# GRAPE RIPENING AND WINE STYLE: SYNCHRONIZED EVOLUTION OF AROMATIC COMPOSITION OF SHIRAZ WINES FROM HOT AND TEMPERATE CLIMATES OF AUSTRALIA

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#### Abstract:

**Context and purpose of the study** - Grape ripening is a process driven by the interactions between grapevine genotypes and environmental factors. Grape composition is largely responsible for the production and final concentrations of most wine aroma compounds even though many compounds in wines (aromatic and non-aromatic) are substantially transformed during fermentation and wine ageing. The aim of this study was to investigate if a common pattern in grape/wine flavour plasticity related to ripening exists irrespective of a grape growing region. A further aim was to identify and highlight compounds present in Shiraz grapes and wines in which plasticity is directly related to grape ripening and is consistent over several vintages.

**Material and methods** - Commercial vineyards of Shiraz were chosen in two Australian wine geographical indication (GI) regions: Griffith (warm to hot climate) and Orange (temperate to temperate-warm climate). In these vineyards, own rooted vines were grown under drip irrigation, and trellised to a sprawling training system and in vertical shoot positioning for Orange. Sequential harvests were performed using berry sugar accumulation as a physiological indicator of grape maturity. At each harvest date, triplicates of 100 berries were collected and frozen in liquid nitrogen in the field for later chemical analyses. Approximately 60 kg of grape per replicate were randomly harvested at each harvest date and small scale vinifications carried out.

Amino acids in grapes were analysed by high performance liquid chromatography (HPLC) coupled to fluorescence detector. Grape volatiles analyses were performed with gas chromatography coupled to mass detection (GC-MS). Juice was analysed for set of parameters relating to the technical maturity of grapes (total soluble solids, titratable acidity and pH) and yeast assimilable nitrogen was measured. Wine aromatic compounds were quantitated by HS-SPME-GC-MS. Descriptive sensory evaluation with predefined descriptors was conducted approximately six months after bottling.

**Results** - Irrespective of the macro and meso climates, differences in both grape and wine chemical analyses and wine sensory description produced a clear separation of samples according to the harvest stage. Shiraz wines from the first harvest (H1) were associated with red fruit descriptors and higher perception of acidity. Wines from the third harvest (H3) were correlated with dark fruit characters and a higher alcohol. Later harvest dates resulted in higher concentrations of some amino acids in the Shiraz grapes, with higher alcohol acetates, ethyl esters (ethyl propanoate and ethyl butyrate) of short chain fatty acids and dimethyl sulphide in the wines. Conversely, concentrations of <u>(Z)</u>-3-hexenol, ethyl isobutyrate, ethyl leucate and ethyl dihydrocinammate were lower in these wines compared to earlier harvest dates. Observed trends were significant and consistent across two vintages and two different GIs. From the plateau of berry sugar accumulation, no direct nexus was observed between berry sugar concentration and grape and wine flavour evolution. This study also demonstrated a common evolution of Shiraz grapes, influencing the chemical and sensory properties of the subsequent wine.

**Keywords**: Grapevine, Australia, Shiraz, warm and temperate climates, sequential harvests, fruit and wine composition, sensory analyses.

# 1. Introduction

The association of agricultural products to a provenance or specific region (terroir) that imparts a typical and unique sensorial profile is an important concept for high value products. Increasing consumer demand and interest for foods and wines associated with, and which identify with specific regions and places of production, are evident, and are important economic and marketing factors for product differentiation and uniqueness (Charters et al., 2017).

Wine style depends principally on grape composition which is largely determined by cultivars, abiotic factors, grapevine physiology and cultural practices. Differences in grape composition pertaining to site arise from vines adapting to different biotic and abiotic environments (Tonietto and Carbonneau 2004), which in turn influence vine physiology and ultimately berry composition (Deloire et al., 2008).

Harvest decision is largely determined by a range of objective measures of grape maturity (e.g. °Brix, titratable acidity and colour), however these indices do not provide information about the aromatic potential of the grapes or the resulting wine flavour profiles (Calderon-Orellana et al. 2014; Deloire, 2014; Bindon et al., 2013). The aims of this study, across two Australian GIs (Riverina and Orange), were to determine: i) if a common pattern in grape/wine flavour plasticity related to grape ripening exists irrespective of a grape growing region, ii) if commonalities in flavour evolution exists across vintages and iii) to identify and highlight compounds present in grapes and wines which are directly related to grape ripening while practising sequential harvest.

