5.3), romance (4.3), relaxation (4.9) and nostalgia (4.4). *Smoky* was the aroma most associated with feelings of disgust (3.0), followed by *coconut* (2.4), while *spice* was considered of least relevance to this emotion (1.7). *Toasty* and *woody* aromas were associated more highly with feelings of happiness (4.4 and 4.8, respectively) than the other six emotions, while *vanilla* aroma was relevant to feelings of relaxation (4.4).

When ANOVA was performed on the emotional responses associated with each aroma, *chocolate, coffee and spice* were found to elicit significantly higher scores for happiness compared to the other emotions. *Coconut, toasty* and *woody* aromas were associated with happiness, relaxed and nostalgic emotions, with romantic and disgusted being of least relevance to *coconut* aroma. *Smoky* aroma was considered to elicit happiness and nostalgia, but it did not elicit disgusted or energetic emotions. *Vanilla* was most likely to elicit feelings of happiness and relaxation. Scores for disgusted were significantly lower than for other emotions, indicating it wasn't considered relevant to the oak aromas studied.

Table 9. Mean consumer liking and emotion scores for eight oak-derived aroma names.

Aroma	Chocolate	Coconut	Coffee	Smoky	Spice	Toasty	Vanilla	Woody
Mean liking	6.9 a	4.8 c	5.8 b	5.1 c	7.0 a	6.0 b	6.1 b	6.0 b
Happiness	5.6 a/a	3.7 de/a	5.0 abc/a	3.6 e/a	5.3 ab/a	4.4 cd/a	4.3 cde/a	4.8 bc/a
Relaxed	5.1 a/b	3.4 e/ab	4.1 cde/b	3.7 de/ab	4.9 ab/b	4.0 cde/ab	4.4 abc/a	4.3 bcd/ab
Nostalgic	4.7 a/b	3.1 d/abc	4.1 abc/b	3.5 cd/a	4.4 ab/b	4.0 bc/ab	3.1 d/b	4.3 ab/ab
Romantic	4.8 a/b	2.4 d/cd	3.7 bc/b	3.1 c/ab	4.3 ab/b	3.0 cd/bc	3.5 c/b	3.7 bc/bc
Energetic	3.7 a/c	3.0 bcd/bc	3.6 ab/b	2.9 cd/b	4.1 a/b	3.0 bcd/c	2.6 d/c	3.5 abc/c
Disgusted	2.0 bc/d	2.4 ab/d	2.0 bc/c	3.0 a/b	1.7 c/c	1.9 bc/d	2.3 bc/d	2.1 bc/d

Different letters within a row/column indicate statistically significant differences between oak aroma/emotion, respectively, by ANOVA where $P<0.05$.

The oak aromas were all perceived positively, but the most liked aromas generated the strongest positive emotions. The aroma which was least liked by consumers, i.e. *coconut*, is a typical descriptor of wines which have been made using oak alternatives. This may have an influence on the consumer perception of this aroma, however more work is needed to validate this suggestion. Consumers' level of wine or oak knowledge did not significantly influence their aroma likings or emotional responses (data not shown).

The emotional responses of clusters generated based on hedonic wine liking scores (refer Chapter 3) were also investigated. Consumers in C1 who preferred FOVAT and Barrel wines, indicated they most liked *spice*, followed by *chocolate* aromas (Table 10). This cluster also gave a significantly lower liking score for *smoky* aroma than the other clusters. The aroma liking of C1 was in good agreement with their wine liking, i.e. both FOVAT and Barrel wines were found to exhibit *spice* attributes by descriptive analysis (refer Chapter 3). Consumers from C2 liked all wines equally; whilst they liked *spice* and *chocolate* aromas the most, they also liked *smoky, toasty* and *woody* aromas considerably more than other consumer clusters. This may explain C2's preference for FOVAT which exhibited the most intense *smoky, charry* attributes. C3 preferred wines made using oak alternatives and gave the highest liking scores to *spice, chocolate* and *vanilla* aromas; again *coconut* and *smoky* were the least liked aromas. C3's aroma liking scores were indicative of their wine preferences, since the wines made with oak alternatives displayed intense *mocha, sweet oak* (vanilla) and *spice* characters. In regards to the emotional response generated by oak aromas, no significant differences were observed between clusters. This suggests that regardless of consumers' liking of oak specific aromas, they do not drive particular emotional responses, and factors such as price, context and occasion are more likely to impact consumers' purchase intent and/or overall wellbeing.

Table 10. Consumer hedonic cluster mean ratings for wine liking, aroma liking and emotional response.

