# Phenolic Extraction and Juice Expression during White Wine Production

by

#### Simon Jonathan Nordestgaard

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# ABSTRACT

Phenolic compounds are important constituents of white wine that can impart bitterness and astringency and influence stability and colour. The specific types and concentrations of phenolic compounds vary between the different grape tissues. For example grape skins contain much higher concentrations of phenolics than grape pulp. Consequently, grape processing techniques, particularly the method by which juice is expressed, can influence the phenolic profile of white wines. Concerns of elevated phenolic levels tend to lead to conservative practices such as limiting the time between machine harvesting and winery processing, and the use of relatively expensive batch draining and pressing equipment.

Phenolic extraction during pomace contact, such as might occur after machine harvesting prior to winery processing, was investigated at a controlled laboratory scale. Contact time significantly increased phenolic extraction as did the fraction of broken berries. However, given the uncertainty in the amount of berry breakage in industrial practice, full scale transport trials should be performed by wineries where economic advantage could be gained from relaxing restriction on times between harvesting and winery processing.

Techniques used for white grape juice expression both now and in the past were critically reviewed and winery sampling was performed to develop a practical understanding of operational issues. For the expression of juice from white grapes, pneumatic membrane presses have been increasingly adopted both for pressing and draining. This is principally a product of their ability to express high yields of juice with relatively low levels of phenolics and suspended solids. These devices can be operated in many different manners and quality and productivity is highly dependent on the specific mode of operation. Adaptive programmes based principally on continuous assessment of juice flow rate, and the use of conductivity measurement to monitor skin extraction are important tools that can aid economic optimisation of expression operations.

The principal problem with the pneumatic membrane press is that it is a batch operation with a relatively low throughput. In the longer term, the ideal outcome would be the development of expression equipment that achieved the high throughput and relatively low cost per tonne of continuous inclined drainers and screw presses, while still maintaining the quality obtained with pneumatic membrane presses. Continuous devices that mimic the repeated cycles of compression and crumbling of these batch presses may be one means of achieving this. Exploration of the different continuous screw presses used historically suggests that there still may be room for improvement in these devices. This merits further specific investigation.

Experiments involving repeated cycles of compression and crumbling were performed with a constant rate laboratory apparatus. These experiments demonstrated the importance of bed height, pressing speed, sieve plate design and crumbling on press operation. Solids content appears to be a major

i

issue in the development of rapid juice expression equipment. One means of achieving appropriately low solids levels, without requiring large devices with large cake beds for juice filtration, may be to maintain the structure of the grape for as long as possible so that juice is filtered as it is expelled from the berry.

# THESIS DECLARATION

This work contains no material which has been accepted for the award of any other degree or diploma in any university or other tertiary institution to Simon Jonathan Nordestgaard 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.

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Simon Jonathan Nordestgaard 05/07/2011

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# TABLE OF CONTENTS

ABSTRACT	i
THESIS DECLARATION	iii
ACKNOWLEDGEMENTS	iv
REFEREED PUBLICATIONS	v
TABLE OF CONTENTS	vi
LIST OF FIGURES	ix
LIST OF TABLES	xiii
CHAPTER 1: INTRODUCTION	
1.1 Project background	2 2
1.2 Thesis objective	2
1.3 Thesis structure	2
1.4 Terminology	3
CHAPTER 2: PHENOLIC COMPOUNDS IN WHITE WINE, THEIR ORIGIN AND INFLUENC	
QUALITY	4
2.1 Phenolic compounds in white wine and their origin	4
2.1.1 Non-flavonoid phenolic compounds	5
2.1.1 Hydroxycinnamates	5
2.1.2 Hydroxybenzoic acids	5
2.1.3 Tyrosol	6
2.1.4 Stilbenes	6
2.1.5 Hydrolysable tannins	6
2.1.6 Volatile phenols	6
2.1.2 Flavonoid phenolic compounds	7
2.1.2.1 Flavan-3-ols and proanthocyanidins	7
2.1.2.2 Flavonols	7
2.1.2.3 Flavanonols	7
2.2 Grape structure and expression related composition	8
2.3 White wine taste and mouthfeel	10
2.3.1 Non-flavonoid phenolic compounds	10
2.3.2 Flavonoid phenolic compounds	11
2.3.3 Complex phenolic oxidation adducts	12
2.4 White wine stability	12
2.5 Conclusions	13
CHAPTER 3: EXTRACTION OF PHENOLIC COMPOUNDS DURING POMACE CONTACT	14
3.1 Materials and methods	14
3.1.1 Grape picking	14
3.1.2 General characterisation	15
3.1.2.1 Grape characterisation	15
3.1.2.2 Juice characterisation	15
3.1.3 Grape component phenolic characterisation	16
3.1.3.1 Solvent extraction	16
3.1.3.2 Phenolic analysis	16
3.1.4 Pomace contact studies	17

3.1.4.1 Complete berry crushing	17
3.1.4.2 Partial berry crushing	17
3.1.4.3 Phenolic analysis	18
3.1.4.4 Regression analysis	18
3.2 Results and discussion	19
3.2.1 General grape and juice characterisation	19
3.2.2 Component phenolic characterisation	20
3.2.3 Berry component and juice phenolic concentration	22
3.2.4 Fundamental processes during pomace contact	24
3.2.5 Pomace contact (complete berry crushing)	26
3.2.6 Pomace contact (partial berry crushing)	33
3.2.7 Yields	38
3.2.8 Analytical techniques	38
3.3 Conclusions	42
CHAPTER 4: JUICE EXPRESSION OBJECTIVES, DESTEMMING, CRUSHING AND DRAINING	<b>4</b> 4
4.1 Objectives in white juice expression	44
4.2 Destemming and crushing	45
4.3 Non-mechanical treatments to facilitate expression	48
4.4 Draining	49
4.4.1 Batch/static drainers	49
4.4.2 Continuous drainers	54
4.5 Conclusions	58
CHAPTER 5: PRESSING	59
5.1 Batch presses	59
5.1.1 Vertical basket presses	59
5.1.2 Rack and cloth presses	59
5.1.3 Horizontal plate presses	61
5.1.4 Pneumatic presses	68
5.2 Continuous presses	78
5.2.1 Screw presses	78
5.2.2 Belt presses	87
5.2.3 Pneumatic presses	88
5.2.4 Decanter centrifuges	90
5.3 Conclusions	91
CHAPTER 6: ECONOMIC CONSIDERATIONS IN JUICE EXPRESSION	92
6.1 Batch and continuous equipment	92
6.2 Pneumatic membrane press operation and press cuts	94
6.3 Conclusions	97
CHAPTER 7: WINERY SAMPLING OF JUICE EXPRESSION EQUIPMENT	99
7.1 Materials and methods	99
7.1.1 Juice expression strategies	99
7.1.2 Sampling points	104
7.1.3 Sample analysis	112
7.2 Results and Discussion	114
7.2.1 Phenolic content	115
7.2.2 Conductivity	117
7.2.3 Solids content	121

7.2.4 Operational observations	126
7.3 Conclusions	126
CHAPTER 8: EXPRESSION TECHNIQUES IN OTHER INDUSTRIES	127
8.1 Red wine production	127
8.2 Waste treatment sludge dewatering	127
8.3 Apple juice production	128
8.4 Oilseed processing	132
8.5 Sugar cane processing	135
8.6 Conclusions	139
CHAPTER 9: FUNDAMENTAL STUDIES OF GRAPE JUICE EXPRESSION	140
9.1 Materials and methods	140
9.1.1 Laboratory pressing apparatus	140
9.1.2 Experimental technique	142
9.1.3 Sample analysis	143
9.2 Results and discussion	144
9.2.1 General observations	144
9.2.2 Crumbling	147
9.2.3 Bed height	149
9.2.4 Pressing speed and sieve plate design	151
9.2.5 Whole or crushed berries	153
9.2.6 Constant rate and constant pressure compression	156
9.2.7 Analytical implications for drainer/press development, optimisation and control	158
9.3 Conclusions	160
CHAPTER 10: KEY FINDINGS AND RECOMMENDATIONS	162
10.1 Harvesting and transport prior to winery processing	162
10.2 Operation of existing juice expression equipment	162
10.3 Development of new juice expression equipment	163
REFERENCES	166
APPENDIX A: ADDITIONAL DATA FOR CHAPTER 3	179
APPENDIX B: ADDITIONAL DATA FOR CHAPTER 7	184
APPENDIX C: ADDITIONAL DATA FOR CHAPTER 9	188

# **LIST OF FIGURES**

Figure 1.1: Simplified white table wine production process with a summary of thesis coverage	1
Figure 2.1: Flavonoid ring system with ring and position labels	4
Figure 2.2: Structure of selected non-flavonoid phenolic compounds	6
Figure 2.3: Structure of selected flavonoid phenolic compounds	8
Figure 2.4: Longitudinal section of one side of a grape berry showing its constituent tissues	10
Figure 3.1: (a) Draining plate and (b) simple ram	15
Figure 3.2: Grape component extract spectra (component concentration, with and without $PV$ treatment)	/PP 21
Figure 3.3: Seed and skin extract spectra (berry concentration, with and without PVPP treatment)	22
Figure 3.4: Spectra of phenolic standards (100 mg/L)	22
Figure 3.5: Influence of pomace contact (100% crushed, 50 mg/kg SO <sub>2</sub> , approximately 12 $^{\circ}$ C) spectra for different lots	on 23
Figure 3.6: Influence of crushing on constituents for pomace contact	25
Figure 3.7: Cellular representation of a section of skin with pulp attached submerged in the alreater expressed bulk juice	ady 26
Figure 3.8: Extraction kinetics for Barossa Valley Chardonnay (100% crushed, Replicate 2)	29
Figure 3.9: Extraction kinetics for Eden Valley Riesling (100% crushed)	31
Figure 3.10: Extraction kinetics for Langhorne Creek Chardonnay (50 mg/kg $SO_2$ , Replicate 1)	34
Figure 3.11: Extraction kinetics for Eden Valley Riesling (50 mg/kg SO <sub>2</sub> )	36
Figure 3.12: Spectra after treatment of juice samples with 100 g/L PVPP (Samples from poma contact experiments, 100% crushed, 50 mg/kg SO <sub>2</sub> , approximately 23 °C)	ace 41
Figure 4.1: Destemmer paddles (view from stem-exit end)	47
Figure 4.2: Destemmer cage (view from outside)	47
Figure 4.3: Crusher rollers	48
Figure 4.4: MacKenzie static type separator	50
Figure 4.5: Potter drainer/fermenter	51
Figure 4.6: MacKenzie carbon dioxide pressure separator	52
Figure 4.7: Miller 20-ton twin screw pressure drainer	53
Figure 4.8: Winery Systems juice separator	53
Figure 4.9: Pera Elite Tank	54
Figure 4.10: Rotating cylindrical draining screen prior to press basket loading	56
Figure 4.11: Inclined drainer in conjunction with a screw press	56
Figure 4.12: Static anti-return device on a partially disassembled inclined drainer	57
Figure 4.13: Inclined drainer feed hopper with brushes on screw thread	57
Figure 5.1: Vertical basket press	60
Figure 5.2: Rack and cloth press	61
Figure 5.3: Vaslin internal spindle press with two moving compression plates	63
Figure 5.4: Willmes WHA external spindle press with one moving compression plate	64
Figure 5.5: Bucher hydraulic press with one moving compression plate	64

Figure 5.6: Ropes in a Willmes ABC press	65
Figure 5.7: Rings and chains in a Vaslin 3.2 m <sup>3</sup> press	65
Figure 5.8: Operation of Vaslin-style internal spindle press with two moving compression plates a ring and chain system	nd a 66
Figure 5.9: Pressure profile for pressing of whole clusters of Sauvignon Blanc grapes in manual m with a Vaslin 22 VT internal spindle press with two moving compression plates and rotating spindle	
Figure 5.10: Vaslin press with annular doors	66
Figure 5.11: Vaslin CEP 250S press	67
Figure 5.12: Vaslin CEP press operation	67
Figure 5.13: Hypothesised variable degree of expression in horizontal plate press (conceptual onl	y)67
Figure 5.14: Willmes pneumatic press	72
Figure 5.15: Internal view of a Bucher-Vaslin XP320 membrane press (membrane retracted)	72
Figure 5.16: Drainage channels in a Bucher-Vaslin XP320 membrane press	73
Figure 5.17: Three-dimensional drainage system for Bucher-(Vaslin) presses	73
Figure 5.18: Vertical central stainless steel juice channels in a Willmes press	74
Figure 5.19: Vertical central flexi-drain juice channels in a Willmes press	74
Figure 5.20: Generalised pneumatic press arrangements	75
Figure 5.21: Standard press programme (conceptual only)	76
Figure 5.22: Sequential press programme (conceptual only)	76
Figure 5.23: Internal view of a Bucher-Vaslin XP320 membrane press showing axial filling (membrane retracted)	inlet 77
Figure 5.24: Spiral elements set under retracted press membrane to convey pressed pomace to door(s) during emptying by tank rotation	exit 77
Figure 5.25: Colin screw press with twin end on end counter-rotating screws	81
Figure 5.26: Screw press toothed obturator wheel	81
Figure 5.27: Mabille screw press c. 1929	82
Figure 5.28: Selected screw presses (partial circular protrusions at the end of the hopper, I conceal toothed anti-return wheels, not at same scale)	ikely 82
Figure 5.29: Coq Bi-valve continuous screw press	83
Figure 5.30: Marzola PAP100 continuous screw press	83
Figure 5.31: First screw section of Marzola PAP1000 continuous screw press	84
Figure 5.32: Second screw section of Marzola PAP1000 continuous screw press	84
Figure 5.33: Anti-return device of Marzola PAP1000 continuous screw press	85
Figure 5.34: Partially assembled Marzola PAP1000 continuous screw press with anti-return de installed	evice 85
Figure 5.35: Overhead view of a hopper of an empty Marzola PAP1000 continuous screw press	86
Figure 5.36: Marzola PAP1000 continuous screw press screens viewed from (a) inside and outside the chamber	d (b) 86
Figure 5.37: Rietz/Vincent Cushion Cone continuous screw press	87
Figure 5.38: Sernagiotto NOLM belt press	88
Figure 5.39: MacKenzie pneumatic press	89
Figure 5.40: Siprem continuous membrane press	90

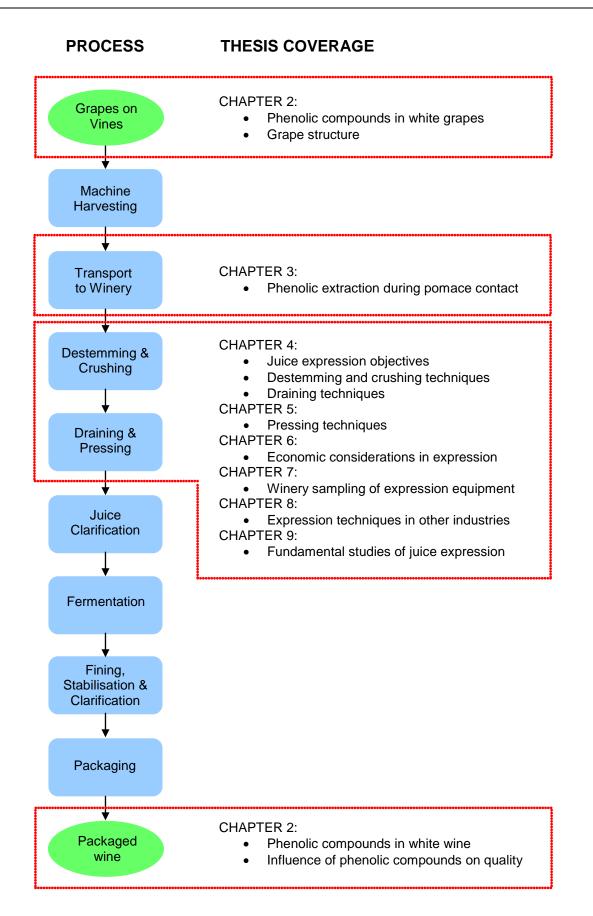
Figure 5.41: Westfalia decanter centrifuge91
Figure 7.1: Simplified flow diagram of juice expression at Winery A 101
Figure 7.2: Simplified flow diagram of juice expression using Process 1 at Winery B 102
Figure 7.3: Simplified flow diagram of juice expression using Process 2 at Winery B 103
Figure 7.4: Draining section at feed to destemmer-crusher105
Figure 7.5: Underside of draining section at feed to destemmer-crusher (drain hole to buffer tank shown) 105
Figure 7.6: Buffer tank for destemmer-crusher run-off 106
Figure 7.7: Centrifugal pump for pumping out destemmer-crusher run-off buffer (sampling location marked) 106
Figure 7.8: Miller 800 mm inclined drainer107
Figure 7.9: Inclined drainer screens (juice dripping onto plastic drip sheets) 107
Figure 7.10: Underside view of inclined drainer (sampling location marked) 108
Figure 7.11: Coq 800 mm Bi-valve continuous screw press 108
Figure 7.12: Winery A continuous screw press sampling locations 109
Figure 7.13: Miller static drainers (door of drainer #5 opening to evacuate pomace onto screw- conveyors) 109
Figure 7.14: Velo PMC550 membrane press110
Figure 7.15: Two of the buffer tanks into which static drainer juice or membrane press juice flows 110
Figure 7.16: Sample tap immediately downstream of static drainer and membrane press buffer tanks 111
Figure 7.17: Marzola PAP1000 continuous screw presses       111
Figure 7.18: Rotary screens servicing 2 screw presses (Winery B - Process 1 sampling locations marked) 112
Figure 7.19: Stainless steel strainer and PP funnel for preliminary bulk solids removal 113
Figure 7.20: Spin test tubes for maximum (a) 3%, (b) 6%, and (c) 10% solids content 113
Figure 7.21: Juice sample spectra from Winery A and Winery B – Process 1 117
Figure 7.22: A <sub>280</sub> along the length of the screw press at winery A 117
Figure 7.23: Conductivity along the length of the screw press at Winery A 119
Figure 7.24: Conductivity with respect to yield and time during sampling of a membrane press at Winery C while axial filling, draining and pressing 120
Figure 7.25: Relationship between conductivity and A <sub>280</sub> for juice samples collected during winery sampling
Figure 7.26: Solids content along the length of the screw press at Winery A 123
Figure 7.27: Microscopy (100 ×) for Winery A samples 124
Figure 7.28: Microscopy (100 ×) for Winery B – Process 1 samples 125
Figure 7.29: Microscopy (100 ×) for Winery B – Process 2 samples 125
Figure 8.1: Bucher HPX 5005iP horizontal hydraulic single plate press with cloth covered drainage cores and schematic of operating prinicple 129
Figure 8.2: Jones Pressmaster vertical screw press 131
Figure 8.3: Ensink belt press for apple juice production 131
Figure 8.4: Flottweg belt press for apple juice production132

Figure 8.5: Schematic of Flottweg belt press for apple juice production	132
Figure 8.6: Partially disassembled oilseed screw press	134
Figure 8.7: Anderson Super duo Expeller 55 with conditioner	134
Figure 8.8: Traditional three-roll sugar cane mill	137
Figure 8.9: (a) Simple two-roll sugar cane mill model and (b) mill pressure profile	137
Figure 8.10: Sugar cane mill roll surfaces with Messchaert grooves	138
Figure 8.11: Sugar cane Lotus roller	138
Figure 8.12: Bundaberg High Extraction two-roll sugar cane mill	139
Figure 9.1: Laboratory pressing apparatus	141
Figure 9.2: (a) Standard, and (b) alternate sieve plates	142
Figure 9.3: Laboratory pressing apparatus (a) mount, and (b) compression cylinder	142
Figure 9.4: Pressure and juice expression profile during the first press cycle of 1 kg of whole vintage Barossa Valley Chardonnay grapes at 10 mm/min (with corresponding photos of pomace	
Figure 9.5: Representation of part of the cake structure during different stages of compression	146
Figure 9.6: Pressure and juice expression profile during repeated press cycles of 1 kg of whole vintage Barossa Valley Chardonnay grapes at 10 mm/min	2009 146
Figure 9.7: Yield comparison with and without crumbling for pressing of 1 kg of whole 2009 vi Barossa Valley Chardonnay grapes at 10 mm/min	ntage 148
Figure 9.8: Yield comparison with and without crumbling for pressing of 1 kg of 100% pre-cru 2009 vintage Barossa Valley Chardonnay grapes at 10 mm/min	ushed 149
Figure 9.9: Effect of bed height on solids and phenolic content for whole 2009 vintage Barossa V Chardonnay grapes pressed at 10 mm/min	Valley 150
Figure 9.10: Effect of bed height and press speed on juice yield for whole 2009 vintage Lang Creek Chardonnay grapes	horne 151
Figure 9.11: Effect of pressing speed on solids and phenolic content for 500 g of whole 2009 vi Langhorne Creek Chardonnay grapes	ntage 153
Figure 9.12: effect of pressing speed and sieve plate design on solids and phenolic content for of whole 2009 vintage Adelaide Hills Chardonnay grapes	125 g 153
Figure 9.13: Influence of the fraction of pre-crushed berries on cumulative yield for 1 kg of vintage Eden Valley Riesling pressed at 10 mm/min	2008 155
Figure 9.14: Effect of pre-crushing on solids and phenolic content for 500 g of 2009 vi Langhorne Creek Chardonnay grapes pressed at 10 mm/min	ntage 155
Figure 9.15: Representation of juice expellation under different regimes	156
Figure 9.16: Effect of juice back-addition on solids and phenolic content for 500 g of pre-crushed vintage Langhorne Creek Chardonnay grapes pressed at 10 mm/min	2009 156
Figure 9.17: Generalised pressure and juice expression profile under constant rate and cor pressure compression for an individual press cycle	nstant 158
Figure 9.18: Relationship between conductivity and A <sub>280</sub> for juice samples collected during 200 2009 vintage laboratory pressing experiments	8 and 159
Figure 9.19: Relationship between pH and $A_{280}$ for juice samples collected during 2008 and vintage laboratory pressing experiments	2009 160

# LIST OF TABLES

Table 2.1: Indicative levels of phenolics in young white table wines without oak contact	5
Table 3.1: Berry component mass fractions	19
Table 3.2: Berry diameter and mass	19
Table 3.3: General juice analyses	20
Table 3.4: HPLC analysis of selected juice samples	24
Table 3.5: Regression analysis for Barossa Valley Chardonnay (100% crushed)	30
Table 3.6: Regression analysis for Eden Valley Riesling (100% crushed)	32
Table 3.7: Regression analysis for Langhorne Creek Chardonnay (50 mg/kg SO <sub>2</sub> )	35
Table 3.8: Regression analysis for Eden Valley Riesling (50 mg/kg SO <sub>2</sub> )	37
Table 3.9: Indicative quantification of phenolic levels for Langhorne Creek Chardonnay regression model (20% crushed, 50 mg/kg SO <sub>2</sub> , 400 L/tonne, 20 $^{\circ}$ C)	using 38
Table 3.10: HPLC analysis of juice samples with and without PVPP treatment	42
Table 6.1: Capital cost comparison for batch and continuous expression equipment	94
Table 6.2: Loss in revenue from press cuts prior to the yield where the free-run quality wo detrimentally affected	ould be 96
Table 7.1: Winery A analytical results	114
Table 7.2: Winery B – Process 1 analytical results	114
Table 7.3: Winery B – Process 2 analytical results	115
Table 7.4: Coefficient of determination ( $R^2$ ) between $A_{280}$ and conductivity for juice samples coduring winery sampling	llected 121
Table 9.1: Analytical results for samples collected during pressing of 1 kg of whole 2009 v Barossa Valley Chardonnay grapes at 10 mm/min	vintage 147
	-

Table 9.2: Coefficient of determination ( $R^2$ ) between  $A_{280}$  and conductivity or pH for juice samplescollected during 2008 and 2009 vintage laboratory pressing experiments160





1

# **CHAPTER 1: INTRODUCTION**

Phenolic compounds are important constituents of white wine that can impart bitterness and astringency and influence wine stability and colour. Many phenolic compounds in white wine are derived from the grape, where specific phenolic compounds occur at different concentrations in the different grape tissues (i.e. pulp, skins and seeds). Consequently grape processing techniques, particularly the method by which juice is expressed, can influence the phenolic profile of white wine.

Concerns of elevated phenolic levels in white wine derived from grape skins or seeds tend to lead to conservative practices and relatively expensive processing techniques. Limiting the time between harvesting and winery processing, and the use of batch draining and pressing equipment, are two examples.

# 1.1 Project background

Interest in phenolic management in the Australian wine industry led to the commencement of a collaborative phenolic-related project between The School of Chemical Engineering at The University of Adelaide, The Australian Wine Research Institute and Pernod Ricard Pacific. The project was funded principally by the Grape and Wine Research and Development Corporation with additional funding from Pernod Ricard Pacific and in-kind contributions from all collaborating organisations. The work reported in this thesis was funded by this project.

# **1.2 Thesis objective**

To meet the goals of the larger project as well as to address wine industry and literature knowledge gaps, the objective of this thesis was to build knowledge that would ultimately:

- (a) Improve the management of phenolics during grape harvesting and transportation;
- (b) Improve the operation of existing juice expression equipment; and
- (c) Guide the development of new and improved juice expression equipment.

## **1.3 Thesis structure**

The relevance of different chapters in this thesis to specific processing steps in the production of white wine is summarised in Figure 1.1.

The phenolic compounds found in white wine, their origin and influence on white wine quality are reviewed in Chapter 2, together with information on the structure of the grape.

Results from laboratory studies of phenolic extraction during pomace contact are presented in Chapter 3.

Chapters 4 and 5 review juice expression techniques used now or in the past for white wine production. Overall juice expression objectives are discussed in Chapter 4 together with destemming, crushing and draining techniques, while equipment used for pressing is reviewed in Chapter 5. Economic considerations in white juice expression are discussed in Chapter 6. Results from winery sampling of juice expression equipment are presented in Chapter 7.

Techniques employed for expression in other industries are reviewed in Chapter 8.

Results from fundamental laboratory studies of grape juice expression are presented in Chapter 9.

Context and background literature where applicable are presented at the start of each chapter. Conclusions are provided at the completion of each chapter and overall key findings and recommendations are presented in Chapter 10.

# 1.4 Terminology

The terms extraction and expression are often used interchangeably. For example, extraction is commonly used in the wine industry in the context of press juice yields.

In this thesis, "extraction" will be reserved to refer to the transport of phenolic compounds such that they appear in a different expressed juice yield.

"Expression" will be the term used for the actual collection of juice. This term will be used to cover the common winemaking unit operations of draining and pressing, and for simplicity, the associated unit operations of destemming and crushing.

When analysing juice expression operations in more detail, the term "expelling" will be used to describe the local release of juice from a grape or section of grape. For example: In the instance of a bed of grapes being compressed the juice is expelled from an individual berry in that bed into the pores of the bed, while the overall global collection of juice from the bed of grapes will be referred to as expression.

# CHAPTER 2: PHENOLIC COMPOUNDS IN WHITE WINE, THEIR ORIGIN AND INFLUENCE ON QUALITY

In this chapter the phenolic compounds in white wine and their origin are summarised. Information on the structure of the grape is also presented. The effect of phenolics on white wine taste and mouthfeel and their association with white wine colour and stability are then discussed.

## 2.1 Phenolic compounds in white wine and their origin

Phenolics (or phenols) are compounds possessing an aromatic ring with one or more hydroxyl substituents (Macheix et al. 1990, Waterhouse 2002). They can be broadly categorised as either flavonoid or non-flavonoid phenolic compounds on the basis of whether or not they posses the flavonoid ring system shown in Figure 2.1. Specific sub-groups of non-flavonoids and flavonoids are reviewed in sections 2.1.1 and 2.1.2, respectively.

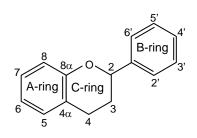


Figure 2.1: Flavonoid ring system with ring and position labels (Adapted from: Kennedy et al. 2006)

White wine phenolic concentrations reported in the literature vary greatly. This is partly a consequence of actual phenolic levels being heavily influenced by grape variety, viticultural and oenological practices; however, it is also a product of the diverse analytical techniques used in their analysis. Waterhouse (2002) provided one of few overall summaries of the concentrations of the different groups of phenolic compounds in white wine. This has been adapted in Table 2.1 to provide order of magnitude levels of the different phenolic sub-groups for the consideration of the reader in subsequent discussions.

Phenolic group	Concentration (mg/L) <sup>a</sup>
Non-flavonoids	
Hydroxycinnamates	150
Hydroxybenzoic acids	10
Tyrosol	20
Stilbenes	0.5
Hydrolysable tannins	0
Volatile phenols	Traces
Flavonoids	
Flavan-3-ols	25
Proanthocyanidins	20
Flavonols	2
Flavanonols	0.5
Total	228

Table 2.1: Indicative levels of phenolics in young white table wines without oak contact

<sup>a</sup> Adapted principally from Waterhouse (2002), with the addition of tyrosol (Singleton 1992, Ribéreau-Gayon et al. 2006a), volatile phenols (Singleton 1992), flavanonols (Singleton and Trousdale 1983) and modified values for flavonols (Makris et al. 2006).

## 2.1.1 Non-flavonoid phenolic compounds

#### 2.1.1 Hydroxycinnamates

Hydroxycinnamates and their derivatives are the predominant non-flavonoid phenolics in white wine (Waterhouse 2002, Kennedy 2006). They exist in the grape principally in the form of trans-esters of tartaric acid, and are found both in the vacuoles of the pulp cells and in even higher concentrations in the skin cell vacuoles (Somers and Vérette 1988). They also occur in grape stems (Souquet et al. 2000a).

Caftaric acid is the most abundant hydroxycinnamate ester followed by coutaric and fertartic acids (Somers et al. 1987). The tartaric acid esters undergo partial hydrolysis to free hydroxycinnamic acids during fermentation and aging (Somers et al. 1987), for example caftaric acid (Figure 2.2a) yields caffeic acid (Figure 2.2b). Additionally, a glutathionyl derivative of caftaric acid can result from enzymatically catalysed oxidation of caftaric and coutaric acids during grape and juice processing (Singleton et al. 1985).

#### 2.1.2 Hydroxybenzoic acids

Hydroxybenzoic acids are minor components of new wine, but hydrolysis of gallate esters of condensed and hydrolysable tannins can result in an increase in gallic acid (Figure 2.2c) concentration with time (Waterhouse 2002).

#### 2.1.3 Tyrosol

Tyrosol (Figure 2.2d) is a phenolic compound that may be formed during alcoholic fermentation from the amino acid tyrosine (Singleton and Noble 1976).

