In Vino Analytica Scientia Symposium 2015

Analytical Chemistry for Wine, Brandy and Spirits

14-17 July 2015, Trento, Italy



FINAL REPORT to

AUSTRALIAN GRAPE AND WINE AUTHORITY

Project Number: AGT 1509

Principal Investigator: Jiaming Wang

Research Organisation: The University of Adelaide

Date: August 2015

Project title: Determination of Key Aroma Compounds in Australian Rosé Wine

Project No: AGT 1509

Author: Mr Jiaming Wang

Date: August 2015

Copyright Statement: This work is copyright. Apart from any use permitted under the Copyright Act 1968, no part may be reproduced by any process without written permission from the University of Adelaide.

The University of Adelaide
School of Agriculture, Food and Wine
Private Mailbox 1
Glen Osmond SA 5064
Australia

Telephone: (08) 8313 0284
Facsimile: (08) 8303 7116

Email: jiaming.wang@adelaide.edu.au

Table of Contents

Abstract: 4

Executive summary: 4

Background: 5

Project Aims: 6

Travel outcomes: 6

APPENDIX I: Abstract of Conference Presentation 10

APPENDIX II: Poster Presentation 11

APPENDIX III: Photos of Laboratories Visiting 12

APPENDIX IV: Budget Reconciliation 13

Abstract:

The 9th In Vino Analytica Scientia Symposium was held in Trentino Alto Adige (Italy) from 14th to 17th July 2015. Approximately 250 registered delegates representing more than 30 countries attended the conference.

The conference covered up-to-date knowledge on excellence in analytical science of grapes, wines and spirits:

- Chemical analysis and composition of grapes, wines and spirits (2 sessions)
- Chemical and biochemical reactions
- Metabolomics, Chemometrics and Authenticity of products
- Sensory analysis

The travel grant provided by AGWA allowed Mr Jiaming Wang to attend the IVAS 2015 conference and present the poster presentation titled "Comprehensive study on Australian dry rosé wines – characterisation of chemical and sensory profiles". The grant also gave Mr Wang the opportunity to visit two laboratories in Europe, being those of Professor Rémi Schneider's group in Montpellier, France, and Professor Vicente Ferreira's group in Zaragoza, Spain. The travel inspired Mr Wang by connecting with great minds, helped him develop professional networks and allowed for the formulation of more ideas about his research.

Executive summary:

Mr Jiaming Wang, a PhD candidate at the University of Adelaide, received an AGWA travel grant in order to attend the 9th IVAS 2015 conference, held in Trento, Italy. The conference was held throughout 14th – 17th July. Mr Wang attended the conference with a poster presentation, "Comprehensive study on Australian dry rosé wines – characterisation of chemical and sensory profiles". The conference brought the pleasure and excitement of the continuous evolution in analytical science that inspires hundreds of researchers from all continents to gather every two years in an area renowned for the production of superb wines (to analyse and to taste) for the most up-to-date forum about excellence in analytical science

of grapes, wines and spirits. Over 4 days the symposium comprised 7 keynote presentations, 30 oral presentations and 203 posters. There was also a workshop on “Metabolomics applications to the grapevine and vine knowledge – the CRI-FEM approach” that was run before the oral presentations. There were 3 side events involving a technical visit, gala dinner and post-congress tour in Alto Adige – South Tyrol. According to the book of abstracts, the themes throughout the conference involved “a continuation of a successful series of conferences. This international meeting aims to bring together researchers dedicated to the different aspects of the chemistry of wine production: even including such aspects as environmental concerns, vines, grapes, and the final products, establishing a forum to discuss and present the latest developments of Analytical Chemistry.”. Attendance at the conference resulted in obtaining new knowledge and extending networks in the field of analytical chemistry in grape, wine and spirits which may lead to collaborations in the future. The additional laboratory visits offered a great opportunity to obtain first-hand information and learn on-site, which provided a lot of inspiration and motivation for the current research project. Also, these visits offer prospects for collaborative projects in the future.

Background:

Wine is a very complex matrix containing thousands of compounds with different physicochemical characteristics at different concentration ranges. When combined with the biological variability of grape and microflora, there are still many challenges remaining with regard to understanding and characterising wine. Nonetheless, the knowledge of chemistry in wine has been developing considerably. With the help of modern analytical instruments, along with metabolomics, chemometrics and sensory evaluation, not only has the amount of data grown geometrically, but also the quality of data has been improved in order to gain deeper knowledge and establish fundamental relationships between compositional data and sensory outcomes. Through this approach, eventually products could conceivably be modified to match specific market demands.