		C1 n=35	C2 n=55	C3 n=26
Wine Liking	Barrel	5.0 b	6.2 a	4.8 b
	SS +OAK	4.7 b	6.1 a	6.1 a
	F1 -OAK	3.3 b	6.6 a	6.2 a
	F1 +OAK	3.9 b	6.3 a	6.5 a
	FOVAT	5.5 b	6.9 a	3.4 c
Aroma Liking	Vanilla	6.1	6.1	6.1
	Coconut	5.0	4.7	4.7
	Spice	7.2	7.1	6.8
	Smoky	4.4 b	5.5 a	4.9 ab
	Toasty	5.4 b	6.4 a	5.7 ab
	Woody	5.7 ab	6.4 a	5.6 b
	Coffee	5.8	5.9	5.4
	Chocolate	6.5	7.1	6.7
Emotional Response	Happiness	4.0	4.7	4.2
	Romantic	3.1	3.7	3.6
	Disgusted	2.1	2.0	2.0
	Relaxed	3.7	4.2	3.7
	Nostalgic	3.4	4.1	3.7
	Energetic	2.9	3.5	3.2

Different letters within a row indicate statistically significant differences between samples by ANOVA where $P<0.05$.

Cluster analysis based on individual oak aroma liking scores identified three distinct consumer segments that provided a different perspective from which to interpret the data. Consumers were segmented according to their oak aroma name liking scores and the emotional response of each segment obtained (^{b} differentiates these clusters from the previous wine liking clusters, Table 11). C1^b comprised 22 consumers, of which 68% were male and 50% were aged over 50 years (data not shown). This cluster also had a higher average spend per bottle of wine than the other two clusters. C2^b was the largest cluster, comprising 65 consumers, of approximately equal numbers of males and

females; a large proportion of whom were aged under 35 years (29%). C3^{b} comprised 29 consumers of which 69% were female. This group was highly educated, with 45% holding postgraduate qualifications, additionally, C3^{b} were most likely to read wine back labels. While there were no significant differences in the aroma liking scores between clusters, the predominantly male cluster, C1^{b}, liked all aromas but preferred *spice* most (7.8) and *coconut* least (5.5), while C2^{b} liked *spice* and *chocolate* most (6.8 and 6.6, respectively), and *coconut* and *coffee* least (3.9 and 4.8, respectively). C3^{b}, the predominantly female cluster, indicated their greatest liking of *chocolate, vanilla* and *spice* (7.2, 7.1 and 7.0, respectively), but liked *smoky* and *woody* least (3.2 and 4.8, respectively). The aroma liking of C3^{b} is in good agreement with a previous study which found that female consumers prefer *vanilla* over other oak-derived aromas (Bruwer *et al.*, 2011). Significant differences were found between the clusters' emotional responses. C1^{b} scores were significantly higher (than the scores of the other two clusters) for all emotions except disgusted, which was significantly higher for C2^{b}. C2^{b} and C3^{b} gave similar scores for all other emotions.

Table 11. Mean aroma liking and emotion ratings of consumer clusters segmented on aroma liking.

Cluster	C1^{b} n=22	C2^{b} n=65	C3^{b} n=29
Vanilla	6.9	5.4	7.1
Coconut	5.5	3.9	6.4
Spice	7.8	6.8	7.0
Smoky	7.3	5.3	3.2
Toasty	7.5	5.6	5.9
Woody	7.3	6.2	4.8
Coffee	7.3	4.8	6.9
Chocolate	7.3	6.6	7.2
Happiness	5.6 a	4.1 b	4.3 b
Romantic	4.5 a	3.4 b	3.3 b
Disgusted	1.6 b	2.3 a	1.9 ab
Relaxed	4.8 a	3.7 b	3.9 ab
Nostalgic	4.8 a	3.7 b	3 b
Energetic	4.1 a	3.3 ab	2.9 b

Different letters within a row indicate statistically significant differences between samples by ANOVA where $P<0.05$.

PCA was performed on the mean ratings obtained for aroma liking and emotional responses, with PC1 and PC2 accounting for 80.7% and 9.2% of the variation, respectively (Figure 8). Clear separation was observed between positive and negative emotions, with disgusted located in the upper left quadrant and positive emotions (i.e. happiness, romantic, relaxed, nostalgic and energetic) located in the right quadrants. *Smoky* and *vanilla* aromas were associated with the disgusted emotion, whereas *chocolate* was positioned near romantic and relaxed emotions, and *woody, spice* and *coffee* aromas related to happiness, nostalgia and energetic. *Coconut* and *toasty* were located in the bottom left quadrant, and were not strongly associated with any emotion.