# 2. Material and methods

## Plant material and growing conditions

The experiment was conducted in 2014 and 2015 in NSW, Australia, in two distinctively different grape growing regions. Griffith (G) is classified as warm to hot grape growing region, according to Huglin index, whereas Orange (O) is a temperate (950m above sea level) to temperate-warm (650m above sea level). Griffith is characterized by a flat terrain and secure water supply, enabling it to maintain a 15% share of the total Australian grape production. Two commercial Shiraz vineyards were selected in Griffith in season 2014, and in 2015 two additional Shiraz vineyards were also utilised; G1, G2, G3 and G4, respectively. Two Shiraz vineyards were also selected in O for experimentations in the 2015 season; O1 and O2, respectively. Shiraz vines were own rooted, grown under drip irrigation, and trellised to a sprawling system in Griffith. In Orange vines were trellised to vertical shoot positioning. During the season mesoclimatic temperatures, stem water potential and soil moisture were monitored to help characterise vineyard conditions.

Harvest dates for vineyards were determined from the point where sugar accumulation per berry reached a plateau or slowed down, with the first harvest (H1), second harvest (H2) and third harvest (H3) picked at 12, 18 and 24 days respectively from this juncture, as summarised by Figure 1. At each harvest date, 100 berry grape samples were collected and immediately frozen in liquid nitrogen. Approximately 60 kg of grapes per replicate were randomly harvested at each harvest date and small scale vinifications performed.

Both grapes and wines were analysed using a range of analytical measurement techniques and sensory evaluation was performed on the finished wines. The analytical and sensory methods are described in Suklje et al., 2014; 2017.

Data sets for multiblock analysis were constructed for specific explanatory factors (EF) by partitioning selected samples from the entire sample set to create balanced ANOVA designs. Data analysis was conducted using the AComDim method (Bouveresse et al. 2011) for balanced ANOVA designs for EF: harvest date (levels H1, H2 & H3); vineyard (levels G1 and G2) and vintage (levels V2014 & 2015). All multivariate analysis was conducted in Matlab version 7.14.0.739 (The Mathworks, Natick, MA, U.S.A.).

#### 3. Results and discussion

Wine sensory evaluation revealed clear differences in wine style according to harvest (Figure 2) with sensory attribute ratings consistently rated in terms of differences for vineyard site and harvest time. Figure 2 illustrates sensory ratings for the Griffith and Orange vineyard (vineyard 2). Other vineyards for each location were rated similarly (data not shown). A clear sensory profile of higher acidity, red fruit and lower perception of alcohol is evident for early harvested fruit. Attributes of dark fruit, astringency and higher perceived alcohol are features of the later harvested wines.

AComDim models show a common clustering of samples according to the explanatory factor *Harvest* with clearly separate confidence intervals (circles) regardless of vintage (Figure 3, H1 and H2), region or site (Figure 4, H1, H2 and H3). A common pattern in grape/wine flavour plasticity related to grape ripening exists for Shiraz irrespective of vintage, grape growing region and vineyard management.

Regional and vineyard influences on wine style are important factors for winegrowers seeking to differentiate products based upon unique terroirs. Identification of specific grape berry composition due to site is challenging and requires a longitudinal study over several growing seasons. In this preliminary investigation a clear separation of samples according to both region and vineyard is evident (Figure 3). Grape components that influenced sample separation were berry amino acids; generally higher in Griffith samples; berry monomeric anthocyanins (higher in Orange samples); wine acetate esters higher and terpenes lower in Griffith wines.

Irrespective of the grape growing region, samples could be clearly grouped according to the harvest date, Figure 4. Unsurprisingly, concentrations of glucose and fructose increased with delayed harvest dates, which resulted in wines with higher alcohol content. Other compounds contributing to the separation of samples were some amino acids (proline,  $\gamma$ -aminobutyric acid, isoleucine) which were at higher concentrations in the H3 grapes compared to grapes from H1. Some higher alcohol acetates as well as dimethyl sulfide were higher in wines from H3 while (*Z*)-3-hexenol, ethyl isobutyrate and ethyl leucate were lower in these wines. (*Z*)-3-hexenolwas the only C6-alcohol, a group of compounds known to contribute to grape maturity in this study. Dimethyl sulfide is known to contribute to dark fruit character in red wine (Lytra et al. 2014) and has been previously reported as marker of wines made with late maturity grapes (Dagan 2006). Interestingly, even though esters are principally produced by yeast during fermentation, final wine concentrations were influenced by grape maturity as recently reported (Antalick et al. 2015).

## 4. Conclusions

This study clearly demonstrated that irrespective of the macro and meso climates, differences in both Shiraz grape and wine chemical analyses and wine sensory description produced a clear separation of samples according to the harvest stage. Observed trends were significant and consistent across vintages and different GIs from Australia (Griffith and Orange wine regions). From the plateau of berry sugar accumulation, no direct nexus was observed between berry sugar concentration and grape and wine flavour evolution. This study also demonstrated a common evolution of Shiraz grapes, influencing the chemical and sensory properties of the subsequent wine.