However, it has been widely realised that one of the largest problem is much more data has been produced than what we can really be understood. In other words, the limitation is no longer at the physical side, that being instruments and facilities, but rather in our minds when deciding what we should do to interpret data to yield some useful information. This limitation can be addressed through a process of continual learning and interacting with likeminded researchers at an international level.

IVAS 2015 conference provided a great opportunity for researchers all over the world to present the latest research outcomes, communicate research ideas, and share research resources, thereby developing the potential for collaborative projects. In particular, IVAS 2015 provided a great platform to be able to share information from our study on "Comprehensive study on Australian dry rosé wines – characterisation of chemical and sensory profiles". Funding from an AGWA travel grant provided financial support which enabled Mr Jiaming Wang to attend IVAS 2015 conference and visit two research groups in Europe to explore linkages.

Project Aims:

The aim of this project was to attend and present a poster at the IVAS 2015 conference in Trento, Italy, 14th – 17th July, 2015 and visit research groups in France and Spain.

Specific aims-

- Dissemination of research findings via conference presentation "Comprehensive study on Australian dry rosé wines – characterisation of chemical and sensory profiles" (Appendix I, II).
- Acquire latest and relevant knowledge, and inspiration regarding research methods that would help Mr Jiaming Wang's research project.
- Networking and sharing of research findings through laboratory visits.

Travel outcomes:

Conference

The conference was launched with an opening lecture entitled "Polyphenols and their relevance to wine sensorial quality" by Professor Victor de Freitas from Portugal. He basically explained the mechanism for astringency, bitterness and colour in regard to some polyphenol compounds. The mechanism for astringency was first proposed to be due to the ability of tannins to bind and precipitate salivary proteins. It is known that astringency increases upon successive exposures to tannins, and bitterness is caused by activating different taste receptors with the exposure of some polyphenols like catechins, tannins and anthocyanidin-3-gluscosides. The colour is due to the anthocyanins, which is extracted during

winemaking from grape skins. Their stability in wines may also be modulated by the interaction with polysaccharides present in wines. This talk brought a lot of new challenges, at the same time, opportunities for researchers, such as how to develop new tools/strategies ("omics") to follow grape "Phenolic" maturation, how to characterize objectively wine taste and astringency perception and how far the chemical transformations in wines can go and how to control them for technological purposes.

Dr Bruce Pan gave a talk on "Chemical characterization of California Chardonnay grapes for estimation of quality". Dr Pan is from one of the world's largest wine producers, E&J Gallo Winery based in Modesto, California. They conducted large-scale chemical profiling of around 8000 Chardonnay vineyards throughout California and tried to estimate the wine quality from the grape. They wanted to use real time objective chemical metrics to assist grape streaming at commercial harvest. Ethyl hexanoate was found as a fruity marker and 2/3-methybutyl acetates were related to stone fruit aroma. The level of YAN was correlated with 2-methylbutyl acetate and C6 compounds were negatively correlated with the value of final wine products. I had several conversations during the conference with Dr Pan and I am really interested in what they are doing. Dr Pan mentioned there are still many things in their big data set which they have not yet realised and understood. This linkage between research and the wine industry may lead to future cooperative projects between our organisations.

Another interesting oral presentation was given by Ernesto Franco-Luesma entitled "Production and/or release of volatile sulfur compounds (VSCs) along bottle storage: Dimensions of the problem and role of closure permeability to oxygen". What they found here is that the levels of free hydrogen sulfide (H_2S), methanethiol (MeSH) and dimethyl sulfide (DMS) of wines stored anoxically increased in all cases and three quarters of wines could be potentially affected by a transient reductive off-odour, and the rest by a permanent one. The proportion of free forms of H_2S increased during storage, suggesting it was the consequence of the release of bound forms, and the reduction of free H_2S and MeSH levels were effectively limited by closures. However, total MeSH and DMS levels were affected to a lesser extent by the closures permeability.