#### 2.1.4 Stilbenes

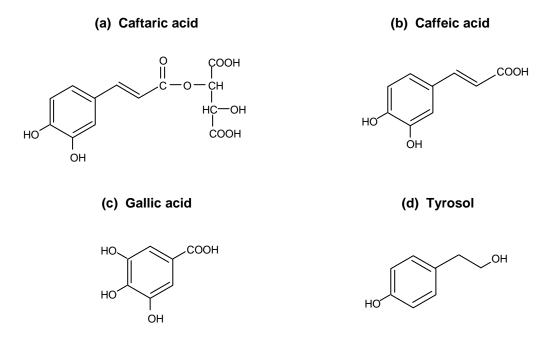
Stilbenes such as resveratrol are another minor phenolic component of white wines. Stilbenes are derived principally from grape skins (Jeandet et al. 1991, Jeandet et al. 1995), where they occur mainly in glycosylated forms (Waterhouse 2002, Sun et al. 2006).

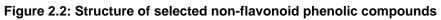
#### 2.1.5 Hydrolysable tannins

Hydrolysable tannins are ester linked oligomers of gallic acid (gallotannins) or ellagic acid (ellagitannins) with glucose or other sugars. They are not present in grapes and are only present in wine treated with oak or oenological tannins (Waterhouse 2002, Ribéreau-Gayon et al. 2006a). The two main ellagitannin isomers in oak used for cooperage are vescalagin and castalagin (Vivas and Glories 1996, Ribéreau-Gayon et al. 2006a).

#### 2.1.6 Volatile phenols

Volatile phenols occur at very low concentrations in wine. They can be formed from hydroxycinnamic acids in reactions catalysed by enzymes from yeast and bacteria, or may be extracted during oak contact (Rentzsch et al. 2009).





## 2.1.2 Flavonoid phenolic compounds

#### 2.1.2.1 Flavan-3-ols and proanthocyanidins

The monomeric flavan-3-ols together with their oligomeric and polymeric forms are the most abundant flavonoid phenolic compounds in grapes and wine (Waterhouse 2002). The major monomeric flavan-3-ols in grapes and wine include (2R, 3S) (+)-catechin (Figure 2.3a) and (2R, 3R) (-)-epicatechin (Figure 2.3b) as well its gallic acid ester, (-)-epicatechin-3-O-gallate (Su and Singleton 1969, Waterhouse 2002, Kennedy et al. 2006). Flavan-3-ols with a tri-hydroxylated B-ring have also been observed in grapes and wine (Monagas et al. 2005).

Proanthocyanidins (also known as condensed tannins, Figure 2.3c) are polymers consisting of multiple monomeric flavan-3-ols. The group name is based on the fact that anthocyanidins are liberated under heated acidic conditions due to cleavage of interflavanic bonds (Porter et al. 1986). Proanthocyanidins are also often described with reference to the specific anthocyanidins liberated, i.e. as procyanidins and prodelphinidins. Proanthocyanidins occur in the seeds, skins and stems of grapes but are either present in very low levels or are non-existent in the pulp. They are qualitatively the same in red and white grape varieties (Souquet et al. 2000b). Seeds contain procyanidins made up of (+)-catechin, (-)-epicatechin and (-)-epicatechin-3-O-gallate (Prieur et al. 1994, Souquet et al. 2000b), while stems and to an even greater extent skins also contain prodelphinidins consisting of Bring trihydroxylated units of (-)-epigallocatechin with trace amounts of (+)-gallocatechin and (-)epigallocatechin-3-O-gallate (Souquet et al. 2000b). Seed proanthocyanidins have a greater degree of gallic acid esterification than those in skins and stems, while skin proanthocyanidins are larger than those in stems and seeds (Souquet et al. 2000b). Seed flavan-3-ols and proanthocyanidins are principally located in the testa of the seeds (Thorngate and Singleton 1994). Skin proanthocyanidins can be found both free in the skin cell vacuoles or bound to proteins on the internal face of the tonoplast or cell wall polysaccharides (Amrani Joutei et al. 1994). The skin proanthocyanidins associated with the cell wall are larger than those in the internal cell fraction (Gagne et al. 2006).

#### 2.1.2.2 Flavonols

Flavonols, like quercetin (Figure 2.3d), are typically found in white wines at low levels (Makris et al. 2006). They exist in grapes as their glycosides (Adams 2006, Makris et al. 2006) in the vacuoles of the epidermal tissue (Monagas et al. 2005) and in grape stems (Souquet et al. 2000a).

#### 2.1.2.3 Flavanonols

Flavanonols (dihydroflavonols) are another minor phenolic component of white wines (Singleton and Trousdale 1983). The flavanonol glycosides, astilbin and engeletin have been found in white grape skins (Singleton and Trousdale 1983, Trousdale and Singleton 1983) and stems (Souquet et al. 2000a).

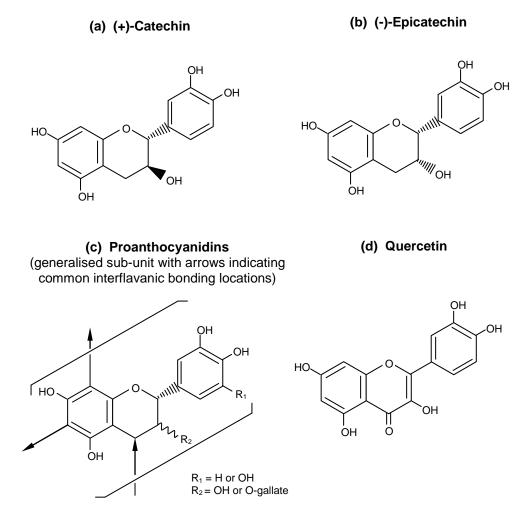


Figure 2.3: Structure of selected flavonoid phenolic compounds

# 2.2 Grape structure and expression related composition

The structure of the grape is presented in Figure 2.4. Simply speaking, grapes consist of skin, pulp/flesh and seeds. Berry component mass fractions vary greatly between varieties, but typically skins comprise approximately 10%, seeds approximately 5% with the pulp making up the balance.

Grape skins consist of the hypodermis and the outer epidermis which is covered by cuticle and wax (Considine and Knox 1979). The epidermis consists of one or two layers of tangentially elongated cells while the hypodermis consist of a variable number of cell layers, the cell-size increasing towards the pulp side (Jona and Foa 1979, Ribéreau-Gayon et al. 2006b).

Grape seeds comprise an embryo and endosperm inside the testa (the seed coat). The testa is commonly further divided into a soft outer layer covered by a cuticle, and an inner layer of hard lignified brown cells (Thorngate and Singleton 1994).

Skin cells are small with thick cell walls, while pulp cells have thin cell walls and are much larger with the vacuole occupying most of the cell volume (Hamatschek 1991, Hamatschek et al. 1995). Rankine (2004) reports that the volume of pulp cells is in the order of 500 times that of the hypodermal cells. Pulp cells are reportedly smaller closer to the grape skin (Coombe and Iland 2004).

A general understanding of the relative size and strength of the cells of the grape is of great practical importance in wine production. The most important inference to be drawn is that as a result of their relative weakness, the pulp cells will tend to yield their contents prior to the skin cells. In describing expression in the literature it has been common to further divide the pulp into three concentric mechanical zones (Ventre 1929, Benvegnin et al. 1951, Amerine and Joslyn 1951, Bonnet 1984, Chabas 1989, Bucher-Vaslin 2007):

- 1. Central zone: Pulp surrounding the seeds.
- 2. Peripheral zone: Pulp near the skins.
- 3. Intermediate zone: Pulp between the central and peripheral zones.

Juice will tend to be first released from the intermediate zone then from the central and peripheral zones of the berry, and then from the skins. In some reports, the skin is defined as part of the peripheral zone (Benvegnin et al. 1951, Bucher-Vaslin 2007), perhaps reflecting the unclear boundary between skin and pulp and the practical reality that on berry rupture the peripheral pulp will tend to remain attached to the skin and when expressing the juice from this peripheral pulp, the skins may contribute significant solutes to the expressed juice.

As already described in section 2.1, many phenolic compounds are grape-derived and these are found at much higher concentrations in the grape skin than in the pulp (Coombe 1987, Coombe and Iland 2004). However other important wine components are approximately co-located with phenolic compounds and consequently will be released in parallel with those phenolic compounds. The grape skin contains much higher levels of potassium and inorganic ions (Coombe 1987, Coombe and Iland 2004) and often higher levels of potentially beneficial aroma compounds and precursors (Gunata et al. 1985) than the pulp. Furthermore, acidity decreases from the centre towards the periphery of the grape (Ventre 1929, Benvegnin et al. 1951, Ribéreau-Gayon et al. 1975).

NOTE: This figure is included on page 10 of the print copy of the thesis held in the University of Adelaide Library.

# Figure 2.4: Longitudinal section of one side of a grape berry showing its constituent tissues (From: Coombe and Iland 2004)

# 2.3 White wine taste and mouthfeel

Phenolic compounds are principally associated with bitterness and astringency. Bitterness is a taste while astringency is a drying and puckering tactile sensation (Bate-Smith 1954) and both are generally seen as undesirable characteristics in white wines. The term 'phenolic' is sometimes even used, i mprecisely, as a pejorative t asting t erm, to d escribe bitter a nd as tringent white wines (Robinson and Harding 2006).

In considering the sensory influence of specific non-flavonoid and flavonoid phenolic compounds, it is important to consider that while alone a compound may have no significant influence on bitterness or astringency, there may be additive or synergistic effects with other phenolic or non-phenolic white wine components. For example, sugar content (Lea and A rnold 1978), ethanol (Fischer and Noble 1994) and acidity (Guinard et al. 1986) can alter perceived bitterness and/or astringency.

## 2.3.1 Non-flavonoid phenolic compounds

It has not been proven that any non-flavonoid phenolics individually contribute significant bitterness or astringency to white wines at the ranges of concentrations they typically occur at.

Sensory analysis of hydroxycinnamates, the principal white wine non-flavonoid phenolics, has demonstrated that when prepared in water, these compounds can elicit bitterness and astringency at concentrations comparable to those in white wines (Ong and Nagel 1978a, Okamura and Watanabe 1981). However, in a later study involving back-addition of hydroxycinnamates to a commercial dry Chardonnay wine with no base bitterness or astringency, no significant bitterness or astringency was observed at additions comparable with the highest levels observed in white wines (Vérette et al. 1988).

The hydroxybenzoic acid, gallic acid, can elicit bitterness and to a lesser extent, astringency (Robichaud and Noble 1990). Dadic and Balleau (1974) found a flavour threshold of greater than 50 mg/L (highest level in their study) in beer, while in a later study where gallic acid was back-added to a white wine, perceived bitterness and astringency did not differ significantly between concentrations of 10, 250, 500 and 1000 mg/L (Robichaud and Noble 1990). Given that hydroxybenzoic acids are reported to typically occur at concentrations in the order of 10 mg/L in white wine (Waterhouse 2002), it seems unlikely that they would significantly contribute to bitterness or astringency.

Tyrosol is also bitter (Singleton and Noble 1976) but Singleton (1992) suggests that it is not very flavourful at the levels typical in white wines.

Contrary to the widespread opinion in the wine industry that non-volatile phenolic compounds derived from oak can impart bitterness and astringency, Pocock et al. (1994) found that hydrolysable tannins contributed by oak were near to or below sensory detection limits. This resulted in their conclusion that volatile oak-derived constituents are the species responsible for the sensory impression of oak treatment.

## 2.3.2 Flavonoid phenolic compounds

Bitterness and astringency in white wine are principally associated with flavan-3-ols and proanthocyanidins. The threshold concentrations in water for proanthocyanidin trimers and larger polymers is reportedly in the order of 4 mg/L in water and not much higher in white wines (Singleton 1992). Monomeric flavan-3-ols are primarily bitter, but with increasing size, astringency increases more so than bitterness (Lea and Arnold 1978, Arnold et al. 1980, Robichaud and Noble 1990, Peleg et al. 1999, Vidal et al 2003). Stereochemistry of monomeric flavan-3-ols (Thorngate and Noble 1995) and proanthocyanidin sub-units and the location of interflavanic bonds (Peleg et al. 1999) also influence bitterness and astringency. Gallic acid esterification and B-ring tri-hydroxylation may enhance astringency (Brossaud et al. 2001).

Flavonols, like quercetin, can also elicit bitterness and astringency (Dadic and Belleau 1974). However, they occur at low concentrations in white wine, possibly too low to significantly contribute to bitterness or astringency.

### 2.3.3 Complex phenolic oxidation adducts

During white grape and juice processing, enzymatically catalysed oxidation of caftaric and coutaric acids to caftaric acid o-quinone in grape must can ultimately lead to the formation of a range of complex phenolic adducts involving both flavonoid and non-flavonoid phenolic compounds (Cheynier et al. 1998). Larger compounds may precipitate and be removed from solution. Conceivably, there could be a variety of intermediate compounds that remain in solution. The concentrations these compounds occur at and their sensory influence does not appear to be well understood.

While intentional must oxidation (hyperoxidation) has been widely demonstrated to reduce overall phenolic concentration and many key flavonoid and non-flavonoid compounds (e.g. Singleton et al. 1980, Cheynier et al. 1989, Ricardo da Silva et al. 1993), interestingly, bitterness was significantly increased by must oxidation for two of six wines treated in one study by Nagel and Graber (1988). In trying to explain their observation, Nagel and Graber (1988) cited a study by Dadic and Belleau (1974) investigating the influence of phenolics on beer flavour. Dadic and Belleau (1974) observed that bitterness and astringency were generally more pronounced with a range of oxidised phenolic compounds than with the initial phenolic compounds. Potentially, oxidation may influence the perceived bitterness and astringency of phenolic compounds in white wine that aren't precipitated from solution.

## 2.4 White wine stability

Pinking and browning are defects in white wine related to wine oxidation. Pinking is the development of a slight pink colour, generally not altering taste or aroma, whereas browning is related to more severe oxidation and undesirable changes in taste and aroma can result (Lamuela-Raventós et al. 2001). Pinking and browning are two distinct phenomena and browning may occur in the absence of pinking (Simpson 1977). The chemical that is pink is still unknown (Lamuela-Raventós et al. 2001).

A white wine's susceptibility to pinking (Simpson 1977, Lamuela-Raventós et al. 2001) and browning (Rossi and Singleton 1966, Singleton and Kramling 1976, Simpson 1982, Schneider 1995) is generally increased with higher phenolic levels.

Another phenolic-related white wine instability is the development of a yellow haze or sediment, through the hydrolysis of flavonol glycosides to their less soluble aglycones (Somers and Ziemelis 1985a).

# 2.5 Conclusions

A diverse range of phenolic compounds exist in white wine with many being principally derived from the grape. The distribution of specific phenolic compounds between the different tissues of the grape and the cellular structure of the grape have important implications for white wine production.

Unbalanced bitterness and astringency as well as colour and colloidal instability associated with phenolic compounds are undesirable characteristics of white wines, which ultimately reduce value. While some phenolic compounds can be removed by fining, fining agents can be both expensive and may detrimentally strip flavour (McLean 2006). Management of juice phenolic levels is therefore of great interest in white wine production.

# CHAPTER 3: EXTRACTION OF PHENOLIC COMPOUNDS DURING POMACE CONTACT

A general principle of white winemaking is to try to limit contact between juice and grape skins, seeds and stems (Ribéreau-Gayon et al. 2006b). Concerns of excessive phenolic extraction are a major reason for this practice.

The majority of white grapes in Australia are machine harvested. With machine harvesting, some pomace contact is unavoidable. In contrast to hand-picking where grape clusters can be delivered to the winery relatively undamaged, machine harvesting partially juices grapes as the berries are shaken from the vine, resulting in pomace contact prior to winery processing.

To maintain quality, wine companies often harvest when it is cooler (typically at night), and minimise times between harvesting and processing at the winery. For example, one wine company will not harvest commercial grade white grapes if the minimum night time temperature exceeds 20 °C or premium white grapes if it exceeds 15 °C (McLean 2006). Another company, to the author's knowledge, contracts other wineries closer to their more distant vineyards to process grapes because of time-related quality concerns, when it would actually be more cost-effective for them to transport their grapes over longer distances to their own processing facilities.

While it has been widely demonstrated that pre-fermentative grape pomace contact can result in increased phenolic levels in juice and wine (Singleton et al. 1980, Ramey et al. 1986, Merida et al. 1991), the topic of phenolic extraction during pomace contact is of ongoing interest to the wine industry as a result of costs and restrictions on practices of the nature described above.

The aim of the work presented in this chapter was to further investigate phenolic extraction and associated phenomena that may occur after machine harvesting, at a controlled laboratory scale with different levels of berry breakage, time, temperature, and sulfur dioxide addition.

# 3.1 Materials and methods

## 3.1.1 Grape picking

Chardonnay grapes from Barossa Valley and Langhorne Creek and Riesling grapes from Eden Valley (South Australia) were handpicked into 50 L polypropylene (PP) crates at commercial maturity during the 2008 vintage. Each grape lot was subsequently distributed representatively into 35 L polystyrene foam containers and refrigerated at 0-4 °C until use.

## 3.1.2 General characterisation

#### 3.1.2.1 Grape characterisation

Five bunches were randomly selected from each of the three grape lots. Each bunch was weighed and the berries were manually removed from the stems. Individual berries from each bunch were weighed and their diameter measured with a micrometer (measured perpendicular to the axis running between the pedicel and stylar remnant). For each bunch, seeds and skins were separated from the pulp and weighed.

#### 3.1.2.2 Juice characterisation

Twenty bunch samples of each lot were destemmed by hand. Three 250 g portions of berries were placed in linear low density polyethylene (LLDPE) resealable bags and the berries crushed manually. Mash was poured over a 316 stainless steel (316 SS) slotted (20 mm × 2 mm slots) draining plate (Figure 3.1a) from which juice was directed by a PP funnel into 50 mL PP centrifuge tubes. Juice samples corresponding with yields of 0-200 L/tonne and 200-400 L/tonne were collected. The 0-200 L/tonne fraction drained easily while mild pressure applied by a simple 316 SS ram (Figure 3.1b) was employed for collection of the 200-400 L/tonne fraction.

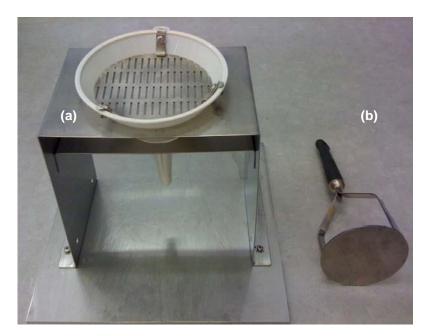


Figure 3.1: (a) Draining plate and (b) simple ram

Juice samples were centrifuged (5 minutes,  $3,100 \times g$ , 4 °C) and the supernatant decanted into fresh 50 mL PP tubes. Juice density was determined using a hand-held refractometer (PAL-1; Atago, Japan), and juice pH and conductivity were determined using a laboratory pH probe and conductivity probe (k = 1.0) with automatic temperature compensation (TPS, Australia).

## 3.1.3 Grape component phenolic characterisation

#### 3.1.3.1 Solvent extraction

Solvent extraction was performed (in triplicate) for grape components from each lot. The solvent extraction technique was based generally on a method used for determination of anthocyanins from red grapes (Cooperative Research Centre for Viticulture 2006). Approximately 4 g of separated grape components (skins, seeds, stems or entire grapes) were added to 20.8 mL of a 2 g/L aqueous potassium metabisulfite (Sigma-Aldrich, USA) solution in screw capped 50 mL PP tubes. The mixture was milled with an Ultra-Turrax T25D with S25N-18G element (IKA, Germany) for 2 minutes at 15,000 rpm. 20 mL of ethanol (Univar, Australia) was added and the tubes were agitated lying sideways on an orbital shaker table (1 hour, 120 rpm). Samples were clarified (5 minutes, 3,100 ×g, 4 °C) and the supernatant distributed into 10 mL PP centrifuge tubes before storage at -20 °C for later phenolic analysis.

#### 3.1.3.2 Phenolic analysis

Phenolic analysis was performed by ultraviolet (UV) spectroscopy, largely using principles described by Somers and Ziemelis (1985b).

Extracts were warmed (1 minute, 50 °C) and then diluted to 10% (v/v) in 50% (v/v) aqueous ethanol.

5 mL aliquots of the diluted extracts were added to 10 mL PP centrifuge tubes containing 0.5 g of polyvinylpolypyrrolidone (PVPP; Polyclar VT; International Specialist products, USA). The mixture was vortex mixed for 1 minute, before being placed horizontal on a bench for a minimum of 30 minutes. The PVPP treated diluted extracts were clarified by centrifugation (5 minutes, 3,100 ×g, 4 °C), and a supernatant sub-sample collected.

UV spectra of both the diluted extracts and PVPP treated diluted extracts were determined in 1 mm path length quartz cells (Starna, UK) against a reverse osmosis (RO) water reference (Pharmaspec UV-1700; Shimadzu, Japan) after clarification by micro-centrifugation (15 minutes, 15,000 ×g). Spectral results were normalised to a 1 cm path length and were corrected for dilution of components during solvent extraction and for the dilution of the extracts prior to analysis. Mass fractions determined during grape characterisation were employed to convert component concentrations into berry concentrations.

Phenolic standards were prepared for spectral comparison. 100 mg/L solutions of caffeic acid, gallic acid, catechin and quercetin (Sigma-Aldrich) were prepared in 50% (v/v) aqueous ethanol, with 100 mg/L potassium metabisulfite. UV spectra were acquired, following the same procedure as that for the extracts and the results were again normalised to a 1 cm path length.

## 3.1.4 Pomace contact studies

#### 3.1.4.1 Complete berry crushing

Concurrent experiments were performed with 54 portions of Barossa Valley Chardonnay or Eden Valley Riesling grapes, at three different temperatures (range: 10-24 °C) using three incubators (IC-140R; Labec, Australia), three levels of sulfur dioxide application (0, 50 or 100 mg/kg SO<sub>2</sub> by treatment with 0, 100 or 200 mg/kg potassium metabisulfite from a 50 g/L aqueous potassium metabisulfite solution), and six time points (0-12 hours). The entire Barossa Valley Chardonnay experiment was replicated on another day.

Grape clusters were preconditioned in incubators overnight, at temperatures several degrees below the desired treatment temperature, such that after necessary processing at ambient conditions, batches of grapes would be at approximately the desired treatment temperature. Grapes for each treatment temperature were destemmed by hand and mixed. 250 g grape portions were distributed into LLDPE resealable bags, which were then crushed manually and poured into 250 mL screw-capped PP vessels. The required additions of potassium metabisulfite were made and each vessel was agitated by inverting twice. Vessels were placed in the appropriate incubator. For each incubator, a temperature control sample (grapes prepared in the same manner but with a thermocouple inserted into the mash through a perforation in the lid) was used to log the temperature for use in later regression analysis. After the relevant treatment time, vessels were removed from their incubator and agitated by inverting twice. For each vessel, juice samples corresponding with yields of 0-200 L/tonne and 200-400 L/tonne were collected using the draining plate and simple ram described in Section 3.1.2.2.

Samples were centrifuged (5 minutes, 3,100 ×g, 4  $^{\circ}$ C) and the supernatant decanted into fresh 50 mL PP tubes where the juice was treated with 1 g/L potassium metabisulfite (from a 50 g/L aqueous potassium metabisulfite solution) for sample preservation. The juice was distributed into 15 mL PP tubes before frozen storage (-20  $^{\circ}$ C) for later phenolic analysis.

#### 3.1.4.2 Partial berry crushing

Experiments were performed simultaneously with 54 portions of Langhorne Creek Chardonnay or Eden Valley Riesling grapes, at three different temperatures (Range: 12-23 °C), six time points (0-12 hours), and three levels of berry breakage (20%, 50%, 100%). Different levels of berry breakage were achieved by weighing separate portions into LLDPE resealable bags, crushing the relevant portion, and mixing the crushed and uncrushed portions together into the 250 mL treatment vessel. All samples were treated with 50 mg/kg SO<sub>2</sub>. The entire Langhorne Creek Chardonnay experiment was replicated on another day.

Experimental techniques were similar to those described in 3.1.4.1. The simple ram was required for expression of both yield fractions from the partially crushed treatments.

#### 3.1.4.3 Phenolic analysis

Phenolic content was determined by UV spectroscopy, predominantly using principles described by Somers and Ziemelis (1985b).

Samples were thawed in a water bath (4 minutes, 50 °C) and clarified by centrifugation (20 minutes,  $17,500 \times g, 4$  °C) before decanting into 11.5 mL polystyrene tubes and further centrifugation (5 minutes,  $2,500 \times g, 4$  °C).

UV spectra were collected with a Multispec wine analyser (Microdom, France), incorporating an autosampler and a UV transmission flow cell (UV path length: 0.2 mm). Results were normalised to a 1 cm path length.

A small number of samples were tested for specific phenolic compound concentrations by a third party using a proprietary reversed phase high performance liquid chromatography (HPLC) method.

Selected samples treated with or without 100 g/L PVPP in a similar manner to that described for solvent extracts were analysed by UV spectroscopy. To further investigate the efficacy of PVPP treatment, several unrelated white grape juice samples were tested by HPLC, with or without 100 g/L PVPP treatment.

#### 3.1.4.4 Regression analysis

Regression analysis with spectral absorbance at 280 nm or 320 nm as the dependent variable was performed in Microsoft Excel using an Ordinary Least Squares Regression Add-in (Stock and Watson 2003, Introduction to Econometrics, Pearson Education, Inc., Version 1.0). Heteroskedasticity-robust standard errors were employed. A general quadratic model was fitted. A dummy variable was included where experiments had been repeated on a second day. Standardised regression coefficients were obtained by repeating regressions after standardisation of observations. The standardised observations for multiplicative terms were calculated by multiplying the already standardised linear terms (Friedrich 1982, Mason et al. 1989, Aiken and West 1991). Standardised coefficients show the mean response in standard deviation units of the dependent variable for a one standard deviation change in an explanatory variable, holding other model variables constant (Bollen 1989).

# 3.2 Results and discussion

#### 3.2.1 General grape and juice characterisation

Berry component mass fractions, berry diameter and mass analyses, and general juice analyses are reported in Tables 3.1, 3.2 and 3.3, respectively. Langhorne Creek Chardonnay grapes were larger and weighed more than the Barossa Valley Chardonnay and the Eden Valley Riesling grapes and correspondingly (assuming similar skin thicknesses) had the lowest skin content. The 200-400 L/tonne juice fraction in each grape lot featured higher conductivity and pH than the 0-200 L/tonne fraction, consistent with increased contribution of juice from nearer to and/or from the skin.

Berry Component <sup>a</sup>	Barossa Valley	Langhorne Creek	Eden Valley Berry	
	Chardonnay	Chardonnay	Riesling	
Seeds	5.3%	4.1%	4.6%	
	(8%)	(9%)	(12%)	
Skins	17.6%	9.7%	12.3%	
	(12%)	(9%)	(7%)	
Pulp	77.1%	86.2%	83.1%	
	(3%)	(1%)	(1%)	

#### Table 3.1: Berry component mass fractions

<sup>a</sup> Average of five bunches. Coefficient of variation between bunches reported in parentheses.

Berry property <sup>a</sup>	Barossa Valley	Langhorne Creek	Eden Valley Berry	
	Chardonnay	Chardonnay	Riesling	
Diameter <sup>b</sup> (mm)	11.0	12.6	11.7	
	(3%)	(4%)	(3%)	
Mass <sup>c</sup> (g)	0.87	1.32	1.03	
	(9%)	(9%)	(9%)	

#### Table 3.2: Berry diameter and mass

<sup>a</sup> Average of five bunches. Coefficient of variation between bunches reported in parentheses. <sup>b</sup> Surface area mass weighted average diameter for each bunch.

<sup>c</sup>Mass weighted average berry mass for each bunch.

Analyte <sup>ª</sup>	Barossa Valley Chardonnay		Langhorne Creek Chardonnay		Eden Valley Berry Riesling	
	0-200 L/tonne	200-400 L/tonne	0-200 L/tonne	200-400 L/tonne	0-200 L/tonne	200-400 L/tonne
Density	21.0	21.1	20.6	20.7	21.2	21.3
(°Brix)	(1%)	(1%)	(0%)	(0%)	(1%)	(1%)
рН	3.49	3.56	3.19	3.22	3.47	3.54
	(1%)	(0%)	(0%)	(0%)	(2%)	(1%)
Conductivity	3.10	3.23	2.62	2.71	2.55	2.66
(mS/cm)	(1%)	(1%)	(1%)	(0%)	(2%)	(4%)

Table 3.	3: General	iuice ana	alvses
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<sup>a</sup> Average of three replicates. Coefficient of variation between replicates reported in parentheses.

#### 3.2.2 Component phenolic characterisation

Spectra of grape component extracts for each of the grape lots are presented in Figure 3.2 (spectra of all replicates are included in Appendix A). Spectra of skin and seed extracts are also reported on a berry concentration basis in Figure 3.3. For comparison, spectra of caffeic acid (a hydroxycinnamic acid), gallic acid (a hydroxybenzoic acid), catechin (a flavan-3-ol), and quercetin (a flavonol) are presented in Figure 3.4.

All component extracts featured a principal peak at approximately 280 nm. This peak was dominant for seed extracts, while the skin extracts featured another peak at approximately 320 nm that tailed into the visible region. Stem and entire grape extracts also featured absorbance in this region; however, there were no clear peaks. The different grape lots featured similar component spectra. Langhorne Creek Chardonnay skin extracts featured higher absorbance at 280 nm on a skin concentration basis than the other grape lots (Figure 3.2b), but had values in between the other grape lots on a berry concentration basis (Figure 3.3b).

PVPP treatment significantly reduced UV absorbance of all component extracts. PVPP is used for phenolic removal in wine (generally at PVPP concentrations below 0.5 g/L) and it has been used previously at high concentrations to selectively and extensively remove phenolic compounds from juice and wine (Somers and Ziemelis 1985b). The considerable reduction in extract UV absorbance after 100 g/L PVPP treatment implies that much of the original extract absorbance was related to phenolic compounds.

Soluble material from PVPP can contribute appreciable absorbance in the far UV, however it was found in preliminary experiments involving PVPP treatment of 50% (v/v) aqueous ethanol that this did not meaningfully influence conclusions from extract analysis. The contribution of the soluble contaminants was found to be minor at 320 nm, but increased with decreasing wavelength,

contributing approximately 4 au at 280 nm and 26 au at 250 nm with respect to the PVPP treated extracts in Figure 3.2.

The phenolic standards; caffeic acid, gallic acid, catechin and quercetin, had spectral maxima at approximately 325, 273, 280 and 374 nm, respectively. Comparisons between extract spectra and phenolic standard spectra suggest qualitative phenolic composition consistent with literature reviewed in Chapter 2. For example, the seed extracts contain flavan-3-ols and proanthocyanidins and possibly gallic acid released from their galloylated forms, whereas the skins also contain hydroxycinnamates responsible for the peak at approximately 320 nm and possibly flavonols, given the tailing absorbance past 385 nm.

Quantitative comparison of the absorbance at 280 nm of different grape component extracts are consistent with previous reports that phenolic compounds are at their highest levels in the seeds, then stems then skins (Cantarelli and Peri 1964, Kantz and Singleton 1990).