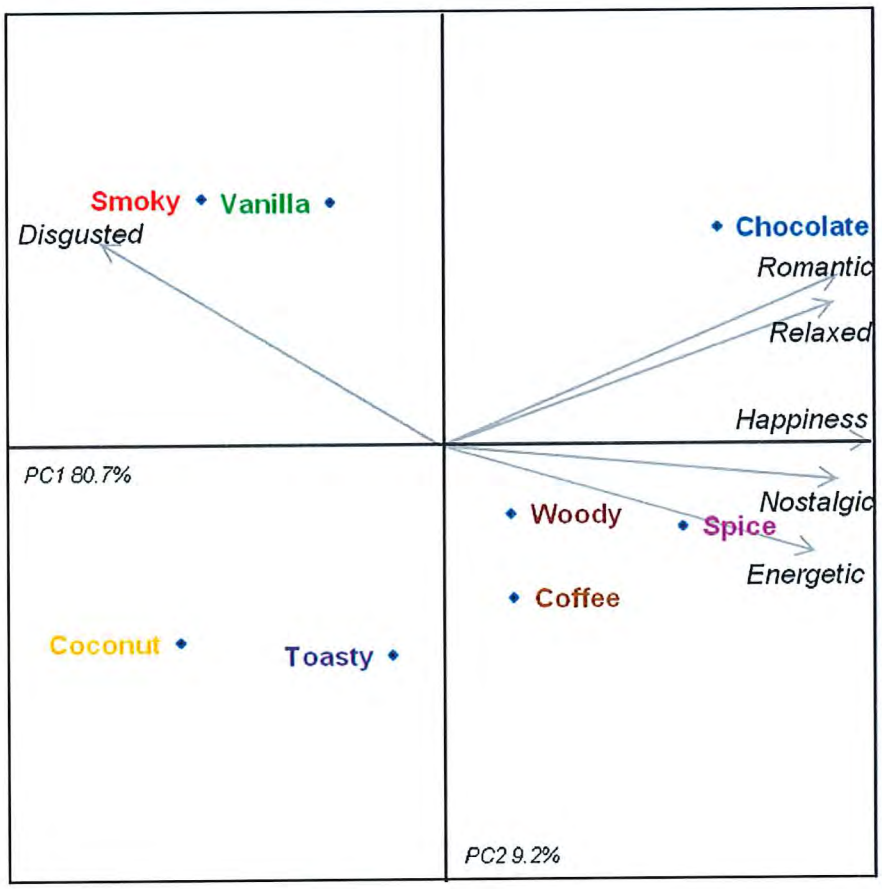

Figure 8. PCA bi-plot of mean consumer data obtained for emotional responses to oak aromas, PC1 versus PC2.

These results suggest emotional responses cannot be directly ascribed to particular aromas and may be a foreign concept to consumers, i.e. little consideration is given to specific emotions during the

purchase or consumption of wine. This study focussed on the emotions elicited by specific oak-derived aromas present in wine but may have focussed too narrowly to provide meaningful results. Future work should instead focus on the emotional responses elicited by broader aroma categories; for example, a range of oak aromas together with 'citrus', 'red berry' or 'tropical' fruit attributes. It may also be more appropriate for consumers to evaluate their emotional responses with respect to a contextual occasion i.e. a backyard barbeque versus a romantic dinner, or when presented with real stimuli.

The aromas used in this study included those commonly described on wine back labels. As such, this research provides interesting insight into consumer perceptions of the oak descriptors reported on wine labelling. Specific sensory descriptors, for example the oak aromas described in this study, can be appealing to highly involved/experienced wine consumers (Gawel, 1997; Mueller *et al.*, 2010) and give wine producers an opportunity to create interest or a point of difference on their wine labels. A recent study by Mueller and colleagues investigated the importance of wine label content to consumers (Mueller *et al.*, 2010). Their research identified a consumer segment (comprising 18% of the total population sampled), who valued the inclusion of information describing production methods (i.e. "matured in French oak barrels for 12 months prior to bottling") and elaborate taste descriptions on wine labels. The outcomes of this study may guide the descriptors chosen by wine producers' for inclusion on wine back labels; i.e. consumers seemed to like some aromas (e.g. *chocolate* and *spice*) more than others (e.g. *coconut* and *smoky*).