The most eye catching presentation was "Metabolomics, metallomics and sensorial analysis reveal the composition of 170-year-old champagne bottles from the Baltic Sea", given by Philippe Jeandet from France. They sampled a 170–year-old champagne wine found in a shipwreck at the bottom of the Baltic Sea. With the help of powerful analytical

instrumentation, they were able to reveal the characteristics in terms of small ion, sugar and acid contents as well as markers of barrel ageing and Maillard reaction products. After 170 years of deep sea aging in close-to-perfect conditions, the distinct aroma composition of these ancient champagne samples was first revealed during tasting sessions and also analysed using stir bar sorptive extraction-liquid desorption-gas chromatography-mass spectrometry (SBSE-LD-GC-MS).

Laboratory visits

After the conference I travelled to Montpellier and visited Nyséos (Appendix III, 1), the company where Professor Rémi Schneider is based. They have a very small but fully functional and comprehensive lab, and they are helping some large wine companies characterize only important volatiles in their base wine; using a software they developed (Kallosme, related news: http://en.sitevi.com/Archives/news/headlines/sitevi-show-vine-fruit-vegetable-olives/2015/innovation-awards/sitevi-2013-nyseos-remi-guerin-schneider-Kallosme-wine-aroma-management-software-innovation-international-show-exhibition-fruit-vegetable-growing-olives-production-vine-wine-making), they can use volatile data to help wine makers with blending options. They only work with those volatiles which have certain and obvious impact on sensory. Also, I visited the Unité Mixte de recherche Sciences Pour I'CEnologie (UMR SPO) and they have a very interesting micro fermentation lab (Appendix III, 2) where they keep the whole room at a relatively low temperature and use light to heat the fermentation flasks under computer control. With their online system they can even monitor the fermentation at home. We went to the Domaine de Pech-Rouge on the second day (Appendix III, 3), which is an experimental winery belonging to Institut national de la recherche agronomique (INRA). They are also doing some work on rosé wine under the collaboration with Provence rosé wine research centre. One thing I am interested in relates to the colour measurement; they are not only measuring the visible colour, but also look at the colour without SO_2 binding (adding acetaldehyde) (Appendix III, 4), and there is an obvious difference between these two forms. As SO_2 is always added into rosé wine in order to prevent oxidation, the colour will be changed once the SO_2 is consumed during storage, which may influence the consumers' liking of the products. This is a point to keep in mind as we continue our rosé project.

I also visited Professor Vicente Ferreira's group which is based in Zaragoza, Spain (Appendix III, 5). Prof Ferreira kindly showed me around in their labs and I was honoured to give their group a brief introduction of the activities of the wine science group at The University of Adelaide (Appendix III, 6). Currently everyone in Prof Ferreira's group is doing something related to wine oxidation aromas. In order to monitor wine aroma changing under ideal conditions, they developed a glove box containing an atmosphere of argon with levels of O_2 below 1 ppm (v/v) (Appendix III, 7). Additionally, it was great that I had the chance to observe the glassware they developed recently for a purge and trap aroma extraction system that we have adopted (Appendix III, 8). The aerodynamic shape and the volume they have are different, but it was good that I could learn about the prototype and note the difference between the two extraction systems.

APPENDIX I: Abstract of Conference Presentation

Comprehensive study on Australian dry rosé wines – characterisation of chemical and sensory profiles

Jiaming Wang, Kerry Wilkinson, David Jeffery

School of Agriculture, Food and Wine, The University of Adelaide

Abstract:

Rosé wine is a versatile and diverse style which is increasing in popularity in Australia and elsewhere, especially with young consumers. It is also an emerging style in Asian countries, being particularly suited to the cuisines from those regions, and development of relatively new markets such as China offers great potential to the Australian wine industry. The desirability of wine is strongly related to its sensory attributes, especially aroma and flavour, and it is important to understand which characteristics make a wine attractive so these can be targeted through viticulture and winemaking. However, there is an absence of research related to compounds that are important to rosé wines produced in Australia. In this investigation, qualitative and quantitative SPME-GC-MS methods have been developed and used to identify over 40 volatile compounds present in a range of commercial rosé wines from Australia (n = 27) and the Provence region of France (n = 3). Wines were also analysed using descriptive analysis (DA) with 12 trained sensory panellists. Attributes generated from DA were applied in blind rosé wine tastings (of representative wines from Australia, France and China) involving Chinese wine industry employees (n = 62) in three major cities in China. Of the different rosé wine styles determined during sensory studies, which included fruit-driven and more developed wines, fruity/floral and tropical/citrus styles were chosen for further exploration to evaluate the key aroma compounds. Information on the composition and sensory properties of Australian rosé wines, together with results from a sensory study involving experts in an emerging market will be presented.