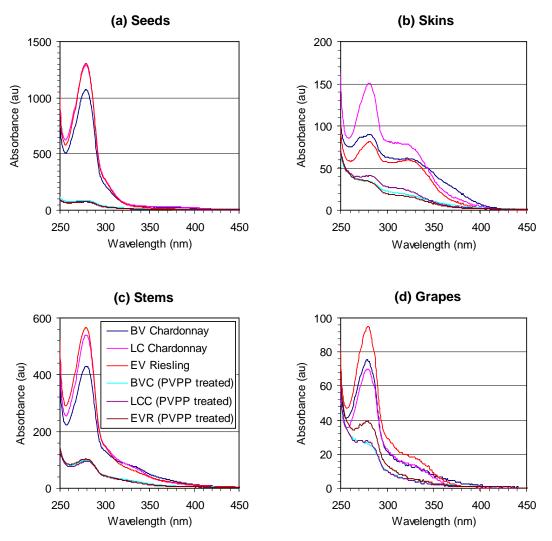


Figure 3.2: Grape component extract spectra (component concentration, with and without PVPP treatment)

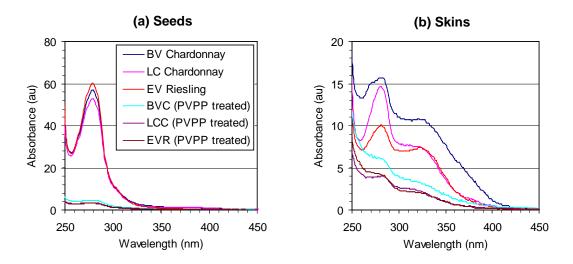


Figure 3.3: Seed and skin extract spectra (berry concentration, with and without PVPP treatment)

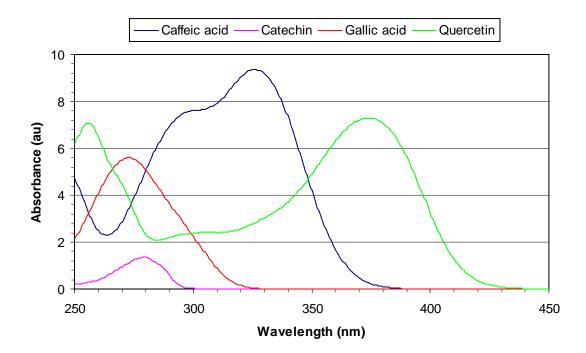


Figure 3.4: Spectra of phenolic standards (100 mg/L)

#### 3.2.3 Berry component and juice phenolic concentration

Juice spectra from pomace contact studies for each of the three grape lots are presented in Figure 3.5 for either no pomace contact or after approximately 12 hours pomace contact. The spectra for juice not subjected to pomace contact, show principal absorbance bands around 280 and 320 nm, consistent with hydroxycinnamates being a key phenolic constituent of the pulp. UV absorbance of both yield fractions increases with pomace contact. Increased absorbance around 320 nm indicates

probable increased levels of hydroxycinnamates. Comparison with seed and skin extract spectra (Figure 3.3) suggest that some, if not all, of the increase in UV absorbance is derived from the skins. Increased levels of quercetin glycosides determined in HPLC analysis (Table 3.4) are also indicative of skin extraction. The relative ease of extraction of phenolic compounds from skins as opposed to seeds during pomace contact has been asserted previously (Du Plessis and De Wet 1968, Ricardo da Silva et al. 1993). Comparison between the magnitudes of UV absorbance of skin extracts (Figure 3.2b) and juice samples (Figure 3.5) illustrates the much higher levels of phenolics in the skins than in the grape pulp, consistent with Singleton and Esau (1969).

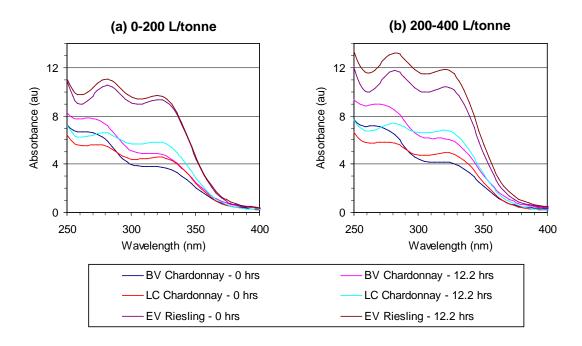


Figure 3.5: Influence of pomace contact (100% crushed, 50 mg/kg SO<sub>2</sub>, approximately 12 °C) on spectra for different lots

	1 div	••••		marysis				•			
			Phenolic compounds (mg/L) <sup>a</sup>								
Sample		Caftaric acid	Coutaric acid	GRP	Caffeic acid	Gallic acid	Catechin	Tannin <sup>b</sup>	Quercetin glycosides	Quercetin	Astilbin
Barossa Valle	Barossa Valley Chardonnay (100% crushed, 50 mg/kg SO <sub>2</sub> , approximately 16 °C)										
0-200	0 hrs	10.0	1.2	9.2	0.0	0.0	0.0	14.7	0.8	0.0	0.3
L/tonne	12.2 hrs	13.0	2.5	8.0	0.0	0.0	0.0	16.7	3.9	0.0	1.0
200-400	0 hrs	10.1	1.2	8.2	0.0	0.0	0.0	16.2	0.9	0.0	0.3
L/tonne	12.2 hrs	14.0	3.0	8.5	0.0	0.0	0.0	16.4	6.4	0.0	1.2
Langhorne C	reek Chardor	nay (10	0% cru	shed, 50	mg/kg	SO <sub>2</sub> , ap	proxim	ately 12	°C)		
0-200	0 hrs	17.1	2.0	10.3	0.0	0.0	0.0	14.0	0.5	0.0	0.2
L/tonne	12.2 hrs	25.2	5.1	8.8	0.0	0.0	0.0	14.6	2.3	0.0	1.1
200-400	0 hrs	20.3	2.2	9.4	0.0	0.0	0.0	n.d. <sup>c</sup>	0.7	0.0	0.2
L/tonne	12.2 hrs	30.5	6.9	9.3	0.0	0.0	0.0	n.d. <sup>c</sup>	3.6	0.0	1.4
Eden Valley F	Eden Valley Riesling (100% crushed, 50 mg/kg SO <sub>2</sub> , approximately 12 °C)										
0-200	0 hrs	55.0	4.5	4.4	0.0	0.0	0.0	20.7	1.9	0.0	0.2
L/tonne	12.2 hrs	47.6	5.5	4.1	0.0	0.0	0.0	15.8	6.6	0.0	0.2
200-400	0 hrs	57.4	5.1	4.1	0.0	0.0	0.0	20.7	2.6	0.0	0.3
L/tonne	12.2 hrs	58.3	8.4	5.4	0.0	0.0	0.0	20.3	13.0	0.0	0.4

Table 3.4: HPLC analysis of selected juice samples

<sup>a</sup> Caftaric acid, coutaric acid, GRP (grape reaction product) concentrations are expressed in caffeic acid equivalents. Tannin is expressed in catechin equivalents. Quercetin glycosides are expressed in quercetin equivalents. Concentrations were measured and reported by a third party using a proprietary HPLC method.

<sup>b</sup> Tannin refers to a complex peak eluting towards the end of the HPLC method.

<sup>c</sup> n.d.: not determined.

### 3.2.4 Fundamental processes during pomace contact

The overall pomace contact process consists of three stages:

- 1. Mechanical damage (e.g. during machine harvesting, manual crushing in these experiments).
- 2. Pomace contact time.
- 3. Final expression.

In the initial mechanical damage, juice is expelled from some grape cells. Which tissue cells rupture, and thus the quantity and quality of juice released, is dependent on the condition of the grapes and the mode of mechanical damage. The expelled juice forms the initial bulk liquid phase for the subsequent pomace contact time. The general scheme of mechanical damage with the crushing performed in these experiments is illustrated in Figure 3.6.

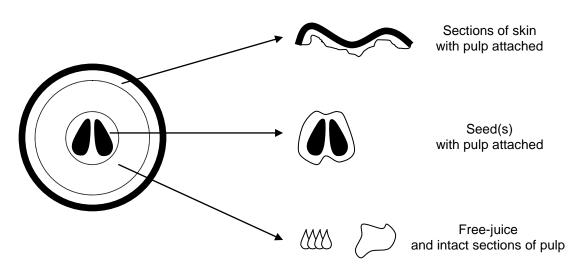


Figure 3.6: Influence of crushing on constituents for pomace contact

During the pomace contact time, a number of processes occur simultaneously. There is likely to be osmosis of water into some intact cells as the bulk juice will tend to have a lower solute concentration than many of the remaining smaller intact cells from or nearer to the skin. There will also be diffusion of compounds, including phenolics, towards the bulk juice, where they are likely to be present at lower concentrations. For phenolic compounds that are located in the vacuole of a cell (e.g. in a skin cell) to appear in the bulk juice, they need to pass out of the vacuole to the cell, out of the cell, through any other material attached to that cell (e.g. pulp cells attached to the skin cell) and then from the external surface of that material to the bulk juice. Phenolic compounds that are bound to solids (e.g. bound to a cell wall) must also dissolve in a contacting fluid to allow transport. The cellular structure of a section of skin with pulp attached submerged in the bulk juice is represented graphically in Figure 3.7. For a phenolic compound in cell A to be transported to the bulk juice it needs to pass through attached pulp cells B and C.

The diffusion would be expected to be heavily dependent on the condition of the pomace from the initial mechanical damage. For example, smaller grape sections consisting of fewer cells means reduced distances and obstructions for phenolics to diffuse through. The destruction of cell walls and adhering membranes is particularly significant as they can provide major resistances to diffusion (Aguilera 2003). Enzymes released during the initial mechanical damage will also have an important influence on phenolic concentration. Endogenous juice pectin-splitting enzymes will tend to degrade the cell walls that provide the cells with their strength. This raises the potential for increased release of juice from smaller and stronger cells, which are also likely those to contain higher levels of phenolic compounds (e.g. skin cells). Oxidation enzymes can catalyse the conversion of caftaric acid and coutaric acid to caftaric acid o-quinone and through a series of complex reactions this can lead to the precipitation of these and other phenolic compounds from solution (Cheynier et al. 1998).

The final expression involves the collection of the juice samples. Draining will collect much of the bulk juice, while further mechanical action will release additional more tightly bound juice (perhaps juice

from cells B and C in Figure 3.7 for example). The juice released will be dependent on the pomace condition at this point in time. For example, the enzymatic weakening of cells during the pomace contact time may mean an increased release of juice from skin cells during the final expression.

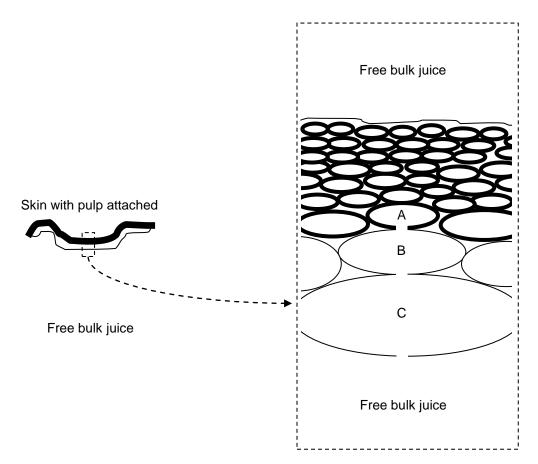


Figure 3.7: Cellular representation of a section of skin with pulp attached submerged in the already expressed bulk juice

### 3.2.5 Pomace contact (complete berry crushing)

Spectral absorbance at 280 nm ( $A_{280}$ ) and 320 nm ( $A_{320}$ ) for both yield fractions of the Barossa Valley Chardonnay (Replicate 2, see Appendix A for Replicate 1) and Eden Valley Riesling are presented in Figures 3.8 and 3.9, respectively (time points have been linked for clarity; each point is a separate treatment). The corresponding regression analyses are reported in Tables 3.5 and 3.6. Standardised regression coefficients have been included to provide the reader with an informal dimensionless means of gauging the relative influence of each variable across the range of explanatory variable values trialled.

The 200-400 L/tonne juice fraction featured higher absorbance than the 0-200 L/tonne fraction at both 280 nm and 320 nm for all samples, consistent with a greater contribution of phenolics originating in the skins.

Phenolic levels increased with time for both yield fractions of Chardonnay. The 0-200 L/tonne fraction was collected by draining and as such, largely consisted of the bulk juice fraction. As the pomace contact proceeded and the concentration of phenolic compounds in the bulk juice increased the concentration gradient between the phenolic compounds associated more closely with the pomace and the phenolic concentration in the bulk juice would have decreased. This would explain some observed reductions in the net phenolic extraction rate with time (note the statistically significant negative squared time term in the regression analysis). The 200-400 L/tonne fraction principally consisted of juice loosely held by the pomace, which needed some mechanical action for final expression. The phenolic concentration of this fraction initially increased more rapidly but the net extraction rate decreased to a greater extent at longer contact times than with the 0-200 L/tonne fraction.

A more rapid initial increase and then tapering as opposed to a more linear increase in phenolic concentration is to be expected for higher yield fractions. For higher yield fractions the cells from closer and closer to the skin are likely contributing more to the juice fraction. As discussed in section 2.2 and illustrated in Figure 3.7, the skin cells are much smaller than the pulp cells, and the pulp cells themselves are also smaller closer to the skin. The skin cells have much higher concentrations of phenolic compounds than the pulp cells, so there will be a relatively high phenolic concentration gradient between any damaged skin cells and the attached pulp cells. This could facilitate rapid initial diffusion. However, the phenolic content of the damaged skin cells would be quickly depleted because of the much lower volume of the skin cells relative to the nearby pulp cells (For example consider skin cell A and pulp cell B in Figure 3.7) and therefore the phenolic sprogress the more gradual the diffusion rate would likely become (For example between pulp cells B and C and between pulp cell C and the bulk juice in Figure 3.7) as the local concentration gradients would be smaller.

The decrease in net extraction rate for the 200-400 L/tonne fraction could also be related to losses of phenolics from the cells that may contribute some juice to this fraction (pulp cell C for example) to the bulk free juice that supplies the 0-200 L/tonne sample. For both juice fractions, the diminishing rate of increase of phenolic levels could alternatively be related to phenolic oxidation and precipitation. However, the similar patterns for samples treated with different levels of sulfur dioxide, an antioxidant and inhibitor of enzymes that catalyse phenolic oxidation (Ribéreau-Gayon et al. 2006b), suggests that this is not likely to be the principal explanation.

Even without any pomace contact (i.e. 0 hours), juice samples from grapes at the higher temperature of 23-24 °C contained higher levels of phenolics. This is likely related to increased respiration and transpiration of grapes during overnight preconditioning at higher temperatures. Transpiration could act to concentrate solutes like phenolic compounds and it would also be likely to reduce the turgidity of grape cells, and hence their strength. As discussed in section 2.2, wine grape pulp cells are generally quite weak; however skin cells are relatively strong in grapes in good condition. With the

less turgid grape cells, there may have been more damage to skin cells relative to pulp cells during the crushing of the berry (than at cooler preconditioning temperatures), meaning a greater initial contribution of phenolic compounds from skin cells. It should be noted that while this temperature effect may be seen to some extent with actual machine harvesting of grapes at different temperatures, it probably would not be to the same level since the grapes are still connected to the vine, and variations in cell turgidity with differing ambient conditions are likely to be less severe.

The coefficient for the linear combination of time and temperature was negative for both yield fractions of the Chardonnay (only statistically significantly for the higher yield fraction) indicating that although the higher temperature grapes produced more phenolic juice without pomace contact, the net phenolic extraction rate was apparently lower at higher temperatures. The negative influence of temperature on extraction rate may be related to the decreased phenolic concentration gradient as a result of the higher levels of phenolics released initially on crushing. These results differ from Ramey et al. (1986) who observed higher rates of extraction for grapes picked at higher temperatures. The reason for this difference is not entirely clear.

Sulfur dioxide concentration also influenced phenolic levels. The effect was particularly noticeable at 320 nm, where hydroxycinnamates have a spectral maximum. The coefficient of the linear combination of time and sulfur dioxide indicates the influence of sulfur dioxide on the net extraction rate and for the Barossa Valley Chardonnay this was statistically significant only for the 0-200 L/tonne fraction. The time-based significance of sulfur dioxide in the 0-200 L/tonne fraction, but not in the 200-400 L/tonne fraction, may be indicative of the greater importance of sulfur dioxide in limiting oxidation and precipitation of phenolics in the relatively free juice than in the juice still more confined to cellular structures. In addition to the time-based significance of sulfur dioxide there, there was also a nontime-based influence. This can be observed graphically by the downwards offset for samples not treated with sulfur dioxide during pomace contact, particularly at 320 nm. It is also evident from the statistical significance of the coefficients of sulfur dioxide and its square. The statistical significance of the negative coefficient of the square of sulfur dioxide is indicative of a diminishing increase in effect at higher sulfur dioxide concentrations. Again, this is likely related to sulfur dioxide limiting phenolic oxidation and precipitation either during treatment or possibly during sample clarification prior to the large preservative addition of sulfur dioxide being made. Generally the results are consistent with previous work reporting higher phenolic levels in white juices and wines subject to pomace contact in the presence of higher levels of sulfur dioxide (Singleton et al. 1980).

Inspection of Figures 3.8 and 3.9 shows major differences between the experiments with the Chardonnay grapes compared to those with the Riesling grapes. Riesling juice phenolic results were relatively more scattered and the Riesling juice featured considerably higher absorbance at both 280 nm and 320 nm than similarly treated Chardonnay juice. This is not unexpected as HPLC analyses presented in Table 3.4 show higher concentrations of caftaric acid in the Riesling juice compared with the Chardonnay juice. Higher concentrations of caftaric acid in Riesling juice when

compared with many other varieties has also previously been reported by Ong and Nagel (1978b). In addition, it has been observed that Riesling juice tends to brown much more than Chardonnay juice. The net phenolic extraction for a given juice fraction is derived from the actual phenolic extraction into that juice fraction minus those phenolics lost to phenolic oxidation and precipitation. It seems likely that there was considerably more phenolic oxidation with the Riesling juice than with the Chardonnay juice and thus the net phenolic extraction was much more closely related to the actual phenolic extraction for the Chardonnay. For this reason Chardonnay has been the main focus in this analysis as net phenolic extraction results give more insight into actual phenolic extraction.

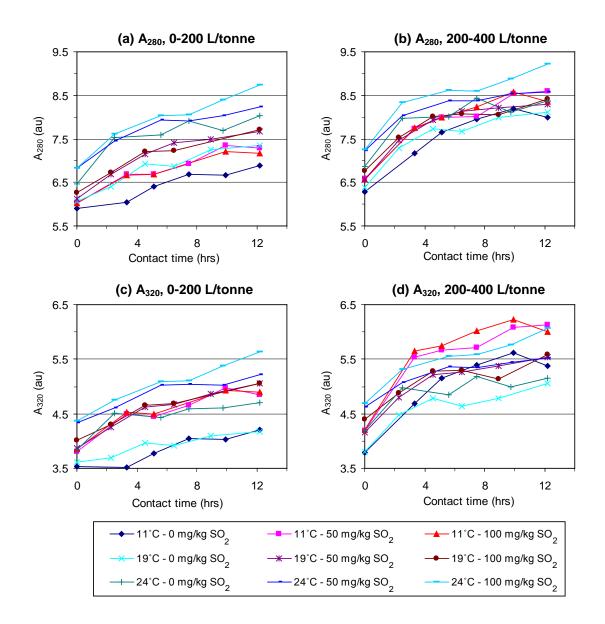


Figure 3.8: Extraction kinetics for Barossa Valley Chardonnay (100% crushed, Replicate 2)

Chapter 3: Phenolic extraction during pomace contact

		<u>0-200 L</u>	<u>/tonne</u>		200-400 L/tonne				
	A <sub>28</sub>	30	A <sub>320</sub>	)	A <sub>28</sub>	D	A <sub>32</sub>	0	
Regressors	βª	$\hat{\beta}_s^{\ b}$	β	$\hat{\boldsymbol{\beta}}_{s}$	β	$\hat{\boldsymbol{\beta}}_{s}$	β	$\hat{\boldsymbol{\beta}}_{s}$	
Intercept	5.7** (0.30)	0.16	3.7** (0.29)	0.32	7.2** (0.38)	0.25	5.8** (0.43)	0.21	
t (hours) <sup>°</sup>	0.18** (0.019)	0.71	0.12** (0.019)	0.62	0.34** (0.027)	0.79	0.29** (0.033)	0.63	
ť	-0.0065** (0.0010)	-0.17	-0.0037** (0.00098)	-0.13	-0.013** (0.0013)	-0.33	-0.010** (0.0015)	-0.29	
Т (°С) <sup>а</sup>	-0.034 (0.030)	0.55	-0.064* (0.029)	0.24	-0.13** (0.037)	0.17	-0.22** (0.042)	-0.24	
T <sup>2</sup>	0.0028** (0.00082)	0.11	0.0028** (0.00077)	0.15	0.0046** (0.00096)	0.18	0.0062** (0.0011)	0.26	
SO <sub>2</sub> (mg/kg)	0.0055** (0.0021)	0.23	0.016** (0.0021)	0.57	0.0047 (0.0029)	0.22	0.013** (0.0033)	0.38	
SO <sub>2</sub> <sup>2</sup>	-4.1×10 <sup>-5</sup> ** (1.3×10 <sup>-5</sup> )	-0.11	-0.00010** (1.3×10 <sup>-5</sup> )	-0.34	-3.9×10 <sup>-5</sup> * (1.7×10 <sup>-5</sup> )	-0.098	-7.0×10 <sup>-5</sup> ** (2.0×10 <sup>-5</sup> )	-0.19	
Day 2 <sup>e</sup>	0.14** (0.033)	0.11	0.14** (0.033)	0.15	0.17** (0.044)	0.13	0.14** (0.050)	0.11	
t × T	-7.5×10 <sup>-5</sup> 0.00085	-0.0024	-0.0011 (0.00084)	-0.048	-0.0031** (0.0011)	-0.097	-0.0040** (0.0013)	-0.14	
$t\times \textbf{SO}_2$	0.00022* 9.9×10 <sup>-5</sup>	0.057	0.00033** (9.6×10 <sup>-5</sup> )	0.11	6.9×10 <sup>-5</sup> 0.00012	0.017	8.3×10 <sup>-5</sup> (0.00014)	0.023	
T × SO₂	4.9×10 <sup>-5</sup> 9.5×10 <sup>-5</sup>	0.015	-0.00011 (9.0×10 <sup>-5</sup> )	-0.047	0.00013 (0.00012)	0.040	-6.6×10 <sup>-5</sup> (0.00013)	-0.023	
Summary				_		_			
R <sup>2</sup> <sub>adjusted</sub> n <sup>f</sup> F-value	0.94 100 247		0.90 100 129		0.90 101 151		0.85 101 105		

#### Table 3.5: Regression analysis for Barossa Valley Chardonnay (100% crushed)

 $^a$   $\hat{\beta}$  is the unstandardised regression coefficient. Standard errors are reported in parentheses underneath these coefficients.

 $^{\text{b}} \, \hat{\beta}_s$  is the standardised regression coefficient.

<sup>c</sup>t is the treatment time. <sup>d</sup>T is the treatment temperature.

<sup>e</sup> Day 2 is a dummy variable to indicate that sample was from the second day of experiments.

<sup>f</sup>n is the number of observations

\* Individual coefficient is statistically significant at 5% level.

\*\* Individual coefficient is statistically significant at 1% level.

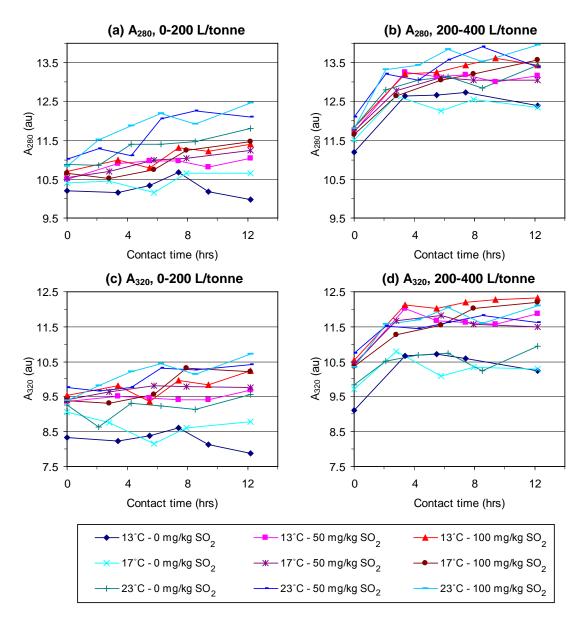


Figure 3.9: Extraction kinetics for Eden Valley Riesling (100% crushed)

		<u>0-200 L</u>	./tonne		200-400 L/tonne				
	A <sub>280</sub>		A <sub>320</sub>	I	A <sub>28</sub>	D	A <sub>32</sub>	20	
<u>Regressors</u>	$\hat{\beta}^a$	$\hat{\beta}_{s}^{b}$	β	$\hat{\boldsymbol{\beta}}_{s}$	β	$\hat{\boldsymbol{\beta}}_{s}$	β	$\hat{\boldsymbol{\beta}}_{s}$	
Intercept	13.8** (0.83)	-0.13	8.5** (0.99)	0.34	14.2** (1.2)	0.36	10.2** (1.5)	0.64	
t (hours) <sup>c</sup>	-0.058 (0.033)	0.42	-0.12** (0.037)	0.22	0.30** (0.049)	0.70	0.28** (0.058)	0.48	
ť	-0.0028 (0.0017)	-0.078	0.0012 (0.0019)	0.030	-0.019** (0.0019)	-0.47	-0.016** (0.0023)	-0.34	
Т (°С) <sup>d</sup>	-0.46** (0.096)	0.59	-0.036 (0.12)	0.39	-0.35** (0.12)	0.25	-0.091 (0.16)	0.00095	
T <sup>2</sup>	0.015** (0.0027)	0.40	0.0027 (0.0033)	0.064	0.011** (0.0033)	0.27	0.0037 (0.0042)	0.074	
SO <sub>2</sub> (mg/kg)	0.014** (0.0040)	0.42	0.034** (0.0045)	0.73	0.013* (0.0059)	0.35	0.036** (0.0072)	0.65	
SO <sub>2</sub> <sup>2</sup>	-6.0×10 <sup>-5</sup> ** (2.3×10 <sup>-5</sup> )	-0.18	-0.00017** (2.1×10 <sup>-5</sup> )	-0.43	-6.0×10 <sup>-5</sup> * (3.0×10 <sup>-5</sup> )	-0.16	-0.00018** (3.5×10 <sup>-5</sup> )	-0.38	
t × T	0.0069** (0.0013)	0.19	0.0055** (0.0016)	0.13	0.00040 (0.0024)	0.0098	-0.0025 (0.0026)	-0.051	
$t\times \textbf{SO}_2$	0.00055** (0.00014)	0.16	0.00084** (0.00019)	0.21	0.00058* (0.00023)	0.15	0.00077** (0.00026)	0.16	
$T\times \textbf{SO}_2$	-0.00027 (0.00016)	-0.075	-0.00054** (0.00020)	-0.13	-0.00024 (0.00025)	-0.059	-0.00050 (0.00029)	-0.10	
<u>Summary</u>									
R <sup>2</sup> <sub>adjusted</sub> n <sup>e</sup> F-value	0.89 51 54		0.89 51 48		0.85 50 46	5	0.85 50 45		

Table 3.6: Regression analysis for Eden Valley Riesling (100% crushed)

 $^{a}$   $\hat{\beta}$  is the unstandardised regression coefficient. Standard errors are reported in parentheses underneath these coefficients.

 $^{\scriptscriptstyle b} \, \hat{\beta}_s$  is the standardised regression coefficient.

<sup>c</sup>t is the treatment time. <sup>d</sup>T is the treatment temperature.

<sup>e</sup> n is the number of observations

\* Individual coefficient is statistically significant at 5% level.

\*\* Individual coefficient is statistically significant at 1% level.

### **3.2.6 Pomace contact (partial berry crushing)**

Experiments with completely crushed berries were useful for studying general phenolic extraction. However, they are really more applicable to pomace contact after explicit crushing than after machine harvesting, where there will be some but not complete berry breakage. Spectral results for experiments with partial crushing of Langhorne Creek Chardonnay (Replicate 1, see Appendix A for Replicate 2) and Eden Valley Riesling are presented in Figures 3.10 and 3.11, respectively. Corresponding regression analyses are reported in Tables 3.7 and 3.8.

Similar trends to those seen in experiments with completely crushed grapes can be observed. Phenolic levels increased with time for both Chardonnay and Riesling juice. The temperature at the time of berry crushing was again important, demonstrated by the higher phenolic levels in samples at 23 °C without any pomace contact time (i.e. 0 hours).

The fraction of crushed grapes was an extremely important factor in determining phenolic content. This is evident in Figure 3.10, where the phenolic levels at each temperature and contact time are generally lower for batches where 20% or 50% of grapes were crushed than in those batches where 100% of grapes were crushed. These lower levels of berry crushing are likely to be more representative of what could happen after machine harvesting.

To provide an indication of the magnitude of phenolic extraction under the more realistic conditions of partial berry breakage, the regression models for 280 nm and 320 nm of the Langhorne Creek Chardonnay (Table 3.7) were applied for 20% berry breakage, with a temperature of 20 °C and contact times of 0, 3, 6 and 12 hours (Table 3.9). The results for the 0-400 L/tonne fraction were calculated by taking the average of the regressed 0-200 L/tonne and 200-400 L/tonne values. Results are expressed as the change relative to the no-contact time treatment. The spectral results are reported together with the change in concentration of hydroxycinnamates and flavonoids, using the techniques of Somers and colleagues (Somers and Ziemelis 1985b, Somers and Vérette 1988 – see section 3.2.8 for a further discussion of these techniques). The flavonoids, the compounds most commonly associated with bitterness and astringency, increased in this 400 L/tonne fraction by 6, 11 and 19 mg/L (catechin equivalents) relative to the zero pomace contact time treatment, at times of 3, 6 and 12 hours, respectively.