Chapter 5: Paper 3 - Preparation of isotopically labelled volatile wine phenols using microwave assisted deuterium exchange

The use of isotopically labelled compounds as internal standards for GC-MS analysis allows accurate quantification of analytes of interest in a given sample (Hislop *et al.*, 2004). Deuterium labelled standards mimic the physical properties of target compounds, but differences in molecular weight allow the internal standard and analytes to be easily distinguished by mass spectrometry. However, labelled compounds are not always commercially available. Synthesis of labelled analogues is often time consuming and laborious, and it can be an expensive exercise (Vining *et al.*, 1981). In 2000, Pollnitz and colleagues reported the synthesis of deuterated 4-ethylphenol using a traditional synthesis method, which involved several production steps and heating of the product for 5 days at 100°C. Previous authors have commented on the importance of incorporating the deuterium label on the benzene ring (Hislop *et al.*, 2004), but few have been able to introduce multiple deuteriums to the ring via traditional synthesis.

Microwave-assisted chemical synthesis has gained popularity, primarily due to the significantly reduced reaction times (Chappelle *et al.*, 2002; Kalpala *et al.*, 2003). The following paper describes the preparation of several deuterium labelled volatile wine phenols using microwave-assisted deuterium exchange, in a simple, one-step reaction. These compounds were initially synthesised to allow the quantitative analysis described in Chapter 3, however, due to time constraints a commercial analytical laboratory was used for volatile analyses. Future studies which seek to measure oak volatile concentrations will be able to employ the methodology reported herein.

5.1 Statement of Authorship

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Anna Crump

Designed and conducted experiments, prepared samples for characterisation by GC-MS and NMR, analysed and interpreted data, drafted and revised the manuscript.

I hereby certify that the statement of contribution is accurate and I give permission for inclusion of the paper in this thesis.

Signed Date 18/2/15

Mark Sefton

Assisted with experimental design, analysed and interpreted data, assisted with revising the manuscript.

I hereby certify that the statement of contribution is accurate and I give permission for inclusion of the paper in this thesis.

Signed Date 18/2/15

Kerry Wilkinson

Assisted with experimental design and preparation of samples for characterisation by NMR, analysed and interpreted data, assisted with revising the manuscript.

I hereby certify that the statement of contribution is accurate and I give permission for inclusion of the paper in this thesis.

Signed Date 18/2/15

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Short communication

Microwave-assisted deuterium exchange: The convenient preparation of isotopically labelled analogues for stable isotope dilution analysis of volatile wine phenols

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ABSTRACT

This study reports the convenient, low cost, one-step synthesis of labelled analogues of six volatile phenols, guaiacol, 4-methylguaiacol, 4-ethylguaiacol, 4-ethylphenol, eugenol and vanillin, using microwave-assisted deuterium exchange, for use as internal standards for stable isotope dilution analysis. The current method improves on previous strategies in that it enables incorporation of deuterium atoms on the aromatic ring, thereby ensuring retention of the isotope label during mass spectrometry fragmentation. When used as standards for SIDA, these labelled volatile phenols will improve the accuracy and reproducibility of quantitative food and beverage analysis.

1. Introduction

Gas chromatography–mass spectrometry (GC–MS) is a powerful technique for the compositional characterisation of complex samples, for example the qualitative and quantitative analysis of volatile compounds in food and beverages. Quantitative analysis typically involves the use of internal standards to compensate for analyte losses during sample preparation, due to evaporation or degradation. Ideally, the internal standard is a compound as similar as possible to the analyte of interest, but not already present in the sample matrix. Stable isotope labelled compounds make excellent internal standards (Vining, Smythe, & Long, 1981), since they possess physical and chemical properties that are virtually identical to those of the analyte. Analytical methods using stable isotope dilution analysis (SIDA) therefore enable accurate and reproducible quantification. However, labelled standards are not always commercially available. While traditional organic synthesis can be used to prepare labelled standards via the incorporation of one or more deuterium atoms, these methods can be complex, technically challenging, time consuming, laborious and expensive (Vining et al., 1981); particularly for the preparation of highly deuterated standards.

Volatile phenols have been reported as constituents of various foods and beverages. For example, eugenol is responsible for the distinctive aroma of cloves and also contributes to the aroma of cinnamon (Tikunov, de Vos, González Paramás, Hall, & Bovy, 2010); several volatile phenols, including guaiacol and eugenol, have been identified as glycosidically bound constituents of tomato and strawberry (Tikunov et al., 2010; Ubeda et al., 2012); while vanillin, guaiacol, 4-methylguaiacol and eugenol are oak-derived volatiles present in wine and spirits matured in oak barrels (Pino, Tolle, Gök, & Winterhalter, 2012; Spillman, Sefton, & Gawel, 2004). Volatile phenols have also been implicated in a range of off-odours and taints. Guaiacol was identified as the microbial metabolite responsible for a smoky/phenolic taint in chocolate milk (Jensen, Varelis, & Whifield, 2001), while 4-ethylphenol and 4-ethylguaiacol were found to cause off-odours in strawberries infected with leather rot (Jeleń, Krawczyk, Larsen, Jarosz, & Golebniak, 2005). In wine, these last two compounds are associated with the 'horsy', 'medicinal', 'smoky', 'barnyard' attributes indicative of *Brettanomyces/Dekkera* spoilage (Boidron et al., 1988; Chatonnet et al., 1992). A range of volatile phenols, including guaiacol, 4-methylguaiacol, 4-ethylguaiacol, 4-ethylphenol and *o*-, *m*- and *p*-cresols, have recently been identified in grapes and wine tainted by bushfire smoke (Kennison, Gibberd, Pollnitz, & Wilkinson, 2008; Parker et al., 2012).