APPENDIX II: Poster Presentation

Comprehensive study of Australian rosé wines: characterisation of chemical and sensory profiles

Jiaming Wang, Kerry Wilkinson and David Jeffery
School of Agriculture, Food and Wine, Waite Research Institute, The University of Adelaide, PMB 1, Glen Osmond, South Australia 5064, Australia. E-mail: jiaming.wang@adelaide.edu.au

Introduction



Rosé wine is a versatile and diverse style which is increasing in popularity in Australia and elsewhere. The development of new markets such as China offers great potential to the Australian wine industry.

It is important to understand what aroma and flavour make a wine attractive. However, there is an absence of research related to the volatile compounds that are important to rosé wines produced in Australia.

Research questions

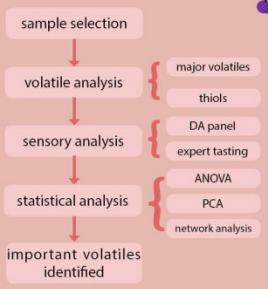
- What volatile compounds are important for Australian rosé wine?
- What styles of rosé wine do Chinese consumers prefer?

Materials and methods



- Rosé wine samples: Australian (26), Provence (2) and Chinese (3).
- Volatile analysis: Major volatiles were measured by a HS-SPME-GC-MS method and 5 thiols were quantified by a recent HPLC-MS/MS method (Capone, 2015).
- Sensory analysis: A descriptive analysis (DA) panel with 12 panellists was conducted in Australia and 62 Chinese wine industry professionals were recruited to take part in one of the three blind rosé wine tastings conducted in three major cities of China.
- Statistical analysis: All data were preliminarily processed with Microsoft Excel 2010. ANOVA and PCA were conducted by XLSTAT. Network analysis (NA) was carried out with Gephi on all significantly different variables.





Results and discussion

- 51 volatile compounds were quantified, including 5 potent sulfur compounds.
- Half were ethyl and acetate esters, which are typically associated with fruity and floral aromas (Darici, 2014).
- The concentration of 3-MH in all samples was above its aroma detection threshold.
- FFT and BM were detected in most samples at concentrations above their aroma detection thresholds.

compounds (µg/L)	threshold[a]	OAV range	OAV mean	descriptor[b]
ethyl acetate	15000	5-17.7	12.4	nail polish
ethyl butanoate	20	0-28	12.8	strawberry
ethyl 2-methylbutanoate	1	0-194	31.7	fruity
ethyl 3-methylbutanoate	3	0-131.7	29.7	fruity
3-methylbutyl acetate	30	0-284.1	100.2	banana
ethyl hexanoate	14	0-134.6	62.5	apple peel
hexyl acetate	670	0.1-12.6	3.9	floral
ethyl octanoate	5	0-1357.4	540.8	melon
ethyl decanoate	200	0.8-28.1	14.9	floral
2-phenylethyl acetate	250	0-2.7	0.7	floral
β-damascenone	0.05	0-594	96	rose
3-MH (ng/L)	60	3.6-54.2	11.9	grapefruit
3-MHA (ng/L)	4	0-2.1	0.3	passionfruit
4-MMP (ng/L)	0.8	0-3	0.6	boxwood
FFT (ng/L)	0.4	0-52.8	8	roasted coffee
BM (ng/L)	0.3	4.3-17.7	9	smoke

Table 1. Main esters and thiols quantified in 26 Australian rosé wines. [a] Refer to Ferreira, et al. (2000) and Dubourdieu, et al. (2009). [b] Obtained from flavornet (http://www.flavornet.org).

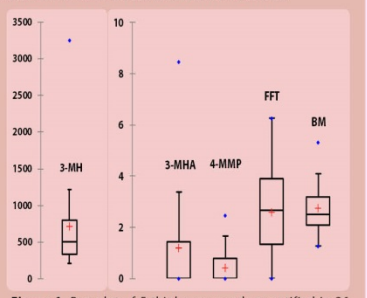


Figure 1. Box plot of 5 thiol compounds quantified in 26 Australian rosé wines (ng/L).