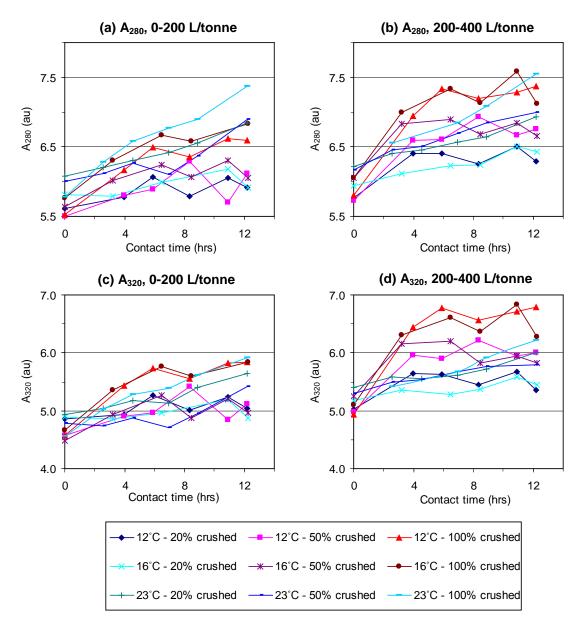


Figure 3.10: Extraction kinetics for Langhorne Creek Chardonnay (50 mg/kg SO<sub>2</sub>, Replicate 1)

		<u>0-200 L/</u>	tonne		200-400 L/tonne				
	A <sub>2</sub>	80	A <sub>32</sub>	0	A <sub>28</sub>	0	A <sub>320</sub>		
<u>Regressors</u>	β <sup>a</sup>	$\hat{\beta}_{s}^{b}$	β	$\hat{\boldsymbol{\beta}}_{s}$	β	$\hat{\boldsymbol{\beta}}_{s}$	β	$\hat{\boldsymbol{\beta}}_{s}$	
Intercept	7.4** (0.42)	-0.49	7.3** (0.49)	-0.77	7.4** (0.56)	0.11	7.1** (0.71)	0.095	
t (hours) <sup>c</sup>	0.027 (0.019)	0.56	0.069** (0.023)	0.56	0.14** (0.026)	0.59	0.17** (0.033)	0.47	
ť	-0.0022* (0.0011)	-0.096	-0.0034* (0.0013)	-0.15	-0.0077** (0.0013)	-0.30	-0.0093** (0.0017)	-0.33	
T (°C) <sup>d</sup>	-0.20** (0.049)	0.45	-0.28** (0.058)	0.019	-0.23** (0.063)	0.037	-0.30** (0.081)	-0.23	
T <sup>2</sup>	0.0068** (0.0014)	0.34	0.0083** (0.0017)	0.44	0.0073** (0.0017)	0.31	0.0097** (0.0022)	0.38	
Fr <sup>e</sup>	-1.09** (0.36)	0.32	-1.9** (0.41)	0.26	1.3** (0.42)	0.55	2.2** (0.53)	0.50	
Fr <sup>2</sup>	0.93** (0.25)	0.25	1.70** (0.29)	0.48	-0.53 (0.29)	-0.13	-0.73* (0.37)	-0.16	
Day 2 <sup>t</sup>	-0.00026 (0.034)	-0.00033	0.025 (0.041)	0.033	0.042 (0.041)	0.046	0.061 (0.054)	0.062	
t × T	0.0011 (0.00092)	0.049	-0.00079 (0.0012)	-0.038	-0.00086 (0.0011)	-0.035	-0.0027 (0.0015)	-0.099	
t × Fr	0.065** (0.012)	0.22	0.066** (0.016)	0.24	0.069** (0.015)	0.21	0.090** (0.021)	0.25	
T × Fr	0.0014 (0.011)	0.0051	-0.0093 (0.014)	-0.036	-0.023 (0.014)	-0.075	-0.070** (0.018)	-0.21	
<u>Summary</u>									
R <sup>2</sup> <sub>adjusted</sub> n <sup>g</sup> F-value	0.8 10 69	1	0.71 101 37		0.80 102 64	2	0.71 102 44		

#### Table 3.7: Regression analysis for Langhorne Creek Chardonnay (50 mg/kg SO<sub>2</sub>)

 $^a$   $\hat{\beta}$  is the unstandardised regression coefficient. Standard errors are reported in parentheses underneath these coefficients.

<sup>b</sup>  $\hat{\beta}_s$  is the standardised regression coefficient.

<sup>c</sup>t is the treatment time. <sup>d</sup>T is the treatment temperature.

<sup>e</sup> Fr is the fraction crushed

<sup>f</sup> Day 2 is a dummy variable to indicate that sample was from the second day of experiments.

<sup>g</sup> n is the number of observations

\* Individual coefficient is statistically significant at 5% level.

\*\* Individual coefficient is statistically significant at 1% level.

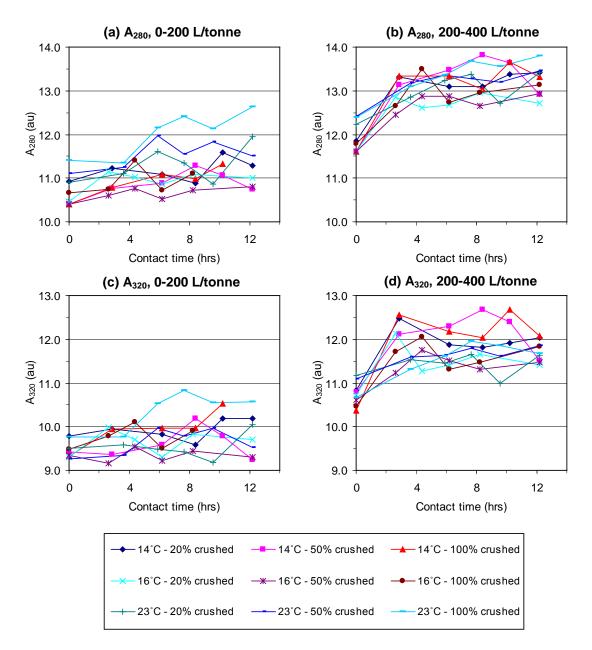


Figure 3.11: Extraction kinetics for Eden Valley Riesling (50 mg/kg SO<sub>2</sub>)

		<u>0-200 L</u>	<u>/tonne</u>	200-400 L/tonne					
-	A <sub>28</sub>	30	A <sub>320</sub>	)	A <sub>2</sub>	80	A <sub>320</sub>		
Regressors	$\hat{\beta}^a$	$\hat{\beta}_s^{\ b}$	β	$\hat{\boldsymbol{\beta}}_{s}$	β	$\hat{\boldsymbol{\beta}}_{s}$	β	$\hat{\boldsymbol{\beta}}_{s}$	
Intercept	18.0** (2.1)	-0.63	16.8** (2.3)	-0.91	19.5** (2.9)	0.019	18.5** (3.6)	0.081	
t (hours) <sup>c</sup>	0.012 (0.054)	0.40	0.018 (0.052)	0.36	0.34** (0.069)	0.62	0.29** (0.076)	0.44	
ť	-0.0036 (0.0022)	-0.11	-0.0034 (0.0024)	-0.13	-0.017** (0.0025)	-0.48	-0.016** (0.0029)	-0.50	
Т (°С) <sup>а</sup>	-0.75** (0.23)	0.34	-0.70** (0.26)	-0.20	-0.85** (0.31)	-0.11	-0.81* (0.39)	-0.49	
T <sup>2</sup>	0.020** (0.0061)	0.46	0.017* (0.0069)	0.51	0.023** (0.0080)	0.47	0.021* (0.010)	0.47	
Fr <sup>e</sup>	-3.9** (0.81)	0.072	-4.3** (0.85)	0.19	-0.61 (1.0)	0.14	0.0090 (1.3))	0.083	
Fr <sup>2</sup>	1.29** (0.46)	0.27	2.03** (0.50)	0.54	-0.043 (0.60)	-0.0080	-0.21 (0.71)	-0.042	
t × T	0.0032 (0.0025)	0.088	0.0014 (0.0026)	0.048	-0.0031 (0.0030)	-0.076	-0.0038 (0.0036)	-0.099	
t × Fr	0.043 (0.027)	0.11	0.061* (0.029)	0.20	0.024 (0.030)	0.056	0.073 (0.039)	0.18	
T × Fr	0.13** (0.035)	0.29	0.11** (0.035)	0.31	0.042 (0.035)	0.082	-0.0053 (0.042)	-0.011	
<u>Summary</u>									
R <sup>2</sup> <sub>adjusted</sub> n <sup>†</sup> F-value	0.75 52 25		0.56 52 16		0.7 54 23	1	0.47 54 11		

<sup>a</sup>  $\hat{\beta}$  is the unstandardised regression coefficient. Standard errors are reported in parentheses underneath these coefficients.

 $^{\scriptscriptstyle b} \, \hat{\beta}_s$  is the standardised regression coefficient.

<sup>c</sup>t is the treatment time. <sup>d</sup>T is the treatment temperature.

<sup>e</sup> Fr is the fraction crushed

<sup>f</sup>n is the number of observations

\* Individual coefficient is statistically significant at 5% level.

\*\* Individual coefficient is statistically significant at 1% level.

Analyta	Time (hours)						
Analyte	0	3	6	12			
A <sub>280</sub> (au)	5.87	+0.25	+0.41	+0.46			
A <sub>320</sub> (au)	4.90	+0.24	+0.38	+0.29			
ΔHydroxycinnamates (mg/L caft. acid eq.) <sup>a</sup>		+5	+7	+6			
ΔFlavonoids (mg/L cat. eq.) <sup>b</sup>		+6	+11	+19			

Table 3.9: Indicative quantification of phenolic levels for Langhorne Creek Chardonnay using
regression model (20% crushed, 50 mg/kg SO <sub>2</sub> , 400 L/tonne, 20 °C)

<sup>a</sup> Change in hydroxycinnamate concentration relative to zero contact time (in caftaric acid equivalents).  $\Delta$ Hydroxycinammates (mg/L caftaric acid equivalents) ~  $\Delta A_{320} \times 10/0.9 \times 7/4$ . <sup>b</sup> Change in flavonoids relative to zero contact time (in catechin equivalents).

 $\Delta$ Flavonoids (mg/L catechin equivalents) ~ ( $\Delta$ A280 - 2/3 ×  $\Delta$ A<sub>320</sub>) × 10/0.14.

### 3.2.7 Yields

In this work, juice fractions corresponding with yields of 0-200 L/tonne and 200-400 L/tonne were collected. The total juice expressed at wineries can be around 750 L/tonne. Preliminary laboratory experiments demonstrated that obtaining total yields anywhere near 750 L/tonne was very difficult to achieve quickly and furthermore significant mechanical action would be required. The specific mode of mechanical action was likely to be a far more influential determinant of phenolic levels than the pomace contact.

While overall juice yields are important, the earlier juice fractions are generally considered to be more valuable than later juice fractions. It was thus decided to focus on early juice fractions in this work and to look more specifically at industrially realistic juice expression in subsequent work. 400 L/tonne could be collected relatively quickly with simple laboratory equipment and also corresponds approximately with the yield of juice that can be obtained in industry by a short period of static draining. It was however decided to collect this juice in two yield fractions, to try and gain an improved understanding of the transport mechanisms involved.

While it is difficult to know what exactly would happen with the higher yield fractions, as discussed in section 3.2.5 higher yield fractions would likely come from closer and closer to and from the skin. Given the small skin cell size relative to the outer pulp cells, but their much higher phenolic concentration, diffusion from damaged skin cells into outer pulp cells would likely be very rapid initially and then decrease quickly. Therefore, it might be expected that for higher juice yields, pomace contact time beyond quite short periods, may have a fairly limited effect on phenolic content for these yield fractions.

### 3.2.8 Analytical techniques

Aggregate UV spectral techniques have been primarily used for phenolic analysis in this thesis. UV techniques are simple and rapid and have been quite widely used both in research and by industry

(particularly in Australia). They have been reported to compare well with HPLC measurements (Somers 1987, Somers and Vérette 1988). HPLC is a superior technique for quantification of specific phenolic compounds and was originally intended to be used extensively in this work; however, funding delays for a project collaborator meant that unfortunately HPLC was not a regular feasible option for this work.

UV absorbance of white grape juice and wine samples is derived from both phenolic and nonphenolic compounds. The significant non-phenolic absorbance has been attributed to nitrogenous compounds, principally nucleotides (Somers and Ziemelis 1972, Somers and Ziemelis 1985b). Somers and Ziemelis (1985b) investigated this background absorbance by fining samples with 100 g/L PVPP (Polyclar AT, originally manufactured by GAF but which has now been superseded by Polyclar VT manufactured by ISP) in order to completely and specifically remove phenolics. They found the remaining non-phenolic absorbance to be quite uniform for 230 commercial wines over several vintages and consequently advocated the use of constants from that study (1.4 au at 320 nm and 4 au at 280 nm) for this non-phenolic background absorbance in routine analysis of white grape juices and wines. Where more accurate results were required, they suggested that stripping of individual samples with PVPP to determine the non-phenolic absorbance was preferable.

Selected samples from maceration experiments were analysed by UV spectroscopy after 100 g/L PVPP treatment (Figure 3.12, and numerous other samples not shown) to more accurately determine the non-phenolic background absorbance of different samples. These measurements demonstrated that the absorbance after PVPP treatment was not constant, and varied between grape varieties, grape lot and with the processing conditions used to derive the sample. Notably, samples that featured higher absorbance without PVPP treatment still typically featured higher absorbance after PVPP treatment. For example, samples subjected to 12 hours pomace contact had higher residual UV absorbance after PVPP treatment than samples not subjected to pomace contact. Similar trends were obtained for PVPP treatment of winery and laboratory draining and pressing samples (see Appendices B and C). This could indicate extraction of UV absorbing non-phenolic components, incomplete removal of phenolic components by 100 g/L PVPP treatment, or a combination of both.

An opportunity arose to have a limited number of samples tested for specific phenolic compound concentration by a proprietary HPLC technique. The results for five juice samples with and without PVPP treatment are presented in Table 3.10. While PVPP appreciably removed many of the phenolic compounds assayed, it left relatively large residual concentrations of GRP (Grape Reaction Product, the glutathionyl derivative of caftaric acid) and a complex peak eluting towards the end of the HPLC method, referred to as "Tannin". Tryon et al. (1988) also noted a variable efficiency of PVPP to strip different phenolics, even at this massive dose. Therefore, the use of PVPP treatment as a means of stripping phenolic compounds for determination of sample non-phenolic UV absorbance is not entirely satisfactory.

Somers and Ziemelis (1985b) further advocated a means of estimating flavonoid concentration:

Flavonoid concentration =  $(A_{280} - 4) - 2/3(A_{320} - 1.4)$  au =  $(A_{280} - 4) - 2/3(A_{320} - 1.4) \times 10/0.14$  mg/L catechin equivalents

This employed the non-phenolic correction factors at 280 nm and 320 nm and a typical ratio of hydroxycinnamate absorption at 280 nm to that at 320 nm of 2/3. A problem evident with the explicit estimation of flavonoid concentration directly from spectral measures is the relatively higher extinction coefficients of the hydroxycinnamates compared to flavan-3-ols and proanthocyanidins (see Figure 3.4). Thus relatively small changes in absorption may indicate a sensorially significant increase in flavonoid concentration, but could be convoluted with small changes in hydroxycinnamate concentration.

Despite some problems with spectral techniques, they remain of practical use in terms of understanding white wine production processes, particularly under process conditions like those in this thesis, where flavonoid and non-flavonoids are largely being co-extracted from the same location in the berry, the skin. The absorbance changes at 320 nm are related to hydroxycinnamates, and can provide useful information on oxidation processes, while absorbance changes at 280 nm can be related to total phenolics more generally. The approach taken in this thesis to manage these complicating factors in phenolic determination, has been to principally look at raw  $A_{280}$  and  $A_{320}$  results, without non-phenolic correction factors, to analyse results for individual grape lots separately, and to look more so at changes rather than absolute values, such that results tend to be overestimated rather than underestimated in the case where there is extraction of non-phenolic compounds.

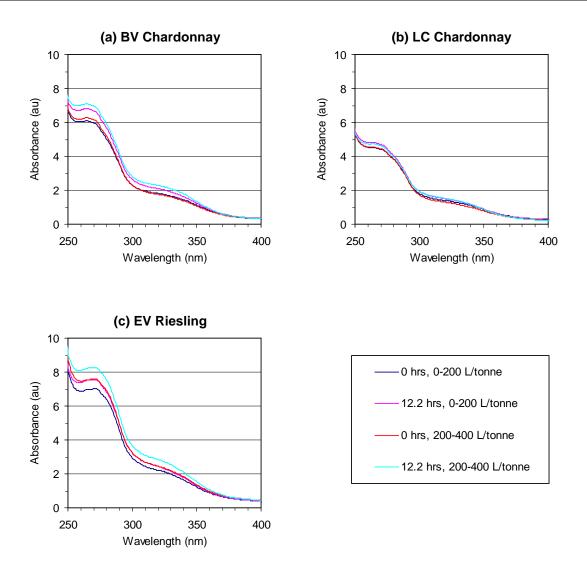


Figure 3.12: Spectra after treatment of juice samples with 100 g/L PVPP (Samples from pomace contact experiments, 100% crushed, 50 mg/kg SO<sub>2</sub>, approximately 23 °C)

		Phenolic compounds (mg/L) <sup>a</sup>									
Sample		Caftaric acid	Coutaric acid	GRP	Caffeic acid	Gallic acid	Catechin	Tannin <sup>b</sup>	Quercetin glycosides	Quercetin	Astilbin
Thompson	-	3.5	1.3	4.2	0.0	0.0	10.2	18.3	17.8	0.0	0.0
Seedless	PVPP	0.8	0.0	3.4	0.0	0.0	0.8	8.1	0.0	0.0	0.0
Chardonnay	-	3.1	0.8	11.8	0.0	0.0	1.2	15.3	0.0	0.0	0.1
A	PVPP	0.2	0.0	4.6	0.0	0.0	0.0	9.8	0.0	0.0	0.0
Chardonnay	-	19.3	8.1	9.7	0.0	0.0	3.4	17.1	1.4	0.0	1.3
B	PVPP	0.9	0.6	4.9	0.0	0.0	0.2	11.7	0.0	0.0	0.0
<b>Riesling A</b>	-	28.8	4.1	8.3	0.0	0.2	1.0	26.0	4.0	0.0	0.3
	PVPP	1.6	0.4	4.1	0.0	0.0	0.6	10.8	0.0	0.0	0.0
Riesling B	-	54.8	11.1	9.8	0.0	0.1	1.4	23.1	5.4	0.0	1.2
	PVPP	4.5	1.5	6.0	0.0	0.2	0.5	13.0	0.0	0.0	0.0

Table 3.10: HPLC analysis of juice samples with and without PVPP treatment

<sup>a</sup> Caftaric acid, coutaric acid, GRP (grape reaction product) concentrations are expressed in caffeic acid equivalents. Tannin is expressed in catechin equivalents. Quercetin glycosides are expressed in quercetin equivalents. Concentrations were measured and reported by a third party using a proprietary HPLC method.

<sup>b</sup> Tannin refers to a complex peak eluting towards the end of the HPLC method.

### 3.3 Conclusions

Spectral analysis confirmed that seeds contain higher concentrations of phenolics than skins, however, spectral changes during pomace contact suggest that much of the extraction during pomace contact was from the skins.

Higher yield fractions had higher phenolic levels with similar treatment conditions. Temperature at the time of berry breakage, contact time, sulfur dioxide concentration and the fraction of broken berries were all influential factors in determining juice phenolic concentration. There was some decrease in the net phenolic extraction rate with time and this was likely principally related to concentration gradient effects rather than enzymatically catalysed phenolic oxidation and precipitation.

The wine industry is correct to be cautious about the temperature at the time of machine harvesting and the time lag between harvesting and winery processing. Where practicable these values should be minimised. However, given the uncertainty in the amount of berry breakage in industrial practice, wineries should perform full scale trials where economic advantage could be gained from relaxing restrictions on times between harvesting and winery processing.

In this experimental work, two yield fractions were collected: 0-200 L/tonne, and 200-400 L/tonne. These early fractions were deemed to be important as they are often the most valuable, and on an

industrial scale may be collected after crushing by a short period of static draining. The total juice expressed from white grapes at wineries may be in the order of 750 L/tonne; however recovery of this further juice requires more extensive agitation and pressing. Phenolic levels in these fractions are likely to be heavily influenced by the method of expression. Juice expression will be addressed more specifically in the following chapters.

# CHAPTER 4: JUICE EXPRESSION OBJECTIVES, DESTEMMING, CRUSHING AND DRAINING

In this chapter, the general objectives of juice expression are outlined together with a review of key principles and practices of equipment that is or has been used in the wine industry for destemming, crushing and draining. In Chapter 5 equipment that has been used for pressing is reviewed. In Chapter 6 economic considerations in juice expression are discussed.

Expression equipment for white wine production has not been systematically reviewed in recent decades. There is also a great deal of information in old advertisements and in European non-English journals and trade magazines, which have not been interpreted or referenced in English.

It is notable that manufacturers now tend to offer very similar equipment with only superficial differences. For example, membrane presses dominate the recent trade literature. In trade and academic journals and at trade shows there also tends to be a disproportionate focus on quality at the expense of productivity, inconsistent with Australian wine industry requirements given that much of Australia's wine output is commercial premium wine. In this work, techniques consistent with large scale production will be considered.

Design and construction of juice expression equipment is highly empirical and therefore records of past experience, whether it be from the wine industry or other industries is critical. The problems associated with juice expression are not new and inferences drawn from the evolution of equipment could aid development of equipment and prevent the unnecessary repetition of device construction with foreseeable inadequacies.

## 4.1 Objectives in white juice expression

The ultimate goal of juice expression is the production of juice that can be fermented into white wine.

Juice for white wine production is typically expressed from harvested white grapes by a sequence of destemming, crushing, draining, and pressing. Individual devices may perform one or more of these steps. For example, draining may be performed in the press during loading. Alternatively, grape clusters may be pressed directly, without destemming, crushing or draining, as is the traditional practice in the Champagne region of France (Peynaud 1984, Rankine 2004, Ribéreau-Gayon et al. 2006b).

As outlined in Chapter 2, low juice phenolic content is important for white wine quality. Low solids content before fermentation has also been demonstrated to be a significant factor in white wine quality (Singleton et al. 1975, Williams et al. 1978, Liu et al. 1987). Additional pre-fermentation juice clarification is typically performed to reduce expressed juice solids levels to appropriate levels

(depending on variety and style). Low solids levels in expressed juice are therefore advantageous. In addition to the overall quantity of solids, the ease of removal of these solids is also important; small solids may be more difficult to remove than large solids and extraction of other grape components like pectins during expression may inhibit solids removal. Limiting juice oxidation to some extent during expression processes is also generally regarded as desirable. This view is supported somewhat by the neutral or negative sensory results from studies of juice hyperoxidation (Singleton et al. 1980, Nagel and Graber 1988, Cheynier et al. 1989, Dubourdieu and Lavigne 1990, Cheynier et al. 1991, Guedes de Pinho et al. 1994), despite the accompanying reductions in phenolic content. A fractional expression is important, whereby the higher quality juice derived principally from the pulp can be collected in a separate fraction or fractions to the lower quality juice derived from closer to and from the grape skins.

In summary, some desirable quality and operational/productivity related features of juice expression equipment that will ultimately influence expression economics are:

Quality related:

- Fractional expression (i.e. the ability to collect the higher quality juice in a separate fraction or fractions to the lower quality juice)
- Low juice phenolic content
- Low and easily removable juice solids content
- Limited juice oxidation

Operational/productivity related:

- High yield (in particular of the higher quality juice fractions)
- High throughout (but with a good turndown capability)
- Small hold-up volume of grapes (low residence time and limits potential losses in the case of a breakdown)
- Small footprint (can be retro-fitted to old facilities with limited space)
- Highly automated (low labour requirements skilled labour in particular)
- Sanitary, easy to clean (preferably avoiding confined space cleaning)
- Easy to maintain
- Flexibility to work with all grape varieties and condition
- Rapid start-up and shutdown and changeovers between different lots
- Low power usage
- Safe

### 4.2 Destemming and crushing

Destemming and crushing are generally the first winery-based processing steps for white wine production. Machine harvested grapes have already been partially destemmed by the action of the grapes being shaken from the vine. Some companies (e.g. Pellenc) are beginning to introduce novel

supposedly gentle harvester-mounted destemmers to complete the action, but these are only a new innovation and have apparently not yet been trialled in Australia.

Stems contain high quantities of phenolic compounds that could potentially be transferred into juice. Inclusion of stems also reduces drainer/press capacity, but it does provide a relatively elastic and open cake structure that can facilitate juice drainage (Peynaud 1984, Ribéreau-Gayon et al. 2006b).

Industrial scale destemming is typically accomplished by a device employing paddles on a horizontal rotating spindle within a cylindrical cage as shown in Figures 4.1 and 4.2. The grapes are removed from the stems and fall through perforations in the cage, while the stems are conveyed out the end of the cylinder. In modern destemmers, the cage typically rotates in the same direction but at a slower speed to the main shaft. Independent adjustment of spindle and cage speeds allows for optimisation. Commonly, a metal ribbon on the external circumference of the rotating cage screw-conveys separated grapes towards an exit chute.

With low paddle speeds, grape clusters are just destemmed. At faster paddle speeds, grapes are also crushed (Agostini 1965). This principle was employed in older-style centrifugal crusher-destemmers to simultaneously destem and crush. These high speed devices tended to result in significant damage to grape cluster components, potentially producing high levels of phenolics and solids (Berti 1965, Anon 1986, Ribéreau-Gayon et al. 2006b).

Crushing is now typically performed in a separate mechanical action by relatively gentle roller crushers. These consist of one or more pairs of rollers, which turn in opposite directions bursting berries as they pass between. Commonly the rollers feature interlocking lobes like those shown in Figure 4.3. Roller spacing is typically adjustable and is set so that the berries are burst but the seeds are not broken (Rankine 2004). While roller crushing is performed before destemming in some smaller devices, in commercial equipment destemming generally takes place prior to roller crushing. Crusher rollers are typically integrated underneath the destemmer on a hinge or tracks and can be easily removed to allow destemming without explicit crushing. Crushing can be performed without destemming with some devices by way of opening a bypass at the entrance to the destemmer.

Modern destemmers with associated roller crushers are available off the shelf from numerous manufacturers with continuous throughputs up to around 80 tonnes/hour (eg. CME, Bucher-Vaslin, Diemme, Miller, Della Toffola, Pera, Velo). Given these high continuous throughputs and the fact that they are relatively much cheaper than pneumatic membrane presses, they are unlikely to be a major processing bottleneck in expression operations.



Figure 4.1: Destemmer paddles (view from stem-exit end)

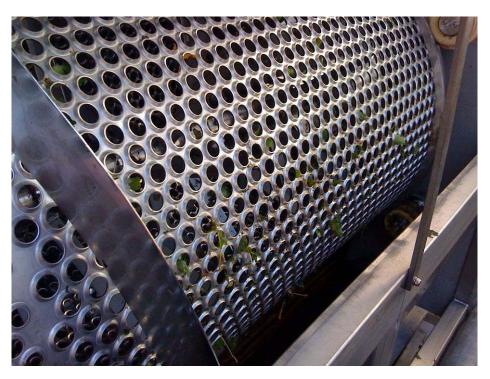


Figure 4.2: Destemmer cage (view from outside)



Figure 4.3: Crusher rollers

## 4.3 Non-mechanical treatments to facilitate expression

Juice expression may also be facilitated by weakening grape cells by non-mechanical means.

Wucherpfennig and Troost (1962) demonstrated that standing crushed Riesling mash for three days allowed the draining and pressing time to be almost halved. They attributed this to endogenous grape juice enzymes breaking down cell wall pectin. Higher levels of phenolics were a negative side effect. Commercial pectinase preparations can also aid the ease of juice expression (Ough and Berg 1974, Ough and Crowell 1979). However, concerns of increased phenolic levels have led to some wineries only adding them after the juice has already been expressed (McLean 2006) to aid juice clarification since pectins can hinder settling (Ough and Berg 1974, Ough and Crowell 1979).

Wucherpfennig and Troost (1962) also investigated the use of high temperature treatment to facilitate expression. Destemmed and crushed grapes were heated to 45 °C and held for 2 hours or heated to 80 °C and then cooled back to 20 °C before draining and pressing. In both cases, draining and pressing time was reduced by around 40% compared with an immediately drained and pressed control. Phenolic levels were much higher than the control though and this is the likely reason that heat treatment is not widely employed.

The use of pulsed electric fields (PEF) is another non-mechanical technique that has been trialled with various fruits and vegetables. PEF involves the application of electric fields to increase cell permeability and therefore, the ease of juice release. The electric field is applied in short pulses to

minimise power consumption and to limit heating (Vorobiev and Lebovka 2006). In contrast to intensive mechanical treatment, electrical permeabilisation of cell membranes leaves the cell walls relatively intact, and therefore undesirable cell wall components, such as pectin, remain largely with the marc (Vorobiev et al. 2007). In very small scale laboratory trials with white grapes, Praporscic et al. (2007) and Grimi et al. (2009) demonstrated an increase in the speed of mechanical expression when employing PEF treatment. However, this technique could be problematic as it may prevent a fractional expression if the skin cells are permeabilised at the same time as the pulp cells.

### 4.4 Draining

Draining is the collection of the free juice that has already been largely expelled from the berry. Draining can usually be performed in the press, however there are some advantages to a separate draining step, such as a reduction in the required press capacity.

Numerous devices have been developed for draining. They are broadly categorised as batch or continuous in the following review. Specific devices have been chosen because they illustrate different principles of operation and in some instances because they have been commonly used in Australia.

#### 4.4.1 Batch/static drainers

Static drainers are batch devices consisting of some form of screen for separation of juice from solids. These screens have been constructed from wooden or metal slats, perforated metal sheet, or bars that taper in thickness from feed to product - a profile that tends to minimise screen blinding (Perry et al. 1998).

In addition to reducing required press capacity, some static drainers can act as a buffer to balance the irregular rate at which grapes arrive at a winery against available press capacity. Depending on the style of press to be used, they may also allow for the collection of a higher quality low yield juice fraction. Static drainers are sometimes located directly over the press to allow for easy press loading or in other cases conveyors are used to transport the drained pomace to the press. Depending on the winery layout and the means of transport, undesirable oxidation or solids generation may result. There are also likely to be labour requirements in managing these transfers between drainers and presses.

As an aside, natural settling for skin separation is another practice that can be loosely grouped with static drainers, since it involves the batch collection of low yield juice but without a screen (Boulton et al. 1996). In this technique crushed grapes are transferred to a tank and the skin cap is allowed to rise. The juice is then drawn off from a point near the bottom of the tank. This practice generally results in juice with high solids (Boulton et al. 1996) and may also be undesirable because of

excessive s kin c ontact t ime (Berti 1965). T his practice was reportedly common in C alifornia (Berti 1965, Boulton et al. 1996). It is not clear if this technique is still employed.

Early static drainers consisted of tanks lined with wooden slats. Crushed grapes were loaded into the tank and the juice was drained through the slats. The remaining material had to be manually removed from t he t ank, at t he c ompletion of dr aining (Ambrosi et a I. 19 66). T he dr ainers r elied on t he development of a filter cake to remove juice suspended solids. During loading the initial juice would not be c lear but by the time a filter cake approximately 20 cm thick had f ormed, the drained juice would be quite clear (MacKenzie 1968). The major disadvantages of this method were the l abour required for r emoval of the pom ace, and t he difficulty in cleaning bet ween the wooden slats. The development of static drainers with a utomatic emptying of pom ace significantly reduced the labour requirements.