The preparation of d_3-labelled analogues of guaiacol, 4-methylguaiacol and vanillin have been previously reported, via synthetic pathways involving methylation with deuterated methyl iodide (Pollnitz, Pardon, Sykes, & Sefton, 2004; Spillman, Pollnitz, Liacopoulos, Skouroumounis, & Sefton, 1997). However, the isotope label is readily lost from these analogues upon cleavage of the deuterated methoxy functional group, during mass spectrometry fragmentation, limiting the selection of suitable fragments for selective ion monitoring of the standard. Incorporation of

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deuterium on the aromatic ring ensures retention of the isotope label (Hislop, Hunt, Fielder, & Rowan, 2004), but can be difficult to achieve via traditional synthesis. Pollnitz and colleagues synthesised d_4-4-ethylphenol from the acetate of polydeuterated phenol (Pollnitz, Pardon, & Sefton, 2000); but via a protracted synthetic pathway.

Microwave-assisted chemical reactions have gained popularity in the field of organic synthesis since they were first reported in 1986 (Gedye et al., 1986), primarily because they significantly reduce reaction times and offer convenient, one-step procedures (Chappelle, Kent, Jones, Lu, & Morgan, 2002). Microwave-assisted synthesis can also achieve deuterium exchange on the aromatic ring (Dungey, Hayasaka, & Wilkinson, 2011). This study reports the use of microwave-assisted deuterium exchange for the preparation of isotopically labelled volatile phenols.

2. Materials and methods

2.1. General

Reagents were purchased from Sigma–Aldrich (St. Louis, USA), Honeywell Riedel-de Haën (Germany), Fluka (Japan), Chem Supply (Australia) and Scharlau (Spain). ^1H and ^{13}C NMR spectra were recorded with a Bruker spectrometer (Alexandria, Australia) at an operating frequency of 300 MHz. Mass spectra were recorded with an Agilent 6890N gas chromatograph coupled to a 5973 mass selective detector (Palo Alto, CA, USA).

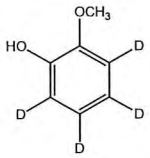
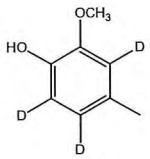
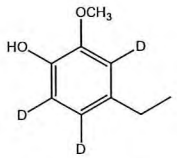
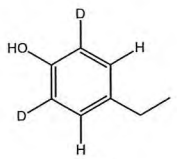
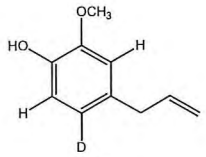
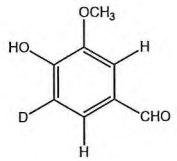
2.2. General procedure for deuterium incorporation into volatile phenols

Approximately 1 g of substrate (Table 1) was suspended in thionyl chloride ($SOCl_2$, 2 mL) and deuterium oxide (D_2O, 20 mL) in the reactor tube of a Discover SP-D microwave apparatus (CEM, Matthews NC, USA). The tube was capped and irradiated at the temperatures and for the durations outlined in Table 1. The reaction mixture was then neutralised with careful dropwise addition of 10% sodium hydroxide solution and extracted with dichloromethane (3 × 50 mL). The combined organic extracts were dried and concentrated, and the crude product purified by Kugelrohr micro-distillation; except for deuterated vanillin which was purified by recrystallisation from D_2O.

2.2.1. [$^2H_4$]-Guaiacol

δ_H ($CDCl_3$): 5.62 (1H, s, OH), 3.88 (3H, s, OCH_3); δ_C ($CDCl_3$): 146.5, 145.5, 55.8; *m/z*: 128 (M^+, 87%), 113 (100%), 85 (54%), 57 (12%), 32 (17%), 28 (71%).

Table 1
Experimental conditions and compositional outcomes for microwave-assisted deuterium exchange of volatile wine phenols.