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# 6. Litterature cited

Antalick, G.; Šuklje, K.; Blackman, J.W.; Meeks, C.; Deloire, A. and Schmidtke, L.M., 2015. Influence of grape composition on red wine ester profile: Comparison between Cabernet Sauvignon and Shiraz cultivars from Australian warm climate. Journal of Agricultural and Food Chemistry 63 (18):4664-4672.

**Bindon, K.; Varela, C.; Kennedy, J.; Holt, H. and Herderich, M**., 2013. Relationships between harvest time and wine composition in Vitis vinifera L. cv. Cabernet Sauvignon 1. Grape and wine chemistry. Food Chemistry 138 (2–3):1696-1705.

**Bouveresse, J.-R.D.; Pinto, R.C.; Schmidtke, L.M.; Locquet, N. and Rutledge, D.N.,** 2011. Identification of significant factors by an extension of ANOVA-PCA based on multi-block analysis. Chemometrics and Intelligent Laboratory Systems 106 (2):173-182.

**Calderon-Orellana, A.; Matthews, M.A.; Drayton, W.M. and Shackel, K.A**., 2014. Uniformity of ripeness and size in cabernet sauvignon berries from vineyards with contrasting crop price. American Journal of Enology and Viticulture 65 (1):81-88.

**Charters, S., N. Spielmann, B. J. Babin**, 2017. The nature and value of terroir products. European Journal of Marketing 51, 748-771 doi:doi:10.1108/EJM-06-2015-0330

**Dagan, L.,** 2006. Potentiel aromatique des raisins de Vitis vinifera L. cv. Petit Manseng et Gros Manseng. Contribution à l'arôme des vins de pays Côtes de Gascogne. Thesis, Ecole Nationale Superieure Agronomique de Montpellier.

**Deloire, A.,** 2014. Physiologocal Indicators to Predict Harvest Date and Wine Style. In: Beames, K. S., E. M. C. Robinson, P. W. Godden, D. L. Johnson (eds) 15th Australian Wine Industry Technical Conference: Sydney, New South Wales 13-18 July 2013, Sydney, 2013. The Australian Wine Industry Technical Conference Inc., Urrbrae, South Australia, p 47-50.

**Deloire, A., P. Prévost, Kelly M.,** 2008. Unravelling the Terroir Mystique–an agro-socioeconomic perspective. Perspectives in Agriculture, Veterinary Science, Nutrition and Natural Resources 3, 1-9.

**Lytra, G., Tempere, S., Zhang, S., Marchand, S., de Revel, G., Barbe, J. C.,** 2014. Olfactory impact of dimethyl sulphide on red wine fruity esters aroma expression in model solution. J. Int. Sci. Vigne Vin 48, 75-85.

Šuklje K., Antalick G., Meeks C., Blackman J., Deloire A., Schmidtke L.M., 2017. Grapes to wine: the nexus between berry ripening, composition and wine style.

https://doi.org/10.17660/ActaHortic.2017.1188.6.

**Šuklje K., Antalick, G., Meeks, C.B., Blackman, J.W., Deloire, A. and Schmidtke, L.M**., 2014. Optimising harvest date through use of an integrated grape compositional and sensory model: a proposed approach for a better understanding of ripening of autochthonous varieties? 2nd International Symposium Oenoviti International network 3-5 November 2014 Gesenheim, Germany 44-49.

**Tonietto, J. and Carbonneau, A.,**2004. A multicriteria climatic classification system for grape-growing regions worldwide. Agricultural and Forest Meteorology 124 (1–2):81-97.



**Figure 1:** Sequential harvest of Shiraz is done according to the berry sugar loading method (Deloire, 2014). The stages called "fresh fruit" and "mature fruit" are generally reached respectively 12 and 24 days after the plateau of berry sugar accumulation.



**Figure 2:** Spider graphs representing mean ratings for sensory attributes in wines made from grapes harvested at different predicted harvest maturity. H1, H2 and H3 refer to harvest date 1, 2 and 3, respectively. G1 and G2 refer to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in Griffith and O1 and O2 refers to the experimental vineyards located in



**Figure 3**: ACOMDIM model of grape berry composition, wine sensory and wine composition with samples coded for G1 and G2 vineyards for the 2014 and 2015 growing seasons. Ellipses represent the 95% confidence interval for each harvest (Harvest 1 & Harvest 3).



**Figure 4:** ACOMDIM analyses conducted on grape berry, juice, wine and wine sensory results for the 2015 growing season. A: Separation of samples according to the harvest date (Harvest 1, Harvest 2 & Harvest 3) for Shiraz vineyards in Orange and Griffith. Ellipses represent 95% confidence interval for each ANOVA explanatory factor.