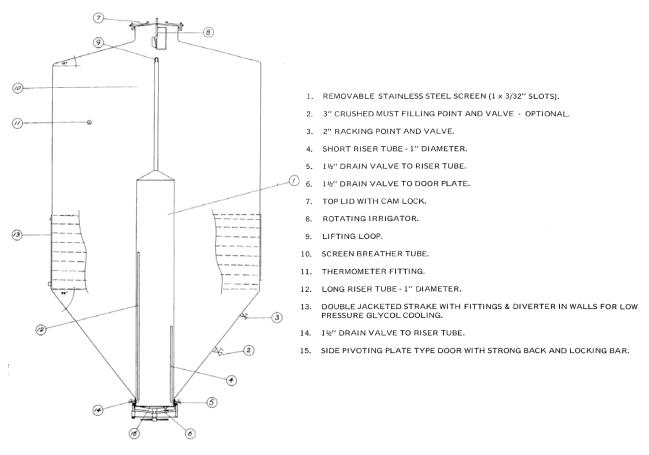
The Mackenzie static type separator, as illustrated in Figure 4.4, is an example of such a drainer. It was used in South Africa and by at least one winery in Australia (MacKenzie 1968). The V-shaped screen-lined c ontainer led t o t he qu ick formation of a gr ape f ilter bed ar ound t he de livery p ipe discharge, and therefore relatively clear juice s oon after beginning to load crushed grapes into the separator. As with many static drainers, grapes were often not destemmed in order to permit faster draining (MacKenzie 1968). However, binding of the relatively dry dr ained material together by the stems did sometimes lead to difficulties with emptying the pomace – a problem not observed with destemmed grapes (Ambrosi et al. 1966).

NOTE: This figure is included on page 50 of the print copy of the thesis held in the University of Adelaide Library.

### Figure 4.4: MacKenzie static type separator (From: Sperling and Ambrosi 1964)

The Potter drainer/fermenter is a drainer that has apparently been used much more extensively in Australia (Figure 4.5). This vessel can be closed to the atmosphere limiting oxidation. Drainage is

through the central screen. On completion of draining the bottom door could be opened freely as the drained pomace is retained by this central screen. The screen is then raised allowing the pomace to fall from the vessel (Agricultural and General c.1970). The Potter drainer/fermenter is a multi-purpose vessel. It can be used for intentional white grape pomace contact to extract desirable skin aroma compounds and precursors, red wine fermentation and for general storage. Multi-purpose devices of this nature are desirable as they can be utilised year round, not only during vintage (Troost 1972).



#### Figure 4.5: Potter drainer/fermenter (From: Agricultural and General c.1970)

In addition to employing vessels that are relatively closed, carbon dioxide has also been utilised in many devices to further limit oxidation. In some devices, pressurisation with carbon dioxide has been used to increase the speed of juice draining and/or increase juice yield. The South African MacKenzie carbon dioxide pressure separator (Figure 4.6), the Australian Miller pressure drainer (Figure 4.7), and the American Winery Systems juice separator (Figure 4.8) are three such examples.

The Miller pressure drainer is of particular interest because they have been commonly used in Australia. They were first installed in 1968 (Rankine 1996). Use of the Miller pressure drainer involves first purging the chamber of air using a pressurised supply of carbon dioxide. Must is pumped into the chamber and draining starts immediately. Carbon dioxide pressure is maintained at approximately 0.3 bar and slow agitation is provided at the centre of the must to aid drainage. For the discharge of the

drained pomace, the carbon dioxide supply is turned off, the discharge doors opened and the pomace is discharged by parallel twin counter-rotating screws. The use of twin screws prevents bridging of drained pomace over the screws (Draper 1973). Miller drainers are still used in the Australian wine industry and have recently been observed at one winery being used solely for gravity draining without carbon dioxide pressure. The agitator bar had also been removed.

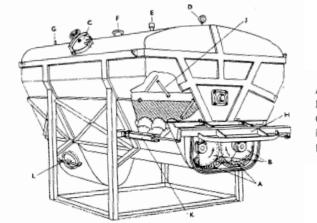
Waste c arbon d ioxide f rom fermentation has been r ecycled f or us e i n s ome dr ainers. T his was reportedly the case for the Mackenzie carbon dioxide pressure separator (MacKenzie 1967).

Carbon dioxide pressurised drainers required some means to ensure that carbon dioxide pressure continued to act to drive juice through the pomace filter bed for as long as possible. For example the American Winery Systems juice separator shown in Figure 4.8 used a central vertical screen like the Potter drainer but employed horizontal bu kheads with valves to ensure that the carbon dioxide pressure continued to act to force the juice through the cake as the juice level fell (Cottrell 1975, Zepponi and Cottrell 1975).

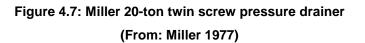
NOTE: This figure is included on page 52 of the print copy of the thesis held in the University of Adelaide Library.

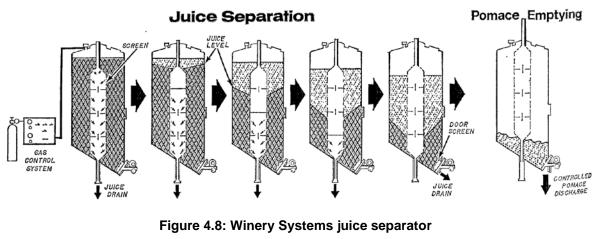
Figure 4.6: MacKenzie carbon dioxide pressure separator

(From: MacKenzie 1967)



A—Draining Screens B—22" dia. Screws C—18" dia. Manhole D—Pressure Gauge E—Safety Valve F---Must inlet G---CO<sub>2</sub> Gas Inlet H---Discharge Door J---Agitator K---Air Cylinders





(From: Cottrell 1975)

A much more recent development in static draining technology is the Pera Elite Tank (Figure 4.9). This device uses an inflatable membrane to drive additional expression when gravity juice drainage subsides. It is similar to a pneumatic membrane press but without the ability to rotate and crumble the cake. A low incrementally increasing pressure programme is employed (0.1–0.4 bar) and screw conveyors are fitted for pomace removal.

Generally, static drainers produce juice with relatively low suspended solids and phenolics as a result of the pomace being relatively static and not subject to significant shearing. Passage through the pomace bed also provides a filtering effect.

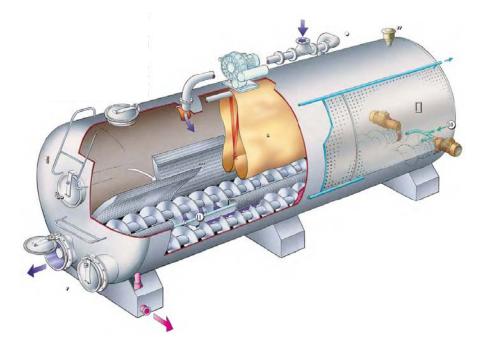


Figure 4.9: Pera Elite Tank (From: Pera 2010)

### 4.4.2 Continuous drainers

A wide variety of different continuous draining equipment has been used in the wine industry including rotating cylindrical screens, vibrating screens, Dutch States Mines screens, drag cleat screens and inclined drainers (Fabre 1929, Ventre 1929, Benvegnin et al. 1951, Sperling and Ambrosi 1964, Berti 1965, Ambrosi et al. 1966, Troost 1972, Peynaud 1984, Boulton et al. 1996).

Rotating screens may have been relatively commonly used in an earlier era based on visual depictions in several early winemaking textbooks (Fabre 1929, Benvegnin et al. 1951). An example of a rotating cylindrical screen is presented in Figure 4.10. Crettenand et al. (1969) reported that rotating screens only collected 30-40% of the available juice, and found that they produced very high juice solids. This is a likely reason for their apparent fall from favour. The low rate of juice collection is consistent with likely low residence times, and small heads of pressure from small cake heights. High solids levels are consistent with the shearing action on the grapes created by screen rotation and the constant breaking up of the cake, limiting filtration. Screen blinding may have been another difficulty, but some devices were reportedly fitted with brushes to clear the screen apertures (Fabre 1929). Other early continuous drainers likely suffered from similar issues, particularly low juice yields and high solids. Berti (1965) states that drag cleats screens tend to produce high solids juice and that vibrating screens can significantly aerate juice.

The most widely used type of continuous drainer is the inclined drainer. This device principally consists of an inclined screw conveyor encased by a cylindrical screen, through which juice drains. To achieve further expression from the drainer, some models apply additional pressure to the pomace by

a reduction of the screw pitch and/or an adjustable pomace discharge door. Inclined drainers are very similar to continuous screw presses but work at lower pressures. An example of an inclined drainer being used in conjunction with a screw press is presented in Figure 4.11. Inclined drainers have many of the same features as continuous screw presses. For example to prevent backflow of material towards the hopper, similar anti-return devices are employed (these are discussed in further detail in section 5.2.1). Toothed wheels, with their axis perpendicular to that of the main screw were used on earlier devices. The teeth would engage between the ridges of the screw limiting backflow. In more recent times, static anti-return devices such as that shown in Figure 4.12 have been used. To aid screen cleaning, some inclined drainers, such as the one presented in Figure 4.13 have featured brushes on the screw thread.

Inclined drainers have a number of advantages over static drainers. They can achieve a very high throughput (up to around 50 tonnes/hr), possess a small footprint and have a simple continuous operation with low labour requirements. They also conveniently raise the pomace so it can be easily fed to a press. Inclined drainers also typically collect a higher yield than static drainers. While basic gravity static drainers may only collect approximately 50% of the available juice, inclined drainers can separate up to 80% of the available juice (Peynaud 1984). However, inclined drainers are reported to produce higher solids juice compared to static drainers (Agostini 1965, Troost 1972, Menegazzo et al. 1977, Boulton et al. 1996, Ribéreau-Gayon et al. 2006b). This is likely related to shearing of pomace producing solids and the continual break up of the already thin pomace cake, limiting filtration through the cake. This is apparently a major issue with their use. Because of their higher yield than static drainers, to collect additional juice (Agostini 1965, Amerine et al. 1980).

NOTE: This figure is included on page 56 of the print copy of the thesis held in the University of Adelaide Library.

Figure 4.10: Rotating cylindrical draining screen prior to press basket loading (From: Benvegnin et al. 1951)

NOTE: This figure is included on page 56 of the print copy of the thesis held in the University of Adelaide Library.

Figure 4.11: Inclined drainer in conjunction with a screw press (Adapted from: Terrier and Blouin 1975)



Figure 4.12: Static anti-return device on a partially disassembled inclined drainer



Figure 4.13: Inclined drainer feed hopper with brushes on screw thread

## 4.5 Conclusions

Key objectives in juice expression include the production of juice low in solids and phenolics that can be fermented into quality white wine and the expression of this juice at high yields and throughputs with low labour requirements.

Continuous equipment capable of high throughputs is now typically employed for destemming and crushing and should not be a bottleneck in production. Both batch and continuous equipment have been used for draining. Batch static drainers typically produce higher quality juice lower in solids than continuous inclined drainers; however, inclined drainers achieve much higher throughputs.

The next step in juice expression for white wine production, pressing, is reviewed in the following chapter. There is some overlap with the current chapter as pressing can be performed without destemming, crushing and draining and often destemmed and crushed grapes are drained in the press itself.

# **CHAPTER 5: PRESSING**

Pressing is performed to express additional juice to that expressed from draining alone. As mentioned in the previous chapter, presses are also often used to perform the draining function, instead of employing a separate drainer. The following review divides presses into two categories based on their mode of operation: batch or continuous.

### 5.1 Batch presses

#### 5.1.1 Vertical basket presses

The vertical basket press is the traditional wine press, an example of which is illustrated in Figure 5.1. Pressure is applied by a plate to grapes in a basket, either by raising the basket or by lowering the plate via spindles or hydraulics. After a pressing the cake is manually crumbled before further cycles of pressing and crumbling in order to express more juice. Pressures up to approximately 14 bar are used (Mondavi 1965, Ribéreau-Gayon et al. 2006b). The expressed juice flows between the wooden slats that comprise the basket. This juice is relatively low in solids as a consequence of the pomace cake being quite static and because of the gentle manual cake crumblings. Furthermore, extrusion of solids through the basket would be expected to be rather limited given that the direction of plate movement is perpendicular to the screen. The major problems with vertical basket presses are their relatively low capacity and speed and the labour requirements associated with manual press cake crumbling.

Vertical basket presses have been used in large scale operations and older winemaking textbooks (e.g. Fabre 1929, Ventre 1929, Benvegnin et al. 1951, Amerine and Joslyn 1951) show facilities with many spare press baskets on wheels that can be filled while waiting for other baskets to finish being pressed.

#### 5.1.2 Rack and cloth presses

Another older style of press is the rack and cloth press. In this type of press, a cloth is placed over a square frame sitting on a rack. A measured amount of mash is added, and the cloth is folded over to form a "cheese". The frame is removed and a second rack is placed on top of the cheese. The process is repeated until a stack with many cheeses is prepared. The stack is then pressed under a hydraulic ram to express the juice (Cockram 1993). Unlike the basket press, the thin layers of mash in the rack and cloth press result in a relatively short path for juice to escape. Unlike all the other batch presses discussed in this section, the cake is not explicitly crumbled, however, stages at different pressure may be employed.

Rack and cloth presses are highly labour intensive and require thorough washing of racks and cloths for sanitary reasons (Crowe 1970). Rack and cloth presses have primarily been used for fruit juice

expression, apple juice in particular, but they have been employed for wine production to a limited extent (Amerine and Cruess 1960, Troost 1972).

In larger facilities, s everal s tations would be a vailable s o that new stacks could be prepared for pressing while another one was being pressed, as illustrated in Figure 5.2.

NOTE:

This figure is included on page 60 of the print copy of the thesis held in the University of Adelaide Library.

Figure 5.1: Vertical basket press (From: d'Arenberg wines 2010) NOTE: This figure is included on page 61 of the print copy of the thesis held in the University of Adelaide Library.

Figure 5.2: Rack and cloth press (From: Christmann c. 2010)

#### 5.1.3 Horizontal plate presses

An important adaptation of the vertical basket press was the horizontal plate press. These devices feature c ompression plates t hat adv ance f rom ei ther one or both e nds of a horizontal cylindrical basket. Plates may be driven on internal (Figure 5.3) or external (Figure 5.4) mechanical spindles or hydraulically (Figure 5.5). A key advantage of the horizontal plate press over the traditional vertical basket press was the automation of cake crumbling. This was achieved by cage rotation and was also further t ypically aided b y internal plastic r opes (Figure 5.6) or rings a nd c hains (Figure 5.7) that helped to break the cake apart as the plates were retracted. The internal plastic ropes or ring-and-chain systems may also have helped to maintain juice channels in the cake to some limited extent during compression.

A widely used style of horizontal plate press was the Vaslin press. Commercial sized models generally featured two plates on an internal spindle. Half of the spindle was threaded in one direction to ac commodate one of the plates and the other half of the spindle was threaded in the op posite direction to ac commodate the other plate. The plates moved on guider ails on the cage. This arrangement meant that with the spindle held still, rotation of the cage in one direction would cause the plates to move together while rotation in the other direction would cause them to move apart. The cage could typically be rotated at more than one speed. The general operation of this style of press with rings and chains to aid crumbling is illustrated in Figure 5.8. The twin threaded spindle with twin plates has the ad vantage of faster convergence/divergence of pressing surfaces than models with only one plate on a single threaded spindle. Despite this advantage, with the typical pitch on the screw thread, many cage rotations were still required for significant plate movement. This could cause shearing to the pom ace during crumbling and excessive solids levels. An ap parent ad vantage of hydraulically operated horizontal plate presses was that the movement of the plate was independent

of the rotation of the cage. To decouple the plate movement and basket rotation in the mechanical spindle press and allow plate movement with fewer basket rotations, some models of Vaslin press (particularly the larger models) featured a spindle that could be rotated in the opposite direction.

An example of the pressure profile during pressing of whole clusters of Sauvignon Blanc grapes is presented in Figure 5.9 with a manual press programme in a 2.2 m<sup>3</sup> Vaslin press with a rotating spindle.

Apart from facilitating cake crumbling, cage rotation can be desirable during filling as it allows for improved immediate drainage of juice from crushed grapes and for greater quantities of crushed grapes to be loaded into the press. Loading of some models of horizontal plate presses was through large doors on the press cage, thereby preventing rotation of the cage, without first closing the doors. In other presses, the press cage could be advantageously rotated without cessation of filling. An early press with one moving compression plate was able to be loaded axially through the opposite end of the press, allowing rotation and draining during filling (Böhringer and Stührk 1953). This arrangement would apparently not be so easily realised in presses with two moving compression plates. Later models of presses with two moving plates, however, were fitted with two annular ports (Figure 5.10) that could remain stationary while the rest of the cage rotates. The ports could be placed upwards for filling or downwards for emptying (Vaslin 1976a). Subsequent models such as that shown in Figure 5.11 featured one annular door for filling, but were slightly tilted. This would seem to aid distribution during filling and emptying. The general modes of operation are illustrated in Figure 5.12.

Moving plate presses have featured varying degrees of automation: from manual control to selfoptimising cycles based on pressure measurements at the plates. For example, in automated Vaslin CEP presses, plate movement is stopped when a set pressure level is reached. The next mode of operation is then determined from the measured decrease in pressure over a given period while the plates are stationary, which is indicative of the juice flow rate (Bonnet 1984). If the pressure decrease is sufficiently rapid the plates are tightened again at an appropriate speed, otherwise the system transitions to a higher pressure level, or crumbling is performed (Cuénat et al. 1986). The specific operation of moving plate press has a large influence on the quality of juice obtained. Ribéreau-Gayon et al. (2006b) reports that moving plate presses have often been incorrectly used to press more quickly than they should be, producing juices of inferior quality with high suspended solids.

Allowing for some rotation to give improved draining during filling as well as the automation of press programmes are technological advances that would seem to have made these styles of press increasingly appropriate for larger operations. However, it is notable that the largest presses produced by Vaslin only had a cage volume of 12.5 m<sup>3</sup> (Vaslin 1976b, Vaslin 1989). Looking at the mode of pressing, it is apparent that there would be a tendency for the core of the cake to be wetter than near the cage or plate surfaces, as illustrated generally in Figure 5.13, and one would expect this effect to be exacerbated with increasing cage size. In one patent (Constructions Meca-Metalliques

Chalonnaises 1956) the makers of the Vaslin press actually state that "experience shows that this arrangement does not enable uniform pressure to be applied to the interior of the compressed product, and this pressure, which is particularly great near the plates, decreases progressively from the plates towards the median plane of the space there between and from the periphery of the screen towards the centre". Crumbling would tend to redistribute the differentially expressed cake radially in preparation for the next compression, however, one would expect the axial redistribution would be rather poor even with the ring and chain system in place and the cake near the plate surfaces would have disproportionately more juice expressed from it. The introduction of the sloped plate press may have partially improved the axial redistribution. These considerations would tend to limit the size that this style of press could be constructed at and still be effective without large numbers of crumblings or very high pressures. Inspection of the maximum pressures used in Vaslin CEP presses shows that presses with cage volumes of 1.8, 3.0, 4.0 and 8.0 m<sup>3</sup> had maximum pressures of 6, 8, 10 and 12 bar, r espectively. T he a pparent increased pr essure r equirements to achieve s ufficient j uice expression in larger presses, corresponds somewhat with comments by Ribéreau-Gayon et al. (2006b) that there are basket-related mechanical constraints with larger presses.

NOTE: This figure is included on page 63 of the print copy of the thesis held in the University of Adelaide Library.

Figure 5.3: Vaslin internal spindle press with two moving compression plates (From: Seltz 1958)

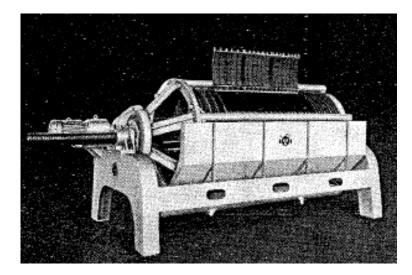


Figure 5.4: Willmes WHA external spindle press with one moving compression plate (From: Stollenwerk 1962)

NOTE: This figure is included on page 64 of the print copy of the thesis held in the University of Adelaide Library.

Figure 5.5: Bucher hydraulic press with one moving compression plate (From: Crettenand et al. 1969)

NOTE: This figure is included on page 65 of the print copy of the thesis held in the University of Adelaide Library.

Figure 5.6: Ropes in a Willmes ABC press (From: Troost and Fetter 1964)



Figure 5.7: Rings and chains in a Vaslin 3.2 m<sup>3</sup> press (From: Agriaffaires.co.uk 2010)

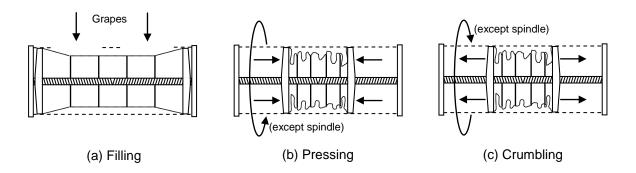


Figure 5.8: Operation of Vaslin-style internal spindle press with two moving compression plates and a ring and chain system

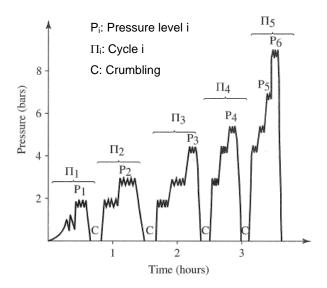


Figure 5.9: Pressure profile for pressing of whole clusters of Sauvignon Blanc grapes in manual mode with a Vaslin 22 VT internal spindle press with two moving compression plates and rotating spindle

(From: Ribéreau-Gayon et al. 2006b)

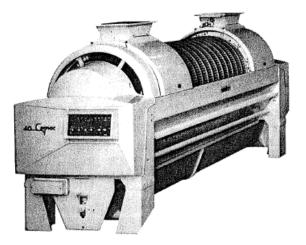


Figure 5.10: Vaslin press with annular doors (From: Vintec 1980)

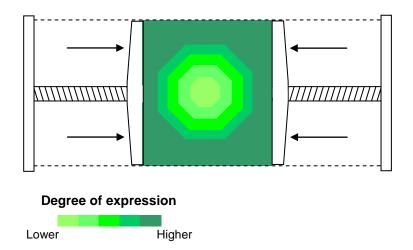


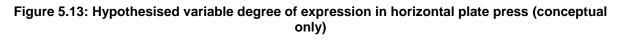


Figure 5.11: Vaslin CEP 250S press (From: Interempresas.net 2010)

NOTE: This figure is included on page 67 of the print copy of the thesis held in the University of Adelaide Library.

Figure 5.12: Vaslin CEP press operation (Adapted from: Officina Meccanica BEG 2010)





#### **5.1.4 Pneumatic presses**

The predominant type of batch press now in use in the wine industry is the pneumatic press.

The original pneumatic grape press was introduced in 1951 by Willmes (Troost 1972, Link 1996) and this is shown in Figure 5.14. This press featured a centrally mounted rubber bladder that was inflated by an air compressor to press the pomace against the circumference of the slotted stainless steel drum. Cake crumblings were performed by tank rotation after bladder deflation. The maximum pressure employed was approximately 6 bar and the pressing and crumbling cycle was typically repeated 6-8 times (Böhringer and Stührk 1953). The axial arrangement of the bladder resulted in a relatively thin circumferential layer of mash and short juice path being formed between the bladder and the screen. This in contrast to the relatively long juice path in vertical basket presses and horizontal moving plate presses. Crumbling with this press was however inefficient (Haushofer 1981, Trogus 1993). After several cycles, the door would sometimes have to be opened to manually scrape off the pomace adhering to the drum in order to facilitate crumbling of the cake by rotation (Böhringer and Stührk 1953). Pressing above approximately 2 bar in the initial phases also risked extruding pomace through the drum slots (Böhringer and Stührk 1953). It is notable that unlike the vertical basket press or horizontal moving plate press, the direction of movement of the compressing surface, the rubber bladder, is the same as that of juice exit through the screen, which would seem likely to facilitate solids extrusion through the screen. The rubber bladders were also prone to become brittle over time (Troost 1972) and were susceptible to damage (Haushofer 1981, Trogus 1993).

In 1974, Willmes introduced the pneumatic membrane tank press (Petgen 2002). Variants of this device have now become an industry standard for batch pressing, and in addition to Willmes are manufactured by many other companies (e.g. Bucher-Vaslin, Diemme, Pera, Velo, Scharfenberger, Miller, Della Toffola). These devices apply pressure by inflation of an essentially non-elastic membrane, now typically polyester reinforced polyurethane (Covertex 2010). The relatively non-elastic membranes are likely much more resilient than the rubber bladders in the original Willmes presses. The pressures applied are also lower, with a typical maximum of 2 bar and the majority of juice being expressed at much lower pressures. This appears to be another advantage as there would be a reduced tendency to extrude pomace through the screens when compared with the original Willmes press where higher pressures were employed. In the membrane press, crumbling is performed by deflating and retracting the membrane by vacuum, followed by drum rotations. The most common arrangement of membrane press is to have the membrane attached to the wall of one half of the tank with slotted drainage channels attached to the opposite wall (Figures 5.15 and 5.16). Juice passes into these drainage channels and then flows down them to one or both ends of the tank where it is discharged.

The raised drainage channels shown in Figure 5.16 from a Bucher-Vaslin membrane press have a triangular profile that may allow for mash to slide down their sides self-cleaning the slots to some extent. Additionally, the slots run in the direction of the circumference of the tank - a feature that

would seem to also promote self-cleaning of slots as the tank is rotated. In some models of membrane presses, the slots themselves increase in size from the main press compartment side to the inside of the drainage channel to further limit slot blinding.

To further aid drainage, Bucher-Vaslin membrane presses can be fitted with flexible elements that protrude into the mash reducing the exit distance for expelled juice. As shown in Figure 5.17, these elements have external grooves down which juice can run towards the drainage channels. However, with these elements fitted, the membrane may be damaged if repeatedly inflated to the extent that it pushes against them. Thus when these flexible drainage elements are fitted to the press, the press must be sufficiently full (Bucher-Vaslin 2007) and the automatic washing cycle cannot be performed as it includes low pressure inflation of the membrane (Samuel Plummejau, Bucher-Vaslin, personal communication, January 2010).

An alternative to having raised internal drainage channels on the inside of the press tank is to have the wall of the press tank slotted. This can provide greater screen surface area for draining and pressing, however, it may also allow greater oxidation and limits the possibility of using the tank for maceration. Even in a normal "closed" press with internal juice channels, complete removal of oxygen is not achieved as prior to each crumbling the membrane is retracted by a vacuum to the starting position, thereby drawing air through the juice outlets into the tank. Consequently manufacturers have introduced systems where nitrogen is introduced through the juice outlets as the membrane is retracted. The inert gas is sometimes recycled into an inflatable bag to minimise operating costs associated with using fresh inert gas. These inert gas systems are relatively new and at this stage it is unclear whether there is real economic merit in this additional protection from oxygen; for many grape varieties at least. Schandelmaier (2006) postulates that it is unlikely that this technology will prevail in Germany as experiences have shown that moderate must oxidation is usually beneficial in terms of subsequent clarification by flotation.

The side membrane press configuration discussed so far, is not universal, and some manufacturers have adopted quite different membrane and screen arrangements. For example, Willmes have a central juice collection system on their newer presses, where the membrane coats most of the interior of the drum and juice is drained through vertical stainless steel juice channels (Figure 5.18). For smaller presses, these channels can be easily removed from the outside of the press for cleaning; however, they were cumbersome to remove for larger presses with corresponding larger juice channels. This led to the development of flexible polyester drainage screens with  $1.5 \times 1.5$  mm square holes supported by a stainless steel spiral as illustrated in Figure 5.19 (Gann 2006). Another alternative membrane/screen arrangement is to have the membrane mounted centrally, in a similar manner to the original Willmes rubber bladder presses. These devices typically feature a central star axle, which appears to assist in the mounting of an essentially non-elastic membrane of sufficient inflated circumference. The different pneumatic press arrangements discussed are illustrated in Figure 5.20. Schematics with the diaphragm retracted in the filling position and inflated during

compression are presented. For an equivalent yield it is very difficult to know which arrangement is optimal and to the author's knowledge there are no unambiguous studies proving the merits of one design relative to another. One may compare these specific configurations as well as different press sizes to some extent on the basis of the cake thickness between the diaphragm and the outflow screen. For an individual compression step, the thicker the cake the lower the solids content is likely to be, however the lower the yield of juice expressed is also likely to be. To get the same yield as a press with a thinner cake, more cake crumblings are going to be required, which in turn may generate solids by shearing. As a result of these counteracting effects it is difficult to make an absolute comparison without extensive trials.

Membrane presses typically utilise air pressure for inflation, however some models of Siprem presses use a vacuum system. Diemme, Siprem and Marzola have also previously offered water-operated versions of their presses (Anon 1986) but from inspection of their respective websites they do not appear to sell these models anymore. Water-operated systems had the advantage of using a water pump instead of a more expensive air compressor; however, a membrane failure could allow water into the pomace (Anon 1986). Furthermore, given that the inflating fluid was a relatively incompressible liquid rather than a gas, one may expect that the level of pressure control may have been inferior to an air operated system.

To facilitate cleaning, some presses feature juice channels with quick releases that allow for their removal and cleaning without entry into the press tank. Some presses also feature automatic washing devices that utilise high pressure water to clean the channels.

The specific operation of a membrane press can significantly influence juice yield, quality and broadly be categorised "standard" throughput. Press programmes can as either or "sequential/cremant" programmes. In standard programmes there is only one pressure set point for each cycle as illustrated in Figure 5.21. In contrast, sequential programmes can involve pressure steps during each cycle as shown in Figure 5.22 (Freund et al. 2008). Programme parameters (i.e. pressures and hold times) are pre-set by the user, together with the specifications for crumbling such as the number of tank rotations at different points in the programme. Additionally, settings for filling, draining and emptying are required. Sequential programmes can be gentler than standard programmes because fewer cycles and thus fewer crumblings that may cause mechanical damage to the grapes are generally required (Freund et al. 2008). Apart from programmes that are completely pre-set by the user, self-optimising programmes are also available with many brands of membrane press. These programmes typically employ continuous assessment of juice flow rate together with proprietary heuristics to optimise the press programme with respect to juice yield and pressing duration (Tong 2001, Schandelmaier 2006, Freund et al. 2008). Juice quality may also benefit as potentially damaging operations such as cake crumbling, and the adjustment to a higher pressure level need only be commenced as dictated by the specific batch properties (Trogus 1993). Operator

knowledge and monitoring requirements are also reduced. Ultimately, the success of a particular manufacturer's programme will depend on what actions they take based on the flow rate information.