Substrate	Temperature (°C)	Duration (hours)	Isotopic Composition (%)					Structure
			d_0	d_1	d_2	d_3	d_4	
Guaiacol	100	36					**100**	
4-Methylguaiacol	100	30				**100**		
4-Ethylguaiacol	100	36				**100**		
4-Ethylphenol	100	144	4	11	**85**			
Eugenol[a]	80	8	3	22	**42**	33		
Vanillin[a]	100	45	3	40	**57**			

[a] One aromatic proton was fully exchanged, but incorporation of the second deuterium occurred incompletely at the remaining positions.

2.2.2. [$^2H_3$]-4-Methylguaiacol

δ_H ($CDCl_3$): 5.46 (1H, s, OH), 3.87 (3H, s, OCH_3), 2.27 (3H, s, CH_3); δ_C ($CDCl_3$): 146.1, 143.2, 129.4, 55.8, 20.8; *m/z*: 141 (M^+, 21%), 126 (18%), 98 (6%), 32 (24%), 28 (100%).

2.2.3. [$^2H_3$]-4-Ethylguaiacol

δ_H ($CDCl_3$): 5.46 (1H, s, OH), 3.88 (3H, s, OCH_3), 2.58 (2H, q J = 7.8, CH_2), 1.22 (3H, t, J = 7.8, CH_3); δ_C ($CDCl_3$): 146.2, 143.4, 136.1, 55.8, 28.4, 15.9; *m/z*: 156 (4%), 155 (M^+, 42%), 154 (3%), 141 (9%), 140 (100%), 139 (6%), 125 (9%), 97 (5%), 94 (6%).

2.2.4. [$^2H_2$]-4-Ethylphenol

δ_H ($CDCl_3$): 7.06 (2H, s, ArH), 4.68 (1H, s, OH), 2.59 (2H, q J 7.6, CH_2), 1.204 (3H, t J 7.5, CH_3); δ_C ($CDCl_3$): 153.3, 136.5, 128.8, 28.0, 15.9; *m/z*: 124 (M^+, 37%), 110 (20%), 109 (100%), 93 (4%), 79 (9%), 78 (7%).

2.2.5. [$^2H_2$]-Eugenol

δ_H ($CDCl_3$): 6.85 (1H, s, ArH), 6.69 (1H, s, ArH), 5.94 (1H, m, CH), 5.49 (1H, s, OH), 5.08 (2H, m, CH_2), 3.87 (3H, s, OCH_3), 3.31 (2H, d J 6.0, CH_2); δ_C ($CDCl_3$): 146.4, 143.8, 137.7, 131.8, 115.5, 114.2, 111.1, 55.9, 39.8; *m/z*: 166 (M^+, 100%), 165 (47%), 151 (31%), 134 (29%), 105 (31%), 93 (20%), 78 (20%).

2.2.6. [$^2H_2$]-Vanillin

δ_H ($CDCl_3$): 9.83 (1H, s, CHO), 7.42 (1H, s, ArH), 6.21 (1H, s, OH), 3.96 (3H, s, OCH_3); δ_C ($CDCl_3$): 191.0, 151.6, 147.1, 127.5, 108.7, 56.1; *m/z*: 155 (8%), 154 (M^+, 58%), 153 (100%), 152 (54%), 125 (7%), 110 (10%), 82 (11%), 28 (13%).

3. Results and discussion

Application to 4-methylguaiacol of the conditions employed by Dungey and co-workers to prepare d_4-guaiacol (Dungey et al., 2011) gave 100% incorporation of deuterium into the aromatic ring (Table 1). However, deuterium incorporation was not achieved to the same extent for 4-ethylguaiacol, or even guaiacol, under the same conditions. Following optimisation of the method, deuterium atoms were fully incorporated into the aromatic ring, to give [$^2H_4$]-guaiacol and [$^2H_3$]-4-ethylguaiacol. The NMR spectra of labelled guaiacol, 4-methylguaiacol and 4-ethylguaiacol closely matched their unlabelled analogues, but with the absence of signals for aromatic protons. The degree of deuterium incorporation was confirmed by GC–MS (Table 1).