Rosé wine tasting in China

- The first investigation into opinions of Chinese wine industry professionals towards Australian rosé wines.
- Judges preferences for all samples were from "neither like nor dislike" to "like moderately".
- The first insight into Chinese rosé wines which tended to exhibit three different styles.
- One Chinese rosé wine was the third most popular rosé of all 23 samples.

Network analysis

- In total, 32 positive correlations ($r > 0.7$) were found among 34 variables.
- Some sensory attributes were correlated closely with preference , quality and expected price .
- Most related volatiles could be explained as arising from alcoholic fermentation by wine yeast.
- This is the first time that relationships among different wine sensory attributes and compositional data have been evaluated using network analysis.

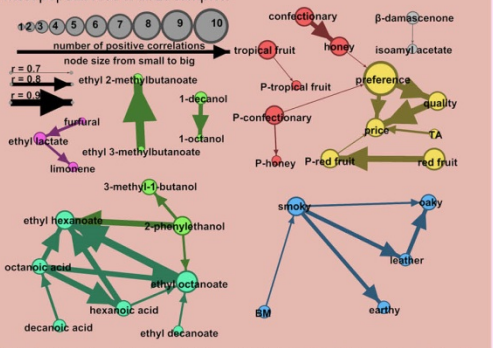
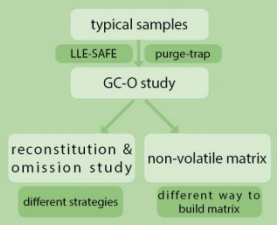


Figure 2. Network analysis on all variables which had positive relationships ($r > 0.7$) with each other in 23 rosé samples included in blind rosé wine tastings in China.

Conclusions and futher direction

- β-Damascenone, 3-methylbutyl acetate and ethyl hexanoate were important in accordance with other studies. Thiols (3-MHA, 3-MH and others) also contributed to rosé wine aroma.
- The preferred rosé wine presented red fruit, floral, confectionery and honey characters with low sugar content and was also described as easy to drink without developed characters.



Scan the QR code

Supplementary information can be found here.

References

1. Capone, D. L., Ristic, R., Pardon, K. H., & Jeffery, D. W. (2015). Simple quantitative determination of potent thiols at ultratrace levels in wine by derivatization and high-performance liquid chromatography–tandem mass spectrometry (HPLC-MS/MS) analysis. *Analytical Chemistry, 87* (2), 1226-1231.
2. Darici, M., Cabaroglu, T., Ferreira, V., & Lopez, R. (2014). Chemical and sensory characterisation of the aroma of Callaroso rosé wine. *Australian Journal of Grape and Wine Research, 20* (2), 340-346.
3. Ferreira, V., López, R., & Cacho, J. F. (2000). Quantitative determination of the odorants of young red wines from different grape varieties. *Journal of the Science of Food and Agriculture, 80* (1), 1659-1667.
4. Dubourdieu, D., & Tominaga, T. (2009). Polyfunctional thiol compounds. In M. V. Moreno-Arribas & M. C. Polo *Wine Chemistry and Biochemistry*. New York: Springer.



About the author
Jiaming Wang, PhD candidate, obsesses in wine and believes in science.

Acknowledgment
Dr. Dimitra L. Capone for thiols analysis.
The dearest panellists and wineries from Australia and China.

THE UNIVERSITY of ADELAIDE

CHINA SCHOLARSHIP COUNCIL

Australian Government
Australian Grape and Wine Authority

APPENDIX III: Photos of Laboratories Visiting



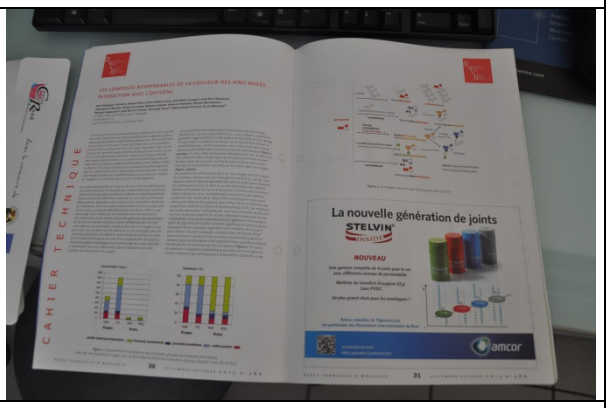
1



2



3



4



5



6



7



8

APPENDIX IV: Budget Reconciliation

The End of Project Financial Statement will be submitted separately via Clarity