Membrane presses can be filled through their doors, however, axial filling, whereby mash is pumped into the press through a pipe at the axis, is common in larger facilities. The axial filling inlet is shown from the inside of one membrane press in Figure 5.23. Axial filling allows for drum rotation and increased drainage while filling. Consequently, larger quantities can be loaded per tank volume. However, axial loading can result in higher juice suspended solids, particularly when the tank is rotated more frequently (Maul 1987, Seckler et al. 2008, Seckler et al. 2009). The increased capacity from increased rotations needs to be appropriately balanced against increases in solids content.

Membrane presses are now extensively employed in the wine industry and are widely acknowledged as being capable of producing high yields of juice with relatively low solids and phenolics contents. While it is difficult to comment on overall trends in the wine industry because of the wide range of techniques used, it does seem from the recent dominance of membrane press advertisements in English language trade journals (Wines and Vines and Practical Winery and Vineyard from the USA, and Australian and New Zealand Grapegrower and Winemaker and Australian and New Zealand Wine Industry Journal from Australia and New Zealand) and the author's personal observations at some large wineries, that membrane presses may increasingly be being used for draining as well as pressing. In some cases, membrane presses are even employed principally as a drainer with a short pressing cycle, with screw presses being used for expression of the last fraction of juice. Similarly the Pera Elite Tank drainer, which is essentially a membrane press that doesn't rotate, is anecdotally being used in this manner in some instances overseas.

There are evidently not the same restrictions on size in membrane press construction as there are with horizontal plate presses as models with volumes as large as 75 m<sup>3</sup> are available (Bucher-Vaslin 2010a), making them increasingly attractive for larger wineries. In terms of scaling up, one major apparent advantage of the membrane press arrangement over the horizontal plate press is that pressure is applied along the whole length of the press drum, so longer presses can be used without very high pressures at each end. Furthermore, the more even expression along the length of the drum than with a horizontal plate press means that axial mixing is less of an issue. Spiral elements behind the membranes in some presses designed to aid emptying by conveying pomace to one or more exit doors, such as those shown in Figure 5.24, may also aid axial mixing during tank rotation. Despite these apparent advantages over horizontal plate presses with regards to scaling to sizes more appropriate for large wineries, there are still some issues with obtaining as even an expression as that possible in smaller presses. For example, if a press is filled axially with simultaneous juice draining, and the filling process takes a significant period of time, which it will for a large press, the first grapes that were loaded into the press will have had significantly more juice expressed from them than has been expressed from the grapes loaded later in the filling of the press. This is a result of them being subjected to more tank rotations and time in the chamber under the compression of the

grapes on top of them. This effect will start to influence the degree of fractional expression from the entire load of grapes.

Despite the significant improvements in pneumatic presses in recent decades, they are a still a batch operation that takes several hours. The larger volume presses appropriate for large wineries have a large hold-up volume and a large footprint, and still have much lower throughputs than continuous inclined drainer and screw press lines.

NOTE: This figure is included on page 72 of the print copy of the thesis held in the University of Adelaide Library.

#### (From: Huntsinger 1956) Figure 5.14: Willmes pneumatic press



Figure 5.15: Internal view of a Bucher-Vaslin XP320 membrane press (membrane retracted)



Figure 5.16: Drainage channels in a Bucher-Vaslin XP320 membrane press



Figure 5.17: Three-dimensional drainage system for Bucher-(Vaslin) presses (From: Bucher-Vaslin 2010b)



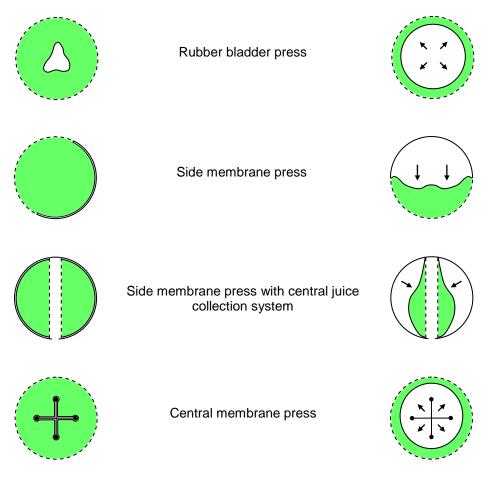
Figure 5.18: Vertical central stainless steel juice channels in a Willmes press (From: Willmes 2010a)



Figure 5.19: Vertical central flexi-drain juice channels in a Willmes press (From: Scott Laboratories 2010)

#### (a) Diaphragm retracted

(b) Compression



- ----- : Screens (commonly an internal channel drainage system for side membrane presses)
  - : Grapes

(Doors not shown, membrane sizes approximate only)

# Figure 5.20: Generalised pneumatic press arrangements (Adapted from: Lemperle and Kerner 1978, Willmes 2010b, KVT 2010)

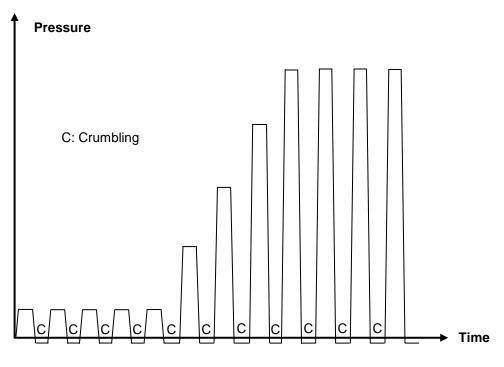


Figure 5.21: Standard press programme (conceptual only) (Adapted from: Vialla 1989)

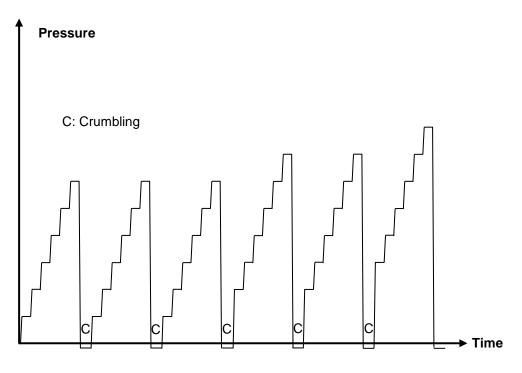


Figure 5.22: Sequential press programme (conceptual only) (Adapted from: Bucher-Vaslin 2007)



Figure 5.23: Internal view of a Bucher-Vaslin XP320 membrane press showing axial filling inlet (membrane retracted)



Figure 5.24: Spiral elements set under retracted press membrane to convey pressed pomace to exit door(s) during emptying by tank rotation

(From: Bucher-Vaslin 2010b)

# 5.2 Continuous presses

#### 5.2.1 Screw presses

The screw press is the most common type of continuous press employed in wine production. Its key advantage over batch presses is throughput, with presses capable of processing up to approximately 50 tonnes/hour available (on an initial crushed grape mass basis, with pre-draining).

Screw presses have been used in wine production as early as 1900 (Benvegnin et al. 1951, Garolla 1956). Similar in design to the inclined drainer, the screw press is essentially a screw conveyor running inside a perforated cylinder, with the discharge end obstructed by a door or cone. Generally in models designed for wine production, they have been horizontal with a relatively constant diameter and with the screw finishing well before the discharge end of the chamber to allow a thick core of marc to form at the end.

Some means of preventing the pressed pomace returning from zones at higher pressure back towards the feed hopper is required. Backflow is undesirable as it can reduce throughput and result in extra shearing and juice solids. Backflow was seemingly limited in some early model screw presses by the use of twin opposite thread screws rotating in opposite directions, arranged either side-by-side (Nord 1962, Troost 1961) or end on end (Benvegnin et al. 1951, Troost 1961) such as the Colin screw press presented in Figure 5.25. Juice from early high speed twin screw presses produced very poor quality wine and the use of single screw presses was reportedly a major improvement (Agostini 1965). For the more common single screw press, specific anti-return systems (obturators) are required. In early single-screw presses, these devices were traditionally one or more toothed wheels with a plane of rotation perpendicular to that of the screw, such that one tooth of the wheel engaged between two ridges of the screw thread (Dessoris 1973). This concept is illustrated in Figure 5.26 and a schematic of a screw press featuring an anti-return wheel at the end of the feed hopper is presented in Figure 5.27. This was evidently a common system, as illustrations in old advertisements and textbooks for many brands of screw press (e.g. Garolla, Coq, Pera, Marzola, Mabille, Diemme, López Romero, Lorsa, PMH) feature external partial circular protrusions at the end of their hoppers that likely conceal these wheels. Some examples are presented in Figure 5.28. Unfortunately with this system, some matter would still return towards the hopper as the teeth could not extend to the screw axle in order to allow rotation of the anti-return wheel (Dessoris 1973, Sperling 1971). Furthermore, friction would result in wear on the screw and the wheel teeth. The anti-return wheels would also grind the pomace and their covers were prone to fill with fermenting and rotting grapes, reducing juice and wine quality (Sperling 1971).

The static 'Bi-valve' was an alternative anti-return device introduced by Coq in approximately 1970 (Sperling 1971). An excerpt from an advertisement demonstrating its operation is presented in Figure 5.29. The Bi-valve is a disc with two openings. When mash has been conveyed through one of the Bi-valve openings by the escape rim at the end of the first section of the screw, it is caught by the

leading rim at the start of the second section of the screw, limiting return of matter towards the hopper (Dessoris 1973), even with slippery grapes (Coq 1971). One of the radial edges on each part of the Bi-valve is bevelled (on the hopper side) meaning that any small hard contaminants in the feed (such as metal objects) pass through the Bi-valve without causing damage (Dessoris 1973). Additionally, the Bi-valve provides outboard support for the screw shaft, reducing the potential for contact between the screw and the screens and therefore, the grinding of pomace caught in between (Sperling 1971, Dessoris 1973). The reduced friction when compared with the anti-return wheel systems also reportedly resulted in reduced electricity requirements (Sperling 1971). The Bi-valve also provided a more definitive break point for different juice quality fractions, much more so than with the toothed anti-return wheels (Sperling 1971).

Another method to prevent return from the compression zone is to have a section of screw without thread in conjunction with a fixed bar between a sleeve on the shaft and the screen wall (Pera 1977). A variant of this arrangement is apparently used on current Marzola screw presses, of which many are installed in Australia (Agricultural and General Engineering 2006). Several Marzola PAP1000 screw presses were photographed in varying degrees of disassembly at one winery in order to better understand screw press construction. These presses were not fitted with the outside covers shown on the manufacturer's website (Figure 5.30). Instead the screens were simply draped with plastic sheeting during vintage to deflect any squirting juice. As with the Coq Bi-valve screw press, the screw comprised two sections. The first screw section (Figure 5.31) and the second screw section (Figure 5.32) were separated by the anti-return device (Figure 5.33) that was mounted in a fixed position on the press frame. Figure 5.34 depicts the press with the first screw section and the anti-return device in place but with the second screw section and latter screens and discharge section disassembled. In addition to this anti-return device, there is also a small fixed interrupter bar at the end of the hopper, as shown in Figure 5.35. A cut out section in the first section of the screw (see Figure 5.31) allows the screw to rotate without touching the protruding interrupter bar. Visual observations during operation, suggest that this device may aid the feeding of the pomace from the hopper into the actual compression cylinder, by keeping the pomace from rising too high up the left hand side of the hopper so it can easily be picked up by the screw and be fed into the cylinder, as well as acting to scrape excess pomace off the screw that might otherwise be ground between the edge of the screw and the edge of the compression cylinder entrance. With this press, the hopper screens feature 2 mm  $\times$  20 mm slots, whilst the remaining press screens incorporate 2 mm  $\times$  50 mm openings, with a very smooth internal surface, and apertures that taper inwards, as discussed previously a profile that should tend to prevent blinding (Figure 5.36).

The single screw presses described so far effectively have two sections of screw separated by an anti-return device, defining a feeding/draining section and a compression section. An alternative approach is to use a screw with many interruptions in flight. This paradigm was employed in the Rietz/Vincent screw press presented in Figure 5.37. These devices also featured stationary interrupter bars. Rietz/Vincent screw presses were at one point used by a number of Californian

wineries (Rietz 1971). Unfortunately, there are no definitive reports to the author's knowledge on the relative success of this style of interrupted flight design press compared to the more common wine industry designs previously discussed.

Some models of screw press have allowed for the regulation of the marc plug length by axial displacement of the screw. Notably, the anti-return Bi-valve in Figure 5.29 slides along grooves in the cage, allowing it to move backwards and forwards together with the screw. An adaptation of the screw press further utilising this axial displacement was the impulse screw press. The impulse screw press had a similar operation to that of the horizontal plate press in that either the whole screw (or in some models only the part of the screw beyond the anti-return device) would be intermittently driven forward without rotation. This type of screw press achieved results in between batch and standard continuous screw presses, both in terms of juice quality and throughput. This intermittent axial displacement of the screw was apparently superior at handling slippery grapes, which could be problematic with traditional screw presses (J.B. McMahon 1979, Vintec 1981, Anon 1986). Another system to further aid the handling of slippery grapes was the ratchet drive system used in some Coq presses. This provided a somewhat intermittent rotation, suggested to provide a better grip on the pomace, when compared with continuous rotation (J.B. McMahon 1979, Vintec 1981). While impulse screw presses were apparently popular in Australia in the 1980s (Anon 1986), inspection of press manufacturer websites suggests that they are no longer actively sold. It seems likely that their place in the market was taken by large volume pneumatic membrane presses.

Previous reported trials with continuous screw presses suggest that some juice fractions collected from screw presses can be of lower quality than from batch presses, with higher and more difficult to remove solids levels and higher phenolic contents (Maurer and Meidinger 1976, Lemperle and Kerner 1978). This is supported by overviews in several wine production textbooks (Troost 1972, Boulton et al. 1996, Ribéreau-Gayon et al. 2006b). Generally, modern screw presses that feature larger diameters and lower speeds and improved anti-return devices are reported to give superior results to those obtained with earlier models (Agostini 1965, Foulonneau 1972, Trogus 1974, Haushofer and Meier 1976, Erczhegyi and Mercz 1975, Maurer and Meidinger 1976). Furthermore, it has been claimed that with increasing cage diameter and reduced speed, the juice quality produced by a screw press approaches that of a batch press (Lemperle and Kerner 1978, Haushofer and Meier 1976). It is understandable that quality is improved with larger diameters and slower screw speeds, as shearing actions generating solids and phenolics and the speed of break up the cake are reduced, however, the mode of operation of a screw press is quite distinct. For batch presses, operation generally consists of a series of repeated cycles of pressing and crumbling, while the mode of operation for screw presses would seem to be a simultaneous pressing and crumbling, as the screw rotation continually shears the cake. Given this shearing action, it is not surprising that screw presses typically achieve a greater total yield of juice than other available presses (Haushofer and Meier 1976, Lemperle and Kerner 1978), but with higher levels of solids and phenolics.

Despite the quality-related problems, the screw press configuration is a very convenient one. High throughputs can be processed and material is simply loaded into the hopper with juice and dry marc then being produced with little operator intervention. In this review of different designs of screw press and their e volution it is apparent that the grouping of alls crew presses together as necessarily producing low quality wines may be overly simplistic. It is possible that the quality level obtained with current designs of s crew press may b e f urther i mproved upon. The use of different s crew configurations and multiple anti-return devices to try and mimic the multiple stages of essentially non-shearing compression and crumbling of batch membrane presses should be investigated further.

NOTE: This figure is included on page 81 of the print copy of the thesis held in the University of Adelaide Library.

Figure 5.25: Colin screw press with twin end on end counter-rotating screws (Adapted from: Troost 1972)

#### NOTE:

This figure is included on page 81 of the print copy of the thesis held in the University of Adelaide Library.

Figure 5.26: Screw press toothed obturator wheel (From: Établissements Coq 1971)

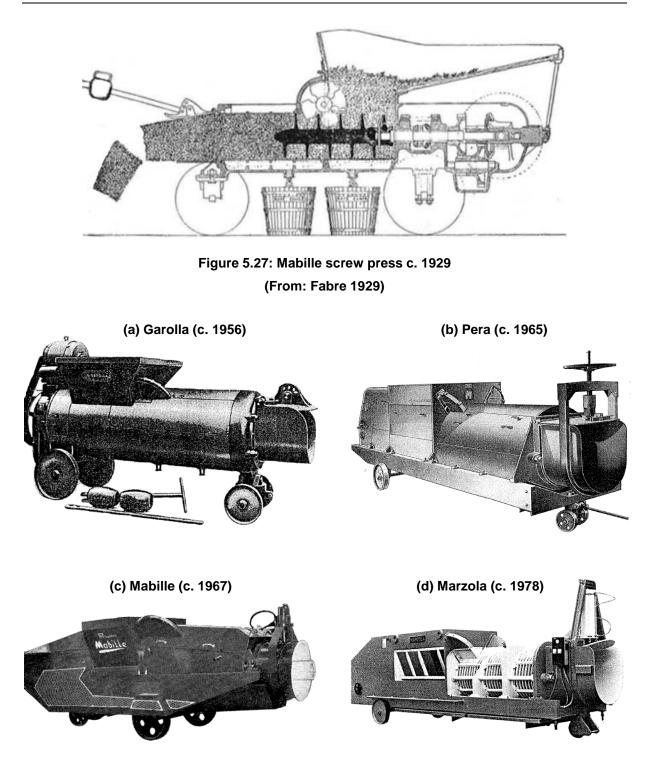


Figure 5.28: Selected screw presses (partial circular protrusions at the end of the hopper, likely conceal toothed anti-return wheels, not at same scale)

(Adapted from: Garolla 1956, Columbit 1965, Mabille 1967, Marzola 1978)

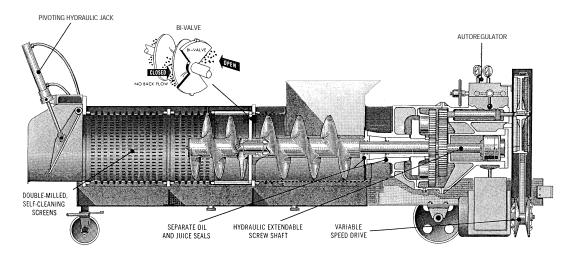


Figure 5.29: Coq Bi-valve continuous screw press (Adapted from: Coq 1971)



Figure 5.30: Marzola PAP100 continuous screw press (From: Marzola 2010)



Figure 5.31: First screw section of Marzola PAP1000 continuous screw press



Figure 5.32: Second screw section of Marzola PAP1000 continuous screw press



Figure 5.33: Anti-return device of Marzola PAP1000 continuous screw press



Figure 5.34: Partially assembled Marzola PAP1000 continuous screw press with anti-return device installed

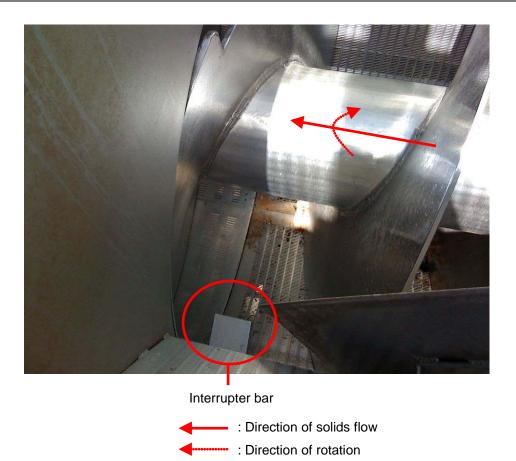


Figure 5.35: Overhead view of a hopper of an empty Marzola PAP1000 continuous screw press

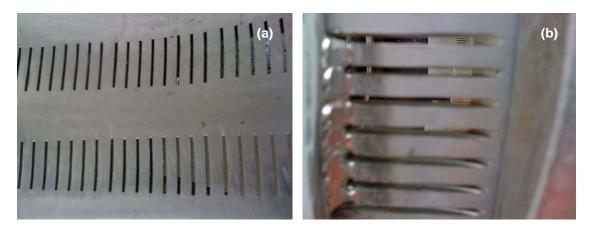


Figure 5.36: Marzola PAP1000 continuous screw press screens viewed from (a) inside and (b) outside the chamber

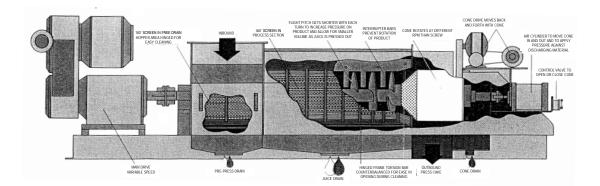


Figure 5.37: Rietz/Vincent Cushion Cone continuous screw press (Adapted from: Rietz 1971)

#### 5.2.2 Belt presses

Continuous belt presses from several different manufacturers (e.g. Sernagiotto, Proipafisa, Diemme) have been used to a much more limited extent in wine production than screw presses. Typically in these devices grapes are transported onto a preliminary draining area, and then pass to a compression zone, where two perforated belts are flattened progressively between a series of rollers (Darias-Martin et al. 2004). A schematic of a belt press is presented in Figure 5.38. This particular continuous machine could process up to 30 tonnes/hr of machine harvested grapes with a residence time of less than 2 minutes (Sernagiotto 1986). Wines were reportedly low in phenolics, but very high solids are produced (Gúrpide Ibbarola 1989). This is a common complaint of belt presses (Haushofer 1981, Vialla 1989, Darias-Martin et al. 2004). In addition, the belts can be difficult to clean (Haushofer 1981, Vannobel et al. 1987) and are susceptible to damage by solid objects in the feed (Gúrpide Ibbarola 1989).

NOTE: This figure is included on page 88 of the print copy of the thesis held in the University of Adelaide Library.

Figure 5.38: Sernagiotto NOLM belt press (Adapted from: Gúrpide Ibbarola 1989)

#### 5.2.3 Pneumatic presses

While pneumatic presses have generally been batch machines, there have been attempts to adapt them into continuous devices in order to improve throughput and make them more appropriate for larger facilities, while still endeavouring to achieve the quality of a batch press.

An early semi-continuous pneumatic press was the MacKenzie p neumatic press (Figure 5.39). It employed intermittently operating perforated stainless steel conveyor belts, but used rubber bladders for application of pressure. In this device the belts advanced then stopped, the section doors would close and the bladders were i nflated t o 1 bar (Anon 19 60). After a per iod o f c ompression, the compressed air was released, the bladders were drawn up by vacuum and the pomace was conveyed to the next pressing c ompartment w here the cycle was repeated. In bet ween pressing stations the pomace was loosened by rotating claws. The MacKenzie pneumatic press was capable of processing approximately 40 tonnes/hr, with a residence time of 12 minutes (excluding prior static draining). The manufacturer reported that while the layer of pomace was thin (approximately 20 cm) this was still thick enough to remain a filter bed and thus limit juice solids content (MacKenzie 1968). This press was invented in South Africa where there were several installations, and there was also one of these presses installed at Orlando Winery in Australia in 1965 (Rankine 1996). Later models featured all pressing stations on one level and used only one perforated stainless steel belt (MacKenzie 1968). There is little information available on these devices. The lack of mention in the literature for many decades suggests they have not been in use for a long time. Potential reasons

may have been susceptibility of rubber bladders to damage, larger footprint and lower yields than continuous screw presses, in which major advances had been made around the same time.

Much more recently, a different configuration of semi-continuous pneumatic press (Figure 5.40) has been introduced by Siprem. The manufacturer reports that the largest model is capable of processing 32-45 tonnes/hr. It consists of a cylindrical press tank, rotatable about its ax is and partitioned into several chambers. The first chamber features a 360° screen, while subsequent chambers are fitted with their own press membranes. Operation in each chamber is similar to that of a normal membrane press. Tank rotation allows transportation past the dividing partition to the subsequent chamber. After the final chamber, marc is dumped. Different juice yield fractions are collected at points along the tank (Anon 2006). As this is a relatively new device there is little information available on performance. There are currently no installations in Australia.

NOTE: This figure is included on page 89 of the print copy of the thesis held in the University of Adelaide Library.

Figure 5.39: MacKenzie pneumatic press (From: Anon 1960)



Figure 5.40: Siprem continuous membrane press (From: Siprem 2010)

#### 5.2.4 Decanter centrifuges

The use of continuous decanter centrifuges (Figure 5.41) as press substitutes has been investigated in several trials (Mäuser and Hamatschek 1993, Hamatschek et al. 1995, Dörr and Hühn 2001, Dörr and Hühn 2002, Pecoroni and Schauz 2004, Hühn et al. 2007). The mash passes through a feed tube into the rotating decanter bowl, where solids collect on the bowl wall as a result of centrifugal force. These solids are transported by a scroll rotating at a slightly faster speed than the bowl, towards the conical end of the bowl where they are further dried before being ejected through apertures. The juice flows between the flights of the scroll in the opposite direction to the outlet point (Hamatschek et al. 1995, GEA Westfalia Separator 2010). One benefit of the decanter centrifuge is its small footprint, which has led to trials where the decanter has been mounted directly on a machine harvester with a destemmer for immediate vineyard juicing of white grapes (Hühn et al. 2007). An apparent major disadvantage with the use of decanters is that while they can produce juice with lower spin test solids than membrane presses, decanter juice can have high levels of fine suspended solids, even more so than that produced with screw presses (Dörr and Hühn 2001, Dörr and Hühn 2002, Hühn et al. 2007).

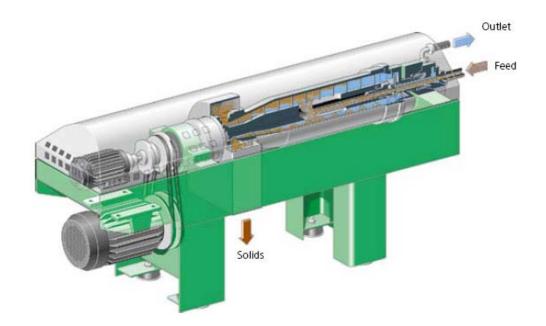


Figure 5.41: Westfalia decanter centrifuge (From: GEA Westfalia Separator 2010)

# 5.3 Conclusions

A variety of batch and continuous equipment have been used for pressing. Pneumatic batch membrane presses are now the predominant batch press and are widely acknowledged to produce high yields of high quality juice low in solids and phenolics. They are still a batch operation with a relatively low throughput. Screw presses are the dominant continuous press, with much higher throughputs than any batch press. However, they are associated with juice fractions with high phenolics and solids contents. There remain opportunities for improvements in continuous press design. The use of different styles of screw press that endeavour to mimic the multiple stages of compression of a batch press should be further investigated. Economic considerations in the choice of batch or continuous equipment and in the operation of membrane presses will be discussed further in the next chapter.

# CHAPTER 6: ECONOMIC CONSIDERATIONS IN JUICE EXPRESSION

Economically, juice expression is a complex trade-off between quality and productivity that will vary between wine companies depending on their range and grades of products and other specific circumstances.

After making some attempts to obtain detailed information on capital and operating costs and juice and wine value from wineries and equipment suppliers it became apparent that detailed economic analyses would be problematic. Capital and operating cost information and juice and wine value at different points in production, of sufficient accuracy could not be obtained. Any detailed economic analysis would need to be underpinned by assumptions that would effectively dictate the results.

It was deemed more useful to look broadly at some key economic considerations in juice expression. Specifically the differences between current batch and continuous equipment and the operation and division of juice fractions when employing batch pneumatic membrane presses. These general discussions can be adapted by individual companies to their specific circumstances.

## 6.1 Batch and continuous equipment

In order to perform an order of magnitude level comparison of capital costs for modern batch and fully continuous expression equipment, indicative prices were obtained from three distributors, who all sold large membrane presses ( $\geq 25 \text{ m}^3$ ), and one of whom sold continuous inclined drainers and screw presses. These were used to derive the comparison presented in Table 6.1. The capital costs for the fully continuous line were approximately half that for the batch membrane press (used for both draining and pressing).

In the consideration of operating costs, newer membrane presses and continuous inclined drainer and screw press lines are both largely automated and when correctly configured both have relatively low labour requirements. However, membrane press operation is still likely to be more labour intensive. For example, consider a winery with a white grape intake of 90 tonnes/hour. Under the conditions described in Table 6.1, three 800 mm diameter continuous lines would be required, which could probably be managed by one dedicated operator. In comparison, for the same throughput using 32 m<sup>3</sup> membrane presses, seven presses would be needed, and two or more operators would likely be required to manage these. Similarly, other operating costs, like energy usage and maintenance would likely be higher for batch pressing than for continuous lines with equivalent capacities as a consequence of the larger number of membrane presses needed to achieve the same rate of production. Membrane presses do have some specific operating cost advantages over the continuous lines. Continuous lines can produce higher absolute solids contents, higher levels of fine solids that are more difficult to remove, and higher levels of other components (eg. pectins) that influence the ease of solids removal, particularly in higher yield pressings fractions. Higher downstream juice processing costs would therefore be expected as a result of the need for additional juice clarification to a level sufficient for quality white wine production. Higher fining costs related to phenolic management are also likely, particularly for heavy pressings fractions.

Despite the above additional associated operating costs, the overall average production cost is still likely to be lower for continuous lines. Nevertheless, the use of continuous inclined drainers and screw presses is apparently in decline due to the perceived lower final quality of the wine produced. The higher average production costs of the membrane press are probably justifiable in many operations, given the growth in demand for premium wines relative to non-premium wines (Anderson 2004). The cost differential between the technologies is probably significantly less than when membrane presses were first introduced in the 1970s. This is a result of larger tank volumes with relatively lower capital costs per unit volume, axial filling allowing greater throughput for the same tank volume (as discussed in section 5.1.4), and lower labour costs per throughput given the relatively fewer filling, emptying and cleaning operations associated with larger tank sizes and axial filling. Additionally, membrane presses are now produced by many manufacturers providing for greater competition.

When purchasing membrane presses, a winery needs to choose between buying a few large presses or several smaller presses to achieve a given capacity. The use of just a few large presses takes advantage of the lower capital costs and labour requirements per unit volume. However, this has to be balanced against scheduling flexibility. To most effectively utilise the large tank volumes, grapes harvested from different vineyards may have to be pressed together. However, if part of the planned press load is late in arriving from the vineyard, there may be unplanned downtime while pressing operations are delayed until the arrival of the remaining grapes. Alternatively, pressing may proceed with a partial load thereby wasting press capacity, with likely increases in average processing cost. There may also be reductions in the degree of fractional expression given probable greater expression from grapes loaded into the press earlier rather than later in the filling cycle as discussed in section 5.1.4.

Equipment	Model	Purchase Price <sup>a</sup>	Capacity <sup>b</sup> (tonnes/hr)	Price/capacity (per tonne/hr)
Batch pneumatic membrane press with compressor	32 m <sup>3</sup> tank volume <sup>°</sup>	A\$180,000	13 (64 tonnes destemmed and crushed grapes, 5 hrs turnaround time)	A\$14,000
Continuous inclined drainer & screw press	800 mm diameter <sup>d</sup>	A\$220,000	30	A\$7,000

<sup>a</sup> Purchase price is highly dependent on exchange rates as these larger presses are typically manufactured in Europe.

<sup>b</sup> Capacity is highly dependent on grape type and condition and press operation. Destemming and crushing has been assumed. It has been assumed that the membrane press is being employed for both draining and pressing, and axial filling is being used with the loading capacity being approximately twice the volume of the press (Trogus 1993).