Only partial deuterium exchange was achieved for 4-ethylphenol, eugenol and vanillin, to give [$^2H_2$]-4-ethylphenol, [$^2H_2$]-eugenol and [$^2H_2$]-vanillin; with the extent of deuterium incorporation again confirmed by NMR and GC–MS. In the case of [$^2H_2$]-eugenol and [$^2H_2$]-vanillin, one aromatic proton was fully deuterium exchanged, but incorporation of the second deuterium occurred incompletely at the remaining positions. Although these compounds did not achieve complete deuteration they were still suitable for use as internal standards for analysis by GC–MS. Eugenol decomposition was observed at reaction temperatures above 80 °C or reaction durations above 8 h, in agreement with a previous study reporting decomposition of eugenol at high temperatures following the addition of hydrochloric acid (Kalpala, Hartonen, Huhdanpaa, & Riekkola, 2003). The same study reported the deuteration of eugenol using only D_2O, but we were unable to reproduce these results. Vanillin polymerised under more forcing reaction conditions.

Microwave-assisted deuterium exchange of several other important oak volatiles, i.e. isoeugenol, furfural and oak lactone, was also attempted using various reaction conditions. However, the neat compounds either polymerised upon addition of thionyl chloride (furfural and isoeugenol) or, in the case of oak lactone, underwent only limited deuterium exchange, with a substantial proportion of the product remaining undeuterated.

4. Conclusion

In the current study, microwave technology has been used to produce deuterium labelled volatile phenols in a convenient, one-step synthesis, with the additional benefit of incorporating the deuterium atoms on the aromatic ring. This method is suitable for making labelled analogues for the identification and quantification of these compounds, which are often found in foods and beverages.

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Chapter 6: Conclusions and future work

Oak barrels have long been the preferred vessel for the maturation and storage of wine, largely due to the desirable mechanical properties of oak wood, together with its positive impact on wine aroma and flavour. Factors such as the species and geographical origin of oak (i.e. France vs. America), barrel volume, duration of maturation and barrel usage influence not only the composition and sensory properties of oak-aged wine, but also winery expenditure on oak. Indeed, the use of oak barrels contributes significantly to overall production costs. As such, the wine industry has increased its use of oak alternatives, i.e. oak chips, shavings and powders, as rapid methods of oak maturation, particularly for less expensive wines (Perez-Coello *et al.*, 1999; Heras *et al.*, 2008; Hernandez-Orte *et al.*, 2009). However, little is known regarding the consumer acceptability of wines made with oak alternatives and the impact of oak maturation regimes on consumer purchase decisions.

This research aimed to investigate the influence of alternative oak maturation regimes on the composition, sensory properties, quality and consumer acceptance of wine. The project's key aims were: (1) to understand consumers' knowledge of the role of oak maturation in wine production; (2) to investigate the influence of innovative oak maturation regimes on the composition, sensory profile and consumer acceptance of wine; and (3) to understand the extent to which oak-derived aromas and flavours elicit emotional responses from consumers.

The main objective of the study described in Chapter 2 was to investigate Australian wine consumers' knowledge of the role of oak in winemaking and their preference for oak-derived sensory attributes. While previous studies have reported the use of oak alternatives by the industry and their contribution to the sensory properties of wine, only one had explored consumers' preferences for wines aged with oak alternatives versus traditional barrels (Perez-Magarino *et al.*, 2011). In the current study, focus groups were initially conducted to explore consumers' attitudes towards the use of oak alternatives. The

data collected was used to design an online survey which then examined consumer knowledge of oak use by the wine industry, their attitudes towards the use of oak alternatives and preferences for specific wine aroma and flavours.

1015 consumers completed the survey. Segmentation identified 4 consumer clusters, each with differing opinions towards the use of oak alternatives and distinct differences in their preferences for wine sensory attributes. As a whole, the consumers scored oak sensory characters favourably. Concerning the use of oak alternatives in wine production, the study identified a segment comprising more knowledgeable consumers who valued traditional oak maturation regimes and who were willing to pay a premium for these wines. A second group of less knowledgeable consumers were found to be more accepting of innovative oak technologies, providing wine quality was not compromised. By profiling the attitudes, sensory preferences and demographics of consumers in each cluster, the outcome of this study justifies the use of oak alternatives for the production of wines at lower price-points, that appeal to specific segments of the market.

The trial described in Chapter 3 evaluated the influence of different oak maturation regimes on the composition, sensory profile and consumer acceptance of wine. This study demonstrated that different maturation regimes can be used to impart oak characters to wine. The study found that the oak type and surface area had a greater influence on the concentration of oak volatiles observed in wines, than the vessel used. Based on descriptive analysis results, a subset of wines with distinct flavour profiles were selected for consumer and expert panel evaluation. The expert panel used the 20-point Australian wine judging system to assess the quality of each wine, with all wines found to be technically sound. The consumer acceptability of each wine was then determined using 9-point Likert scales. Scores ranged from 5.7 to 5.9 indicating there was no significant difference in consumers' overall liking of each wine, but segmentation of consumers based on their wine preferences yielded three distinct clusters.