<sup>c</sup> Press tank volume was chosen based on observation of several presses of this size being used at one large winery.

<sup>d</sup> Diameter was chosen on the basis that this is a common larger size of inclined drainer that can be purchased, and the largest size offered by the manufacturer from which prices were obtained. Screw presses are typically manufactured up to slightly larger sizes, generally for use after static drainers.

#### 6.2 Pneumatic membrane press operation and press cuts

As discussed in section 4.1, a fractional expression of juice is important for high quality white wine production. From literature and practical observation, it appears that as grape grade and value increase, juice is increasingly likely to be separated into two or more fractions, thereby protecting the quality of the earlier juice. The implementation of this processing strategy will no doubt vary between wine companies depending on their product mix. The production of a range of products with different price points would allow for greater flexibility in downgrading and blending. The volumes of these products will also be important; a low grade product may only be able to handle a certain quantity of downgrades from the higher grades before its quality also drops below an acceptable level.

Downgrading and blending practices for different grades of Chardonnay and Riesling grapes at one winery showed that, apart from the lowest grade of grapes, juice was typically split into two fractions: "free-run" and "pressings". Draining was performed in the press itself and axial filling was used to load the press. The free-run fraction consisted of juice expressed during axial filling and draining but also some that was expressed by mild pressing after the press programme had been commenced (the use of "free-run" as a general term to define the high-value juice despite some pressure being used for expression is a convention that was also observed at another large winery). On average, 80% of the Chardonnay juice was kept as the free-run fraction while 20% was diverted as the pressings fraction.

For the Riesling juice, the corresponding split was 75% and 25%. For the Chardonnay grapes, for grades where the free-run juice produced wines retailing for approximately A\$25 or A\$15 (per 750mL bottle), the pressings were downgraded to a product worth only A\$7. In comparison, for grapes where the free-run juice went to the A\$7 product the pressings were downgraded to a A\$2.50 product (per 750mL, derived from original bag-in-box price). For the Riesling grapes, for grades where the free-run juice produced wines that retailed for approximately A\$24, A\$18 and A\$7, the pressings were all downgraded to the A\$2.50 product. The above examples clearly illustrate the stark differences in the commercial value of wine produced from different fractions. The choice of the cut between fractions therefore has the potential to significantly influence profit.

Theoretically, during expression it would be best to separate juice into many different fractions, process them all separately and only then blend wines such that blends could be definitively evaluated in final product form. Realistically, in commercial production this is not practical because of factors like tank requirements and the labour associated with managing many different fractions. It is evidently only practical to divide juice at expression into a small number of fractions, often two.

Different techniques are used to choose the division point or "press cut". Some wineries set the cut point at a particular yield, which appears to be a reasonable practical strategy, provided that it is based on appropriate data. Others determine the press cut based on real time tasting. This seems to be a much more difficult and subjective approach. The winemaker has to balance the risk of damaging the early high quality juice against the potential economic benefit of collecting more high value juice. In some instances, tasting is performed directly from a small buffer trough on the side of the membrane press, into which juice from the press flows. As an example: assume a 30 m<sup>3</sup> press into which 60 tonnes of grapes have been loaded, a 300 L press trough for tasting, and an approximate cut yield around 600 L/tonne. At this yield, 36,000 L of juice will have been expressed, yet the tasting trough will contain only the most recent 300 L of juice, or less than 1% of the free-run juice. Even with continuous tastings/observations over several trough fillings, it is difficult for the winemaker to evaluate the effect of keeping extra juice in the free-run fraction on the quality and hence value of that whole fraction. Furthermore, the winemaker has to make an assessment of potential quality of the final wine based on these tastings of a sugar-laden juice that is still to undergo significant further processing. Given the subjective nature of this approach, and perceived risks in cutting too late, the winemaker would be more likely to be conservative and cut earlier than before the free-run juice quality would actually be meaningfully affected. Furthermore, it has been observed that this repetitive and time-consuming task, often occurring in the middle of the night, was the responsibility of junior or contract winemakers as opposed to senior winemakers at some wineries. Given their lower level of experience and/or lower stature in the company, they would likely be subject to greater negative repercussions if juice/wine was damaged and thus would be prone to being even more conservative in making press cuts. Anecdotally, this practice of cutting by taste is quite common, at least in Australia.

The lost revenue over an entire vintage from cutting before there would be a detrimental effect on the free-run quality is explored in Table 6.2. A conservative price differential between the value of free-run and pressings juices to the actual winery of A\$1 per litre was assumed and further it was assumed that the winery could sell all the different grades that it produced. This price differential was verified as being conservative by personal communications with personnel at one company. Under the assumptions stated, a winery processing 20,000 tonnes of grapes per year that was cutting 20 L/tonne earlier than necessary could increase their revenue by \$400,000 by correcting this practice. While this is easily said, there are potential risks in cutting too late, and the development of more objective measures for real time juice monitoring to allow decisions to be made with confidence would be advantageous.

Early cut by (L/tonne) —	Vintage intake subject to press cuts (tonnes) <sup>a</sup>			
	5,000	10,000	20,000	
1	\$5,000	\$10,000	\$20,000	
5	\$25,000	\$50,000	\$100,000	
10	\$50,000	\$100,000	\$200,000	
20	\$100,000	\$200,000	\$400,000	
50	\$250,000	\$500,000	\$1,000,000	
100	\$500,000	\$1,000,000	\$2,000,000	

 Table 6.2: Loss in revenue from press cuts prior to the yield where the free-run quality would be detrimentally affected

<sup>a</sup> Price differential between free-run and pressings juice to the winery of A\$1 per litre has been assumed.

The significantly lower value of wine made from pressing juice fractions implies that maximising the overall yield from pressing is not necessarily the best means to maximise profits. The pressings juice is the lowest value juice but it is also the most difficult and therefore often most time-consuming to express and also likely to be subject to other higher processing costs such as fining for phenolic removal. Depending on a wine company's range of products, it may be beneficial to adapt the end yields to which juice is expressed from different grades and varieties and the splits between different juice fractions, depending on market fluctuations in grape prices.

It is also interesting to note that in wine production there is generally a cost premium in keeping small lots separate instead of blended together into large tanks. With lower grade products there is a tendency to keep products in very large batches as early as possible in the production process to reduce costs. Yet in membrane press operation, the lowest value juice is being treated more like a high value product. Many small lots of low value heavy pressings juice are being expressed individually in batch processes, when ultimately they are highly likely to later be blended together anyway. This analysis tends to suggest that employing membrane presses to express the high value juice and then a lower cost technique, such as a screw press to express the last fraction of juice from the pomace consolidated from many different membrane presses may be more economically sensible.

The specific operation of membrane presses is also a complex trade-off between quality and productivity. For example, actions like cake crumbling present some risk to quality, however, they are a practical requirement to achieve reasonable yields without high pressures or excessive processing times. Similarly, tank rotations during axial filling can result in higher levels of solids, as a result of increased shearing action on the load and reduced filtration, but they also allow for greater draining during filling. There are inevitable differences between lots of grapes and the process of expressing juice needs to be suitably adapted to achieve both quality and productivity requirements for each batch. As an example, in a press programme, if juice flow has nearly ceased at the set pressure level in the cycle, it is more efficient to proceed on to the next step in the programme, than staying on the current step producing a very low flow rate of juice. Conversely, if there is still significant flow of juice at the set pressure, proceeding to crumbling the cake or moving to a higher pressure, unnecessarily risks the juice quality. As discussed in section 5.1.4, many wine press manufacturers offer "intelligent programmes" that optimise the pressing programme in real time for a specific batch of grapes, based principally on continuous assessment of the juice flow rate. Conceptually, this is a clever means of handling variations in feedstock, and is an option worth considering when purchasing a press. Ultimately the success of an individual manufacturer's programme will depend on how they make use of information on flow rate.

Another important economic aspect associated with juice expression is temperature. There is a general understanding that white wine quality is superior if grapes and juice are subjected to cooler temperatures during harvesting and winery juice expression (Rankine 1977, White et al. 1989). The must temperature is closely related to the grape temperature at the time of harvesting, which itself is close to the ambient temperature (with a lag time of approximately 1 hour) for grapes not exposed to the sun (White et al. 1989). Therefore the best way to ensure low must temperatures is to pick when it is cool, such as during the night. This has given rise to the common practice of night-time machine harvesting in the Australian wine industry. The potential benefits from additional must chilling to further reduce temperatures have to be balanced against associated capital and operating costs. The economic merit of must chilling remains poorly resolved. It is not clear how effective a remedial action it is for warm grapes that have already been machine harvested and transported.

### 6.3 Conclusions

Inspection of wine value from different juices derived during expression illustrated that there can be stark differences in value for different yield fractions. This suggests that the choice of division of fractions has the potential to significantly influence profit. Some wineries employ real time juice tasting from a press trough containing a small quantity of the most recently expressed juice to decide on the

point of division. Pragmatic evaluation of making press cuts later is of economic interest. More objective measures to provide confidence in doing this would be of great benefit to the wine industry. The use of electrical conductivity as one technique for monitoring skin extraction will be explored in sampling from winery expression equipment and laboratory expression equipment in Chapters 7 and 9, respectively.

# CHAPTER 7: WINERY SAMPLING OF JUICE EXPRESSION EQUIPMENT

The primary aim of work reported in this chapter was to gain an improved understanding of the operations of current high-volume white winemaking juice expression equipment. In particular, it was desired to inspect and sample juice from high throughput continuous equipment that may be losing favour in the industry, and to personally verify literature reports regarding high phenolic levels and solids contents.

Given the large differences in commercial value between juice fractions during expression discussed in Chapter 6, a secondary aim of this work was to consider a rapid tool for studying skin extraction. Desseigne et al. (2003) reported that conductivity can be a useful tool for press monitoring and this was employed during sampling.

Sampling was performed at two large wineries, denoted as Winery A and Winery B, (for approximately one week each) during the 2009 Australian vintage, while Chardonnay grapes were being processed.

### 7.1 Materials and methods

### 7.1.1 Juice expression strategies

Winery A performed juice expression using the scheme outlined in Figure 7.1. Machine harvested grapes were destemmed before being pumped and loaded intermittently to trains consisting of an 800 mm diameter inclined drainer (F. Miller & Co., Australia) and an 800 mm diameter Bi-valve screw press (Coq, France, defunct manufacturer). While destemmed grapes at the winery were typically roller crushed before being pumped to the inclined drainers, roller crushing was not performed during this sampling visit. The winery had been experiencing problems with grapes not feeding well into the crusher rollers and eventually wrapping around the destemmer cage. The inclined drainer rotation speed was controlled by the operator, as was the screw press door pressure and screw extension. The screw typically rotated at 1.6 rpm. Approximate juice yields, estimated by winery operations staff, are shown in Figure 7.1 (they were not explicitly measured).

Winery B performed juice expression using the two different processes outlined in Figures 7.2 and 7.3. In Process 1, machine harvested grapes were roller crushed without destemming, before being pumped to static drainers (F. Miller & Co.). The drainers were used without carbon dioxide pressure or agitation (their agitators had been removed). Removal of the pomace from the drainers commenced approximately 7 minutes after completing filling a drainer. The pomace was screw-conveyed to 1000 mm diameter screw presses (PAP1000; Marzola, Spain). In Process 2, machine harvested grapes were destemmed and roller crushed before being pumped to 52 m<sup>3</sup> membrane presses (PMC550; Velo, Italy). Draining was performed during axial filling of the press. When full, a

short press programme (approximately 50 minutes) was initiated. When a yield of approximately 600 L/tonne (as indicated by an electromagnetic flow meter) had been obtained, the juice was cut from being "free-run" to being "pressings". At the completion of the membrane press programme, marc was intermittently emptied into a hopper and then screw-conveyed to 1000 mm diameter screw presses (PAP1000; Marzola) for final pressing. Screw press speed was typically 2 rpm and all screw press juice passed through rotary screens. Approximate yields were estimated in Figures 7.2 and 7.3 based on observations, discussions with operating personnel and inspection of daily processing summaries.

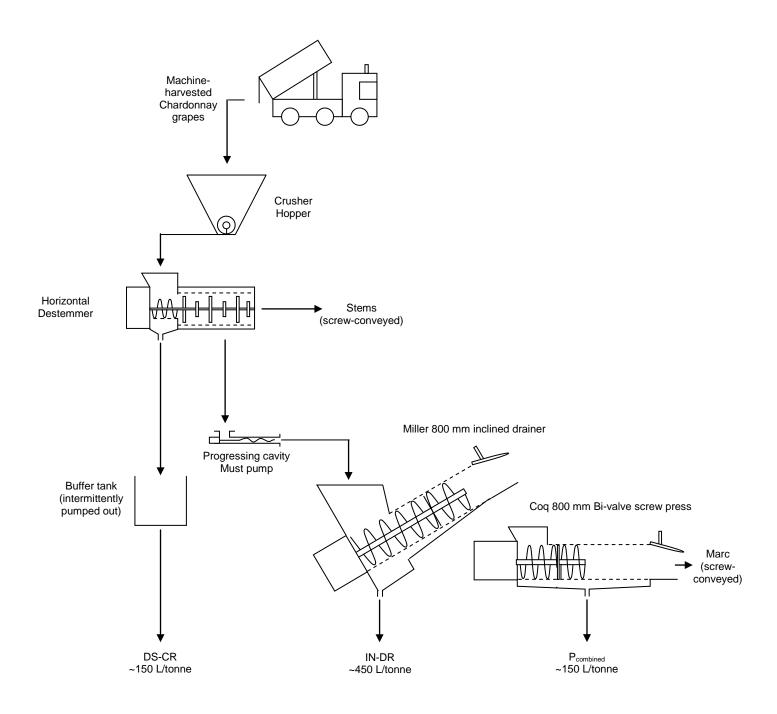


Figure 7.1: Simplified flow diagram of juice expression at Winery A

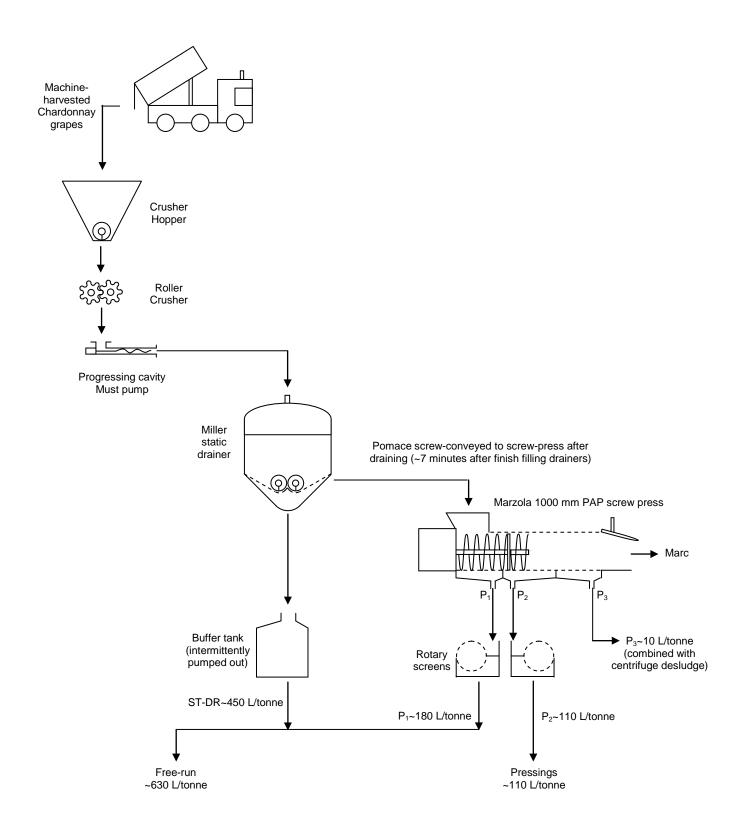


Figure 7.2: Simplified flow diagram of juice expression using Process 1 at Winery B

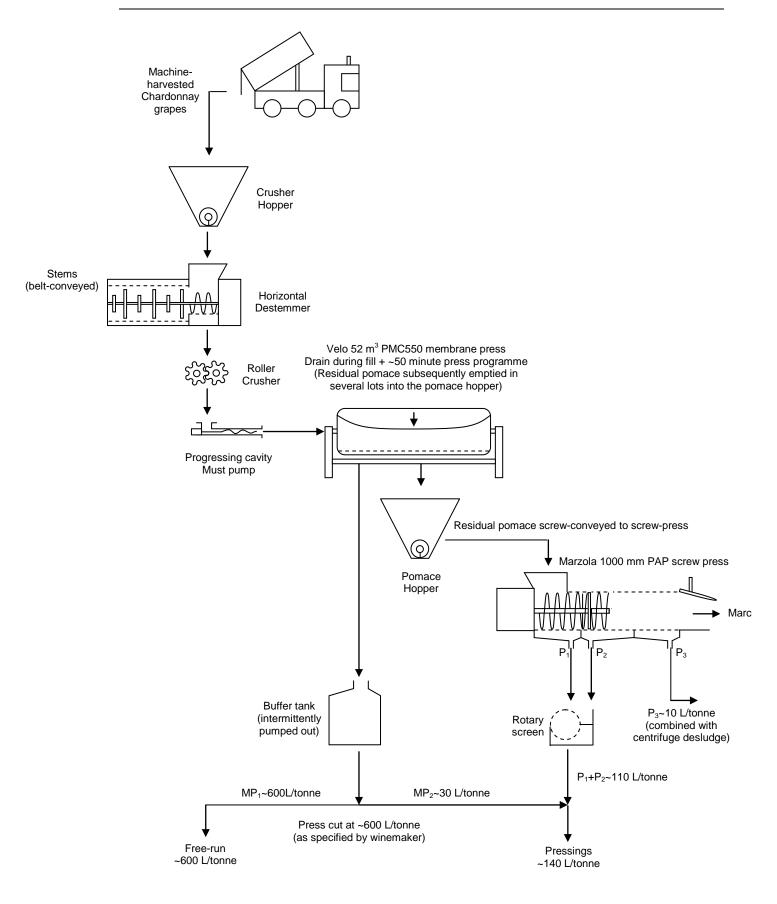


Figure 7.3: Simplified flow diagram of juice expression using Process 2 at Winery B

### 7.1.2 Sampling points

Juice samples were collected in 250 mL PP vessels. Specific sampling details are described below.

At Winery A, samples were collected from the destemmer-crusher run-off (DS-CR), inclined drainer (IN-DR) and at several points along the screw press ( $P_{a-e}$ ). The screw feeding the grapes to the destemmer passed over a slotted screen, through which free juice could drain, as shown in Figures 7.4 and 7.5. This free juice flowed to a buffer tank (Figure 7.6), which was intermittently pumped out by a centrifugal pump (Figure 7.7) activated by the buffer tank level controller. A tap located on the centrifugal pump outlet, was the destemmer-crusher (DS-CR) sampling point. To collect as representative a sample as possible, samples were collected while the pump was running, after it had already pumped out approximately half the volume of the tank. One of the 800 mm diameter Miller inclined drainers is shown in Figure 7.8. A view with expressed juice dripping onto the plastic drip sheets is presented in Figure 7.9. Inclined drainer (IN-DR) samples were collected as juice ran down the plastic drip sheet into the stainless steel collection area, as shown in Figure 7.10. Juice from each screw press flowed into three drip trays, which fed a common buffer tank (see pipes under screw press in Figure 7.11). To enable the collection of samples at different points along the press, juice was collected manually by holding a container underneath the plastic sheets at different locations along the press (Figure 7.12)

At Winery B, juice from the static drainers (Figure 7.13) and from the membrane presses (Figure 7.14), were fed into respective buffer tanks, an example of which is shown in Figure 7.15. These were intermittently pumped out upon activation of a high level sensor until a low level sensor was set off. Centrifugal pumps were used and flow rates were typically 800-1000 L/min. A sample tap located immediately upstream of the respective pumps, as shown in Figure 7.16, was used to collect the static drainer (ST-DR) and membrane press (MP) samples. Again, to ensure as representative a sample as possible, samples were collected while the pump was running, after it had been running for approximately 1-2 minutes. Furthermore where possible, samples were collected during periods of consistent steady processing operations (i.e. not during grape receival delays). This was to ensure a steady flow of juice into the buffer tanks to promote mixing and also to facilitate collection of samples of relatively freshly expressed juice which were not overly diluted with stagnant older juice in the buffer tanks. Screw press (Figure 7.17) juice sample were collected upstream of the rotary screens. The screw press juice samples in Process 1 were collected at the locations indicated in Figure 7.18. At Winery B, each set of rotary screens (consisting of larger open tank with a rotary screen to process screw press fraction P<sub>1</sub> and a smaller open tank with a rotary screen to process screw press fraction P<sub>2</sub>) serviced two screw presses, as indicated by the two pipes feeding into each tank in Figure 7.18. For Process 2, both screw press juice fractions were typically fed to the same rotary screen tank as the ultimate intention was to combine the two fractions. For this case, samples were collected directly from the outlet pipes (prior to mixing of the juices in the rotary screen tank) in order to obtain samples from the separate sections of the screw press ( $P_1$  and  $P_2$ ). The minor screw press fraction ( $P_3$ ) was

not sampled as fixed pipe work was in place to combine this juice from several screw presses into one tank together with desludge wet solids from disc-stack centrifuges.



Figure 7.4: Draining section at feed to destemmer-crusher



Figure 7.5: Underside of draining section at feed to destemmer-crusher (drain hole to buffer tank shown)



Figure 7.6: Buffer tank for destemmer-crusher run-off



Figure 7.7: Centrifugal pump for pumping out destemmer-crusher run-off buffer (sampling location marked)



Figure 7.8: Miller 800 mm inclined drainer

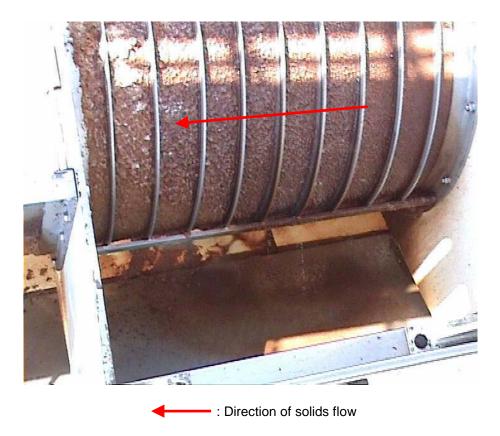


Figure 7.9: Inclined drainer screens (juice dripping onto plastic drip sheets)



IN-DR sampling point

Figure 7.10: Underside view of inclined drainer (sampling location marked)



Figure 7.11: Coq 800 mm Bi-valve continuous screw press

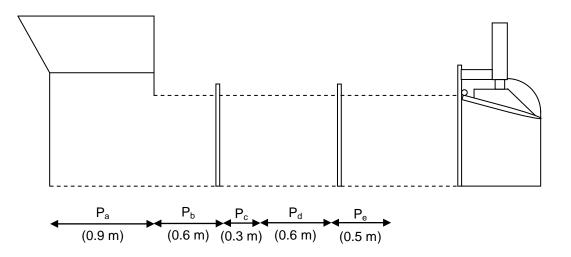


Figure 7.12: Winery A continuous screw press sampling locations



Figure 7.13: Miller static drainers (door of drainer #5 opening to evacuate pomace onto screwconveyors)



Figure 7.14: Velo PMC550 membrane press



Figure 7.15: Two of the buffer tanks into which static drainer juice or membrane press juice flows



ST-DR / MP sampling point

Figure 7.16: Sample tap immediately downstream of static drainer and membrane press buffer tanks



Figure 7.17: Marzola PAP1000 continuous screw presses



Figure 7.18: Rotary screens servicing 2 screw presses (Winery B - Process 1 sampling locations marked)

### 7.1.3 Sample analysis

Samples were poured through a stainless steel strainer (2 mm diameter holes) using a PP funnel (Figure 7.19) into  $2 \times 50$  mL PP tubes to remove bulk solids. Both tubes were treated with 100 mg/L potassium metabisulfite as a short-term preservative measure. One tube was clarified (5 minutes, 1,460 ×g) and the supernatant poured into a fresh 50 mL PP tube. After treatment of juice in this tube with 900 mg/L potassium metabisulfite, it was then distributed into  $3 \times 15$  mL PP tubes that were then frozen (-20 °C) for later phenolic analysis. The other tube was used for immediate analyses. Conductivity was determined as described in Chapter 3. Solids content was estimated by a spin test (5 minutes, 1,460 ×g) in the tapered graduated glass tubes shown in Figure 7.20 (Westfalia Separator, Germany). Juice density was measured using the supernatant, following the method described in Chapter 3. Microscopy of juice samples was also performed. A drop of juice was pipetted onto a microscope slide and a cover slip placed over it. This was then inspected using a LCD digital microscope (Celestron, USA) and photographs were taken at  $40 \times$  and  $100 \times$  magnifications. Magnification was verified using a stage micrometer (Shell-Lap Supplies, Australia).

For phenolic analysis, thawed samples (4 minutes, 50 °C) were first diluted to 25% (v/v) in 0.03% (v/v) acetic acid (Sigma-Aldrich, USA) in RO water (pH ~3.5). Diluted samples were filtered through 0.45  $\mu$ m hydrophilic PP syringe filters with glass fibre pre-filters (Pall, USA) and analysed for phenolic content by UV spectroscopy in 1 mm path length quartz cells against an RO water reference

(Pharmaspec UV-1700; Shimadzu, Japan). The spectral results were corrected for dilution and normalised to a 1 cm path length.



Figure 7.19: Stainless steel strainer and PP funnel for preliminary bulk solids removal



Figure 7.20: Spin test tubes for maximum (a) 3%, (b) 6%, and (c) 10% solids content

### 7.2 Results and Discussion

The results for Winery A, Winery B – Process 1 and Winery B – Process 2 are summarised in Tables 7.1, 7.2 and 7.3, respectively.

Sample <sup>a</sup>	A <sub>280</sub> (au)	A <sub>320</sub> (au)	Conductivity (mS/cm)	Solids (v/v)	RI (°Brix)
Doctommor orughor	7.9	5.1	4.34	1.8%	21.9
Destemmer-crusher run-off (DS-CR)	(17%)	(28%)	(3%)	(62%)	(3%)
Inclined drainer (IN-	9.1	7.0	3.69	2.7%	21.7
DR)	(13%)	(18%)	(6%)	(35%)	(2%)
Screw press (P <sub>a</sub> )	11.6	9.1	4.79	2.2%	22.05
	(9%)	(9%)	(4%)	(15%)	(4%)
Screw press ( $P_b$ )	13.1	10.8	4.83	4.6%	21.8
	(4%)	(7%)	(3%)	(66%)	(4%)
	15.9	14.0	5.52	2.7%	22.1
Screw press (P <sub>c</sub> )	(20%)	(27%)	(14%)	(39%)	(5%)
Screw press (P <sub>d</sub> )	23.4	19.9	6.34	2.8%	22.2
	(39%)	(31%)	(14%)	(47%)	(4%)
Screw press (P <sub>e</sub> )	26.8	24.3	7.53	1.0%	21.8
	(16%)	(16%)	(9%)	(62%)	(4%)

Table 7.1: Winery	A analytical results
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<sup>a</sup> Average of 4-6 samples. Coefficient of variation reported in parentheses. See Appendix B for individual measurements.

Sample <sup>a</sup>	A <sub>280</sub>	A <sub>320</sub>	Conductivity	Solids	RI
	(au)	(au)	(mS/cm)	(v/v)	(°Brix)
Static drainer (ST-DR)	9.1	8.0	3.60	3.3%	23.0
	(10%)	(13%)	(10%)	(37%)	(5%)
Screw press (P <sub>1</sub> )	10.6	9.6	3.76	4.9%	23.3
	(8%)	(10%)	(6%)	(15%)	(5%)
Screw press (P <sub>2</sub> )	16.9	16.8	4.57	5.4%	23.6
	(12%)	(13%)	(12%)	(24%)	(5%)

### Table 7.2: Winery B – Process 1 analytical results

<sup>a</sup> Average of 10 samples. Coefficient of variation reported in parentheses. See Appendix B for individual measurements.

Table 7.3: Winery B – Process 2 analytical results					
Sample	A <sub>280</sub> (au)	A <sub>320</sub> (au)	Conductivity (mS/cm)	Solids (v/v)	RI (°Brix)
Run 1 (135 m <sup>3</sup> / 146 tonr	nes <sup>a</sup> loaded)				
Membrane press (MP) <sup>b</sup>					
137 L/tonne	9.8	8.4	4.60	2.8%	21.8
336 L/tonne	8.4	6.9	4.30	1.5%	20.5
514 L/tonne	9.1	7.4	4.32	1.0%	20.7
562 L/tonne	9.8	8.0	4.33	1.0%	21.0
630 L/tonne	11.5	9.8	4.64	0.8%	21.0
Screw press (P <sub>1</sub> )	9.4	7.0	4.65	2.5%	20.5
Screw press (P <sub>2</sub> )	12.7	8.8	5.24	1.3%	21.8
Run 2 (115 m <sup>3</sup> / 125 tonr	nes <sup>a</sup> loaded)				
Membrane press (MP) <sup>b</sup>					
160 L/tonne	9.1	8.8	3.66	2.3%	22.4
304 L/tonne	9.5	9.8	3.44	1.8%	22.2
416 L/tonne	10.1	10.0	3.64	1.6%	21.3
536 L/tonne	9.6	9.3	3.68	1.0%	21.8
632 L/tonne	10.3	10.2	3.68	0.8%	22.1
Screw press (P <sub>1</sub> )	11.0	9.2	4.24	2.8%	22.0
Screw press (P <sub>2</sub> )	13.4	10.6	4.83	2.5%	22.3

<sup>a</sup> Estimated assuming a must density of 1085 kg/m3 (Rankine 2004) <sup>b</sup> Yields calculated from electromagnetic flow meter measurements.

### 7.2.1 Phenolic content

Typical spectra from one full set of samples at Winery A and from Winery B - Process 1 are presented in Figure 7.21. Phenolic concentration, as estimated by UV absorbance, increased with increasing yield. This was also generally the case for samples from Winery B - Process 2 (Table 7.3). One noticeable discrepancy is that the first sample of Run 1 featured a higher than expected phenolic content. This seems most likely to be a consequence of the juice in the buffer tank being diluted with the juice expressed at the end of the previous membrane press run. This dilution effect becomes less relevant with increasing juice expression.