One segment were driven by leather attributes and weren't overly accepting of any of the wines presented, another liked all wines equally, while the third segment liked wines made using oak alternatives. These results further justify wine producers' use of alternative oak maturation regimes to achieve wine styles that appeal to different segments of their target market.

Future work arising from the outcomes of this study:

- It would be interesting to repeat this study with commercial wines made using alternative oak maturation regimes, to explore consumer preferences for wines both blind and when the maturation regime is explained. During the focus groups described in chapter 2, a number of consumers indicated they had thought wines were always aged in barrels. It would therefore be interesting to evaluate whether or not consumer perceptions change when production information is revealed. Certainly Marin and colleagues found consumers' wine preferences and perceptions of quality decreased significantly when they knew wines were sealed under screw cap (Marin *et al.*, 2007). That said, it's unlikely that wine producers would highlight the use of oak alternatives on the back label.

Chapter 4 describes a study which was undertaken to explore the influence of oak-derived aromas on consumer emotions. The field of emotions is an emerging area of interest within sensory science, but to date, no studies have investigated whether or not wine attributes elicit specific emotional responses. In this study, consumers rated their liking of specific oak aromas, e.g. chocolate, and then, using the Geneva Emotion and Odor Scale (Chrea *et al.*, 2009), they indicated how each aroma made them feel. Overall, consumers scored each of the oak aromas favourably; spice and chocolate were liked most, and smoky and coconut were liked least. Analysis of results according to the consumer hedonic clusters identified in Chapter 3, found that in most cases, consumer liking of specific oak aromas correlated with their wine preference scores. Cluster analysis based on aroma liking responses,

subsequently identified three clusters that differed in their liking of each aroma and their emotional responses to each aroma.

The oak attributes included in this study are commonly listed as descriptors on wine back labels. These results might therefore provide insight into consumer perceptions of wine label content. The study provided further evidence that the majority of wine consumers perceive oak characters favourably, which justifies its continued use in winemaking. These results will enable winemakers to tailor their oak maturation regimes to enhance sensory attributes that appeal to consumers (e.g. spice and chocolate) and wine marketers to take advantage of points of difference that appeal to specific segments within a target market.

As an emerging area of research, there are many opportunities for further research concerning the impact of wine aromas on consumer well-being. Some specific research questions that arose following this study include:

- To what extent do broader sets of wine aromas influence consumers' emotional responses? The aromas used in the current study were quite specific, which may have posed a challenge to wine consumers, particularly those with less wine knowledge and experience. Future research could compare consumers' emotional responses to oak aromas as a single category alongside more familiar attributes such as 'red fruit' or 'tropical fruit'.

- To what extent does occasion influence consumers' emotional responses? For example, consuming a white wine at a backyard barbeque in summer may drive different emotions than when the same wine is consumed while sitting in front of a fire in winter.

The final study involved the preparation of deuterium labelled volatile wine phenols using microwave-assisted deuterium exchange. Microwave–assisted reactions have been growing in

popularity due to their capacity to produce stable compounds in a convenient period of time without resorting to complex synthetic strategies. Deuterated compounds are commonly used for compositional characterisation of substances by GC-MS, and are usually made using fairly laborious synthetic chemistry strategies. Dungey and colleagues first reported the microwave-assisted synthesis of d_4-guaiacol (Dungey *et al.*, 2011), which represented an opportunity to produce similar deuterium labelled compounds commonly used for oak volatile analysis. Deuterated standards were produced for guaiacol, 4-methylguaiacol, 4-ethylguaiacol, eugenol and vanillin, with NMR confirming deuterium placement on the aromatic ring. This afforded the additional advantage that deuterium atoms are retained during mass spectrometry fragmentation. Isotopically labelled analogues of oak lactone, isoeugenol and furfural could not be prepared due to low reactivity or decomposition of starting material.

The research outcomes presented in this thesis have identified that consumers may possess only a limited appreciation for the use of oak in winemaking, but they like the sensory properties imparted by oak maturation. The results presented from the consumer survey and preference test identified market segments that are quite accepting of the use of oak alternatives, and provided that wine quality is not compromised, would happily purchase and/or consume this product. In contrast, other markets exist for wines which have been made using traditional barrel maturation methods, and which generally command higher prices. While the use of oak alternatives is not suitable for every consumer, every wine or every producer, the outcomes of this research justify their use by the wine industry so as to reduce overall production costs, particularly for wines sold within lower price points. Furthermore, this study highlights the importance of combining compositional and descriptive sensory analyses with consumer research, to achieve a broader understanding of wine quality and acceptability drivers.

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