Later screw press fractions at Winery A were found to have considerably higher phenolic contents. This trend is illustrated in Figure 7.22 which shows phenolic results along the length of the screw press. The phenolic levels appear to increase relatively sharply at approximately 1.7 m (P<sub>c</sub>) or 2.2 m(P<sub>d</sub>) samples. This correlates well with the position of the Bi-valve (the anti-return device in the Coq screw press, as shown in Figure 5.29). When the screw was not extended, the Bi-valve was located approximately at the boundary between P<sub>b</sub> and P<sub>c</sub>. When the screw was hydraulically extended by the operator, the Bi-valve was located approximately at the boundary between Pc and Pd. A similar trend

was also observed at Winery B for Process 1. The static drainer (ST-DR) sample and first screw press fraction ( $P_1$ ) were found to have relatively similar phenolic contents. However, the second screw press fraction ( $P_2$ ), which was largely derived from the section downstream of the Marzola screw press anti-return device (as shown in Figure 5.33), was found to have a much higher phenolic content. This observed profile for phenolic concentration along the screw press, where the concentration sharply increases around the location of the anti-return device, corresponds with data reported by Terrier and Blouin (1975).

Ultimately, the relative flow from the different sampling locations determines the importance of these analytical results. While it was not possible to explicitly measure juice flow from each zone in this trial, estimates of the contribution from the different fractions (from discussions with winery personnel and from visual observations of flow at different points), suggested that there was very little flow in the latter sections of the screw presses, corresponding to the very high levels of phenolics, particularly at Winery A. Therefore it is very important not to read too much into these very high phenolic values in this very low yield of juice. Plausibly, this yield fraction of very highly phenolic juice may not have even been expressed in a membrane press, and thus demonising screw presses on this basis would be unwarranted as long as the low quality fractions could be kept separate.

It should be noted that the process configurations have been simplified for this report. At both wineries there were actually several drainers and presses that interacted based on operator control. For example at Winery A, the operator intermittently directed the mash from the destemmer to the hoppers of the different inclined drainers screw press trains. At Winery B the operator also intermittently directed the mash from the crusher to the different static drainers, and on completion of draining would screw-convey the pomace to one of several different screw presses. These factors made actual measurement of flow rate impracticable without major process modification, which was not possible.

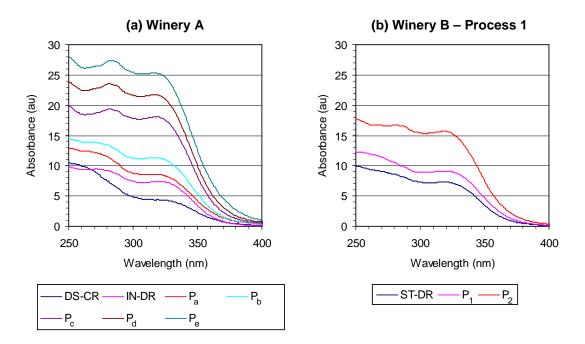


Figure 7.21: Juice sample spectra from Winery A and Winery B – Process 1

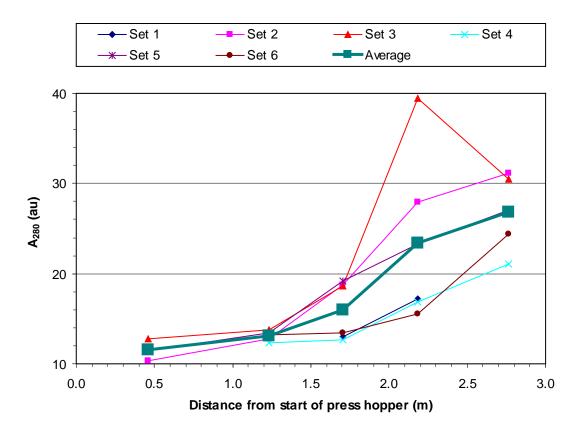


Figure 7.22:  $A_{280}$  along the length of the screw press at winery A

### 7.2.2 Conductivity

Conductivity showed a similar trend to the phenolic content and inspection of the results in Tables 7.1, 7.2, 7.3 and Figure 7.23, lead to similar conclusion. Electrical conductivity is a measure of the

ability of a juice samples to conduct an electrical current, which is related to the ionic species present. Potassium, the predominant mineral cation in grapes (Boulton et al. 1996) is found at much higher concentrations in the skin than in the pulp (Possner and Kliewer 1985, Coombe 1987), corresponding generally with the phenolic distribution in the grape skin and pulp. Therefore, the similar pattern of increases in phenolics and increases in conductivity with different degrees of skin extraction is to be expected. One notable discrepancy was the results for the destemmer-crusher run-off reported in Table 7.1. In contrast to the phenolic results, the destemmer-crusher run-off juice sample (DS-CR) featured a consistently higher conductivity than samples collected from the inclined drainer (IN-DR). A possible explanation of this inconsistency is the augmentation of the endogenous grape juice ions with those from potassium metabisulfite, which was added to the destemmer-crusher hopper.

Conductivity results from unpublished sampling performed at another winery are reported in Figure 7.24. Sampling at that winery denoted Winery C, was principally performed by one of the author's supervisors, Dr Chris Colby during the 2008 vintage. Juice was sampled from the press trough during filling, draining and pressing of one batch each of approximately 45 tonnes of Chardonnay, Semillon or Riesling grapes with a 32 m<sup>3</sup> membrane press. Press operation involved axial filling and gravity draining in the press, combined with a standard press programme (see section 5.1.4 for the definition of a standard programme) in use at the winery. Figure 7.24a displays a relatively consistent conductivity up until approximately 600 L/tonne when the conductivity rose significantly. This rise at around the expected typical cut point does suggest that conductivity could be a useful tool for making press cuts in membrane press operation. However, the change of the slope in curve is probably insufficient to make a cut in real time until much of the upward swing is complete. Interestingly the pattern with time (Figure 7.24b) was guite different for each of the three different batches, partly reflecting different yields obtained at different points in time. Discussions with one winemaker who had performed some trials with conductivity as a technique for making splits between free-run and pressings fractions noted that they found it to be of limited use because sometimes the conductivity would be stable and then rise rapidly around the approximate cut yield, while other times the conductivity would rise more gradually and not pass through a clear inflexion. A less clear inflexion against time than against yield was observed in Figure 7.24. Therefore it seems important that conductivity results are interpreted with respect to yield rather than time. Furthermore, gradual rises in conductivity may be indicative of problems with the condition of the initial fruit and the press programme that are preventing a fractional expression being obtained and should not be discounted simply because there is no clear cut point.

Increased phenolic levels are a major issue at higher juice yields that contribute to lower quality and necessitate division of fractions. Phenolics are difficult to measure rapidly. Even estimation of total phenolic content by spectral absorbance at 280 nm requires time consuming and expensive sample clarification, and as discussed in Chapter 3, it is not a perfect measurement. Conductivity has the advantage of being rapid, cheap and simple (Desseigne et al. 2003). The relationship between conductivity and phenolic levels measured by spectral absorbance at 280 nm in this work are shown

in Figure 7.25. The coefficients of determination for individual sample sets (i.e. collected at approximately the same time from the different sampling locations) are presented in Table 7.4. Table 7.4 shows that the correlation for individual sample sets was often very strong, however, the overall relationship for all samples was poorer ( $R^2 = 0.51$ ). There are different relationships between phenolic content and conductivity even for different batches of the same grape variety. At wineries A and B juice sampling was performed over several days with grapes from many different vineyards being processed. The strong correlation for individual sample sets is likely related to the grapes being from the same vineyard grown under similar conditions and thus with relatively consistent distributions of phenolic compounds and potassium through the berries, such that increased skin extraction results in proportionally similar increases in phenolic levels and conductivity.

Overall, results support the suggestion that conductivity can be a useful rapid indicator of skin extraction during draining and pressing, for individual batches of grapes. One should be aware of the influence of any large poorly distributed point additions of chemicals such as potassium metabisulfite that could skew results. The relationship between conductivity and phenolics will be further explored in Chapter 9, based on laboratory expression experiments.

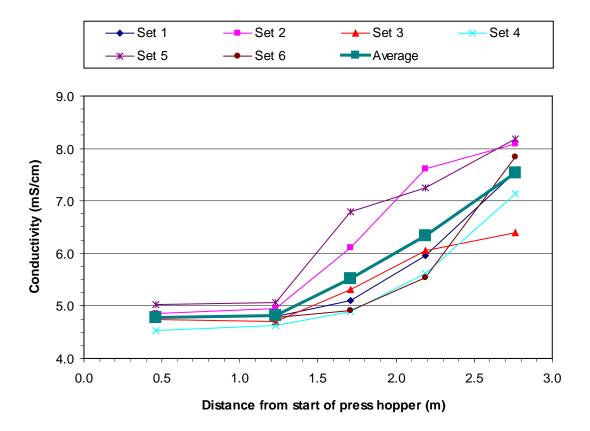


Figure 7.23: Conductivity along the length of the screw press at Winery A

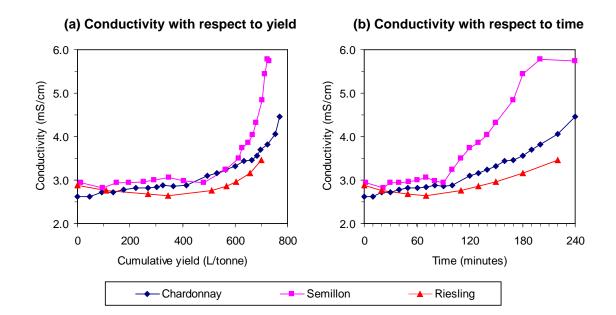


Figure 7.24: Conductivity with respect to yield and time during sampling of a membrane press at Winery C while axial filling, draining and pressing

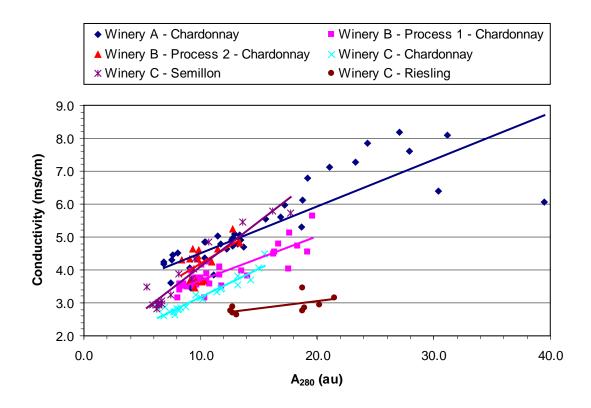


Figure 7.25: Relationship between conductivity and A<sub>280</sub> for juice samples collected during winery sampling

Origin and variety	Sample set	n	A <sub>280</sub> vs. conductivity
	1	5	0.92
	2	5 7	0.98
	3	7	0.80
Winery A - Chardonnay	4	6	0.87
, ,	5	7	0.94
	6	6	0.93
	Combined	38	0.70
	1	3	0.999
	2	3	0.96
	3	3	0.95
	4	3	0.98
Winery B – Process 1 -	5	3	0.95
Chardonnay	6	3	0.999
Charaonnay	7	3	0.72
	8	3 3	0.995
	9	3	0.99
	10	3	0.999
	Combined	30	0.71
Winery B – Process 2 -	1	8	0.75
Chardonnay	2	7	0.90
Chardonnay	Combined	15	0.31
Winery C – Chardonnay	1	22	0.92
Winery C – Semillon	1	19	0.91
Winery C – Riesling	1	9	0.36
All samples		133	0.51

## Table 7.4: Coefficient of determination (R<sup>2</sup>) between A<sub>280</sub> and conductivity for juice samples collected during winery sampling

### 7.2.3 Solids content

Solids contents for individual sampling locations varied considerably between sample sets. This is indicated in Tables 7.1 and 7.2 by the higher coefficients of variation than for any of the other analytical parameters reported. The variation in solids content between different sample sets was likely a result of varying feedstock and the specific mode of drainer and press operation.

At Winery A, the destemmer-crusher solids were relatively low with an average of 1.8%. This is likely a consequence of juice being gradually expelled from relatively intact berries during transportation to the winery. The solids content of the juice from the inclined drainer was higher but still averaged only 2.7%. While the solids were relatively low, it should be noted that solids in this work were determined by a spin test. It is possible that poorer results may be obtained if static settling was used. For example: Menegazzo et al. (1977) found the ratio of solids content as measured by static settling to

that measured by centrifugation to be approximately 1.5 for juice from a static drainer, but to be 4-5 for juice from an inclined drainer.

In individual sample sets along the screw press at winery A (Figure 7.26) the solids levels would typically increase to a maximum at some point along the press before decreasing to a minimum value at the last sampling point ( $P_e$ ). The specific solids content measurements in the juice are a combination of the solids generated by the shearing of the cake and the removal of some of these solids by cake filtration. The specific cells from which juice originates are also important.

Typical juice microscopy for one set of samples at Winery A is presented in Figure 7.27. The general trend was for the screw press samples to have more and/or smaller solid particles relative to the destemmer-crusher run-off (DS-CR) and inclined drainer (IN-DR) samples. The fine solids from further along the press are increasingly likely to be from skin cells. The low solids levels at  $P_e$  shown in Table 7.1 are likely related to the screw not extending to this zone of the press. The cake in this zone is therefore quite static and there is little shearing that could create solids. Additionally, any solids created are more likely to be filtered out by the compact static cake.

For Winery B – Process 1, the solids content increased from the static drainer (ST-DR), to the first (P<sub>1</sub>) and second zone (P<sub>2</sub>) of the screw press. Typical juice microscopy for Winery B – Process 1 samples are presented in Figure 7.28. While there was some variability in microscope photos taken across the trial, the general trend was for P<sub>2</sub> samples to contain more and/or smaller solid particles relative to samples ST-DR and P<sub>1</sub>.

For Winery B – Process 2, the solids content in juice from the membrane press was inversely related to yield (Table 7.3). This is likely a result of the membrane press cake structure initially being relatively open, thereby enabling free juice to pass through with relatively little filtration. Additionally, there would have been a large initial quantity of free juice that had been relatively roughly expelled from the berries by destemming, crushing and pumping. As the yield increased, the solids content decreased to approximately 1%. This is attributed to the gentler expelling of juice from the berries during pressing and improved juice filtration due to the more compact pomace cake. Juice microscopy of the Winery B - Process 2 membrane press samples is presented in Figure 7.29. These photos were similar in both membrane press runs and are consistent with the relatively low values of solids content reported in Table 7.3. Interestingly, the solids content in the samples collected from the screw press after membrane pressing (Table 7.3) were considerably lower than those from the screw press after static draining (Table 7.2). This may be a consequence of the much higher yields already expressed by membrane pressing, compared to static draining (approximately 630 L/tonne compared with 450 L/tonne), such that the juice is increasingly derived from smaller skin cells as opposed to large pulp cells, and that the more compact, drier cake is a more effective filter. The drier cake may also be gripped better by the screw and the cage. Less slippage against the screw thread and cage would limit the tendency for material to backflow along the screw channel or to rotate with the screw

shaft. This is a plausible explanation as it has previously been reported (Ordódy 1960, Troost 1961) that the grapes must be sufficiently pre-drained for juice separation with a screw press to be efficient. It should be noted that the comparison between Process 1 and Process 2 is somewhat limited by the disparity between the number of sample sets collected for Process 1 and Process 2 (10 versus 2 respectively). The lower number of sample sets collected for Process 2 was driven by the more time consuming nature of Process 2 sampling and also by the availability of membrane press sampling results already in the literature (e.g. Desseigne et al. 2003). Additionally, it was considerably more difficult to obtain representative samples of the juice from the screw presses following membrane press to the screw press hopper. In contrast, there were several static drainers in operation in Process 1 and their much more regular emptying into the screw press hoppers allowed for the collection of more representative pseudo steady state samples.

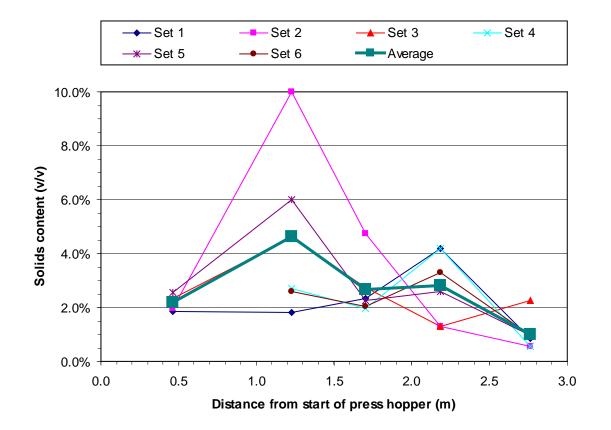


Figure 7.26: Solids content along the length of the screw press at Winery A

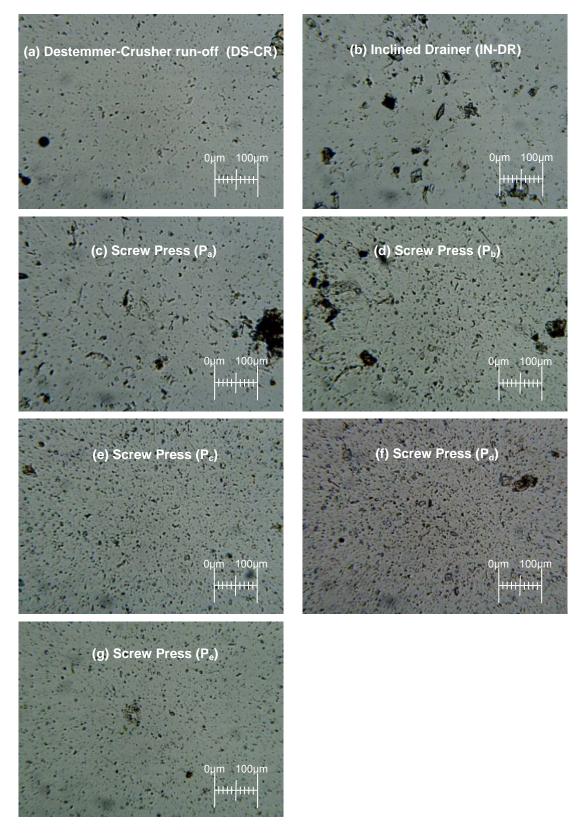


Figure 7.27: Microscopy (100 ×) for Winery A samples

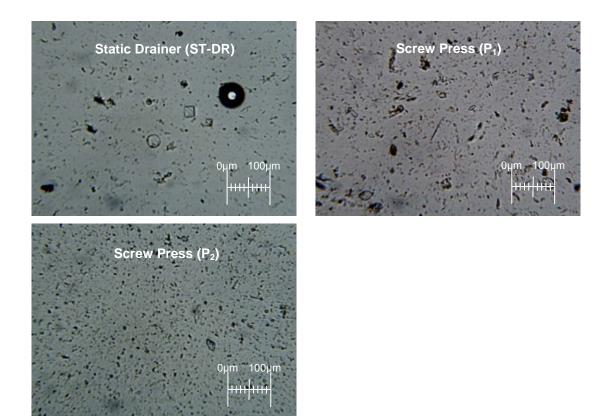


Figure 7.28: Microscopy (100 ×) for Winery B – Process 1 samples

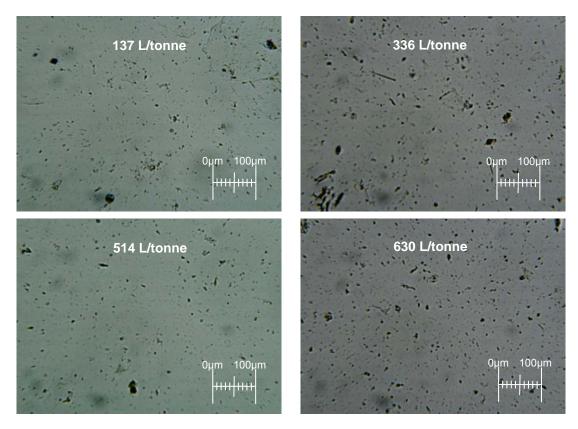


Figure 7.29: Microscopy (100 ×) for Winery B – Process 2 samples

### 7.2.4 Operational observations

During sampling visits at Winery A and B several operational observations were made either directly or from discussions with operators, that offer some insight into process choice.

At Winery A there were four parallel trains of 800 mm inclined drainers and screw presses. These could generally be operated by a single operator. Similarly at Winery B a bank of Marzola screw presses was able to be operated with very little operator attention. An operator monitoring the filling and emptying of static drainers was able to simultaneously also operate the screw presses. This reiterates the low labour requirements of screw presses.

For Process 2 at Winery B, at the completion of the membrane press programme, the pomace had to be emptied into a hopper from which it was screw conveyed to the screw press. The quantity of must loaded into the membrane presses was quite large (125 and 146 tonnes in the two runs sampled), and after pressing, there was still a large quantity of residual pomace to be conveyed to the screw press. In practice this residual pomace could not be emptied into the hopper in a single step as the pomace would tend to bridge over the lone screw conveyor in the hopper. The operators had to empty the membrane press into the hopper in several small lots to avoid this bridging and this consumed operator time as well as preventing the membrane press from being used for the next batch. This is an example of the sorts of problems that can arise when a batch and continuous process are combined. A completely continuous process would ultimately be more desirable. In this particular case operational discontinuities could possibly have been smoothed by the use of several smaller volume membrane presses.

At Winery B, The membrane presses were fitted with a clean in place system using high pressure water. This was utilised once a day, however, because of the high press utilisation, an operator still usually needed to perform a confined space entry to thoroughly clean the internals of the press. This is indicative of the importance of effective clean-in-place systems to minimise labour requirements and safety concerns associated with confined space entries.

## 7.3 Conclusions

Sampling of Chardonnay juice from two wineries provided an important supplement to literature review and laboratory experiments. Phenolic content and conductivity generally increased with juice yield. Screw pressing (even with a modern large diameter model) was found to produce relatively high levels of solids compared to membrane presses. However, the impact of the screw press action was apparently dependent on the dryness of the feed material – the drier the material, the lower the solids content. Conductivity generally correlated well with phenolic content for individual batches, but a global correlation was poorer. It is still suggested as a useful tool for those studying juice expression, optimising press programmes and/or trying to establish press cut yields for different types, sources and conditions of grapes at a winery.

# CHAPTER 8: EXPRESSION TECHNIQUES IN OTHER INDUSTRIES

White wine production is one of many industries where mechanical expression plays an important role. Experiences from other industries may provide insight and inspiration for improvements in juice expression for white wine production. Expression processes used in several industries are discussed in this Chapter. These industries have been chosen because they have a similar feedstock or product, offer some insight into expression, or use expression equipment that is not widely used in white wine production.

### 8.1 Red wine production

A closely related process to white grape juice expression is red wine expression. A key difference in the production of red wine is the inclusion of grape seeds and skins during fermentation with final expression only occurring towards the end of, or after, fermentation. Not surprisingly, similar expression equipment is generally used as in white wine production. However, during fermentation, the cells of the grape are significantly degraded. This means that much more wine can be separated simply by draining prior to pressing than juice from fresh white grapes. The remaining material is also more easily pressed, but is quite susceptible to mechanical damage because of the degraded structure of the grape (Ribéreau-Gayon et al. 2006b).

Red wine is more a whole grape product than white wine, which is principally a pulp-derived product. While minimisation of phenolic compounds is a key principle in white wine production, the same is not always true for red wine production. Bitterness and astringency derived from phenolic compounds at appropriate levels are a critical sensory component of red wines and a key sensory differentiator between red and white wines (Peynaud 1987). Furthermore, anthocyanins derived from red grape skins and complex adducts formed from them are responsible for the colour of red wine. Red wines typically have phenolic concentrations in the order of 5-10 times those in white wines (Singleton 1992, Waterhouse 2002).

### 8.2 Waste treatment sludge dewatering

Continuous decanter centrifuges and belt presses have been extensively used for dewatering of waste treatment sludges, typically after sludge chemical conditioning (Schwartzberg 1997, Tchobanoglous and Burton 1991). Notably, applications of this nature are somewhat different to expression in wine production as solids dewatering is the principal goal, not liquid quality.

Baskerville et al. (1978) explains that belt presses for dewatering sludge appeared commercially in the late 1960s and that they only became a practical proposition for sludge dewatering with the availability of water-soluble high molecular weight polyelectrolytes for sludge conditioning. These can

be so effective as to convert a homogeneous sludge into a heterogeneous mixture of large flocs and water from which water may be readily separated. In a belt press for sludge dewatering there is usually a gravity draining zone (sometimes assisted by a partial vacuum) prior to the compression zone where the partially dewatered sludge is progressively squeezed as it passes between rollers. The action of moving the belt around the rollers creates shear stresses on the sludge cake by differential movement of the belts. Pressure on the cake is alternatively applied at each roller, then released as the belt passes between rollers, allowing the weave to wick away some of the water. A series of high-pressure spray washers are used to clean the belts prior to cycling back to the head of the press (Severin et al. 1998). If free drainage is still occurring when the sludge enters the compression zone, the excess water tends to act as a lubricant promoting the movement of sludge solids out of the sides of the press and also through the belt itself (Baskerville et al. 1978). If sludge extruded into the belt is not sufficiently removed, the drainage problems can be exacerbated (Severin et al. 1996).

### 8.3 Apple juice production

Apples are a much harder fruit than grapes and consequently much more extensive comminution is typically employed to facilitate expression than gentle grape roller crushing (Ashurst 2007). Hammer or grating mills are commonly used (Beech and Carr 1977, Peden 1974, Bump 1989). Coarseness of milling is a compromise between larger pieces of pulp that yield less juice and smaller particles that provide less bulk and will tend to flow out the exit along with the juice (Cockram 1993). Pressing larger pieces of apple will produce juice lower in suspended solids than when pressing smaller pieces (Nelson et al. 1980).

Batch rack and cloth presses as discussed in section 5.1.2, were an early style of press that was widely used for apple juice expression. These presses produced juice low in solids and typically didn't require the use of press aids. The small packets of comminuted mash provided a short juice exit path Horizontal hydraulic single plate presses are widely used for apple juice expression. These devices feature large numbers of flexible grooved plastic cores covered in cloth and stretched between the two ends of the press. Juice flows through the cloth, and then down the core grooves to the end of the press where it is collected (Cockram 1993). This style of press was introduced by Bucher in 1965 (Bucher 2008). The current model, capable of processing 7-10 tonnes/hr of apples, is presented in Figure 8.1, together with a diagram illustrating the principle of operation. Like the horizontal plate presses used in white wine production it works by series of cycles of pressing and crumbling. Crumbling is achieved by retracting the piston and by rotating the unit. Similarly to modern batch pneumatic presses, the newer models also feature self-optimising programmes (Bucher Foodtech 2010). The use of grooved cores that extend into the mash is similar to those employed on some Bucher-Vaslin membrane presses, as shown in Figure 5.17. The cores provide a short juice exit path to assist drainage and aid crumbling as the piston is removed. This arrangement is apparently effective and low solids juice is produced typically without the need for press aid (Bump 1989).

While press aids, either in the form of grape stems or exogenous additives have been employed in white wine production (Anon 1986), their use appears to have been much more common in apple juice production, probably because of the more highly comminuted mash. With grapes, the large quantities of relatively hard and intact skins and seeds remaining after roller crushing likely provide a greater cake structure not found in apple mash. Rice hulls, cellulose and wood fibres have all been used as press aids (Crowe 1970, Bump 1989, Cockram 1993). Press aids serve to provide structure to the mash and channels for juice to exit and depending on the type, scouring of the press cage surface to prevent screen blinding (Bump 1989). They are reportedly particularly important when pressure is applied rapidly or in moving systems like screw presses (Bump 1989). Press aids can be expensive over time, increase the press load, impart off flavours and result in disposal problems (Cockram 1993).



Figure 8.1: Bucher HPX 5005iP horizontal hydraulic single plate press with cloth covered drainage cores and schematic of operating prinicple

### (From: Bucher Foodtech 2010)

Screw presses have also been used in apple juice expression. There have been similar problems relating to high juice solids contents (Crowe 1970, Peden 1974, Bump 1989) as experienced with white grape screw press juice expression. Examples of screw presses that have been used for apple juice production include the vertical Jones Pressmaster presented in Figure 8.2 and the Rietz/Vincent screw press already shown in Figure 5.37 (Bump 1989). Notably both these feature an interrupted flight design.

In apple juice production, there has been a greater use of non-screw type continuous equipment like belt presses and decanters or other centrifuges (Peden 1974, Bump 1989, Cockram 1993) than in white wine production. The use of belt presses seems to be particularly prevalent, with the combined press market share of the horizontal Bucher-style presses and belt presses reported recently to be 90% (Novozymes 2006). Belt presses are significantly cheaper than the Bucher presses, and incur reduced operating costs. However, they achieve lower yields but this can be made up by leaching. Bump (1989) reports that the belt press manufactured by Ensink (see Figure 8.3) operated well with fresh or firm fruits but with soft fruits, solids were very high. Peden (1974) also reported high juice

solids with this style of press processing apples. A modern belt press and schematic of operation are presented in Figures 8.4 and 8.5, respectively. The cake can be very thin in belt presses, with a thickness as small as 0.5 cm at the exit, and there can also be significant pressure and shear around the small rollers where the outer belt has a longer distance to travel (Cockram 1993).

Enzymes usage to assist expression is common in the production of apple juice. Enzymes were first employed for apple juice clarification in 1930s, but it wasn't until the 1970s that they started to become more commonly used at the mash stage to aid pressing (Will et al. 2000, Grassin and Herweijer 2008). While fresh apples are relatively easily pressed giving a high juice yield, stored apples become difficult to press unless macerated with enzymes. This is related to the apple insoluble protopectin being slowly transformed into soluble pectin by endogenous apple pectinases during storage (Grassin and Herweijer 2008). Enzyme treatment has become increasingly important with the increased use of stored excess or defective table varieties as opposed to fresh fruit for apple juice production (Will et al. 2000, Grassin and Herweijer 2008). One enzyme manufacturer notes that their latest enzyme only degrades the soluble pectin but leaves the insoluble pectin intact so that the structure of the press mash will not be destroyed (Novozymes 2006).

Another enzyme process used in apple juice production to achieve higher yields is "total liquefaction". In this process mash is treated with both pectinases and cellulases at approximately 50 °C largely liquefying apple cell walls. This results in higher juice yields but poor sensory qualities, including increased release of phenolics and therefore browning. To maintain the quality of the majority of juice but achieve higher total yields, total liquefaction has been practised as a secondary step on the residual pomace from normal expression (Will et al. 2000, Mehrländer et al. 2002) as opposed to the common practice of leaching with water or low sugar juice (Cockram 1993).

In comparing the techniques used for expression of fruit juices with juice for wine production, it is apparent that fruit juice will ultimately retain much higher sugar content. The sweetness could mask concentrations of undesirable compounds that could be sensorially significant in wine. Therefore more vigorous processing techniques could often be more readily applied.

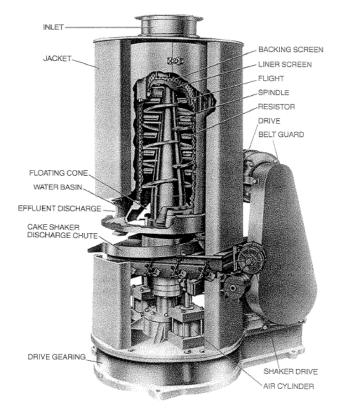


Figure 8.2: Jones Pressmaster vertical screw press (From: Bump 1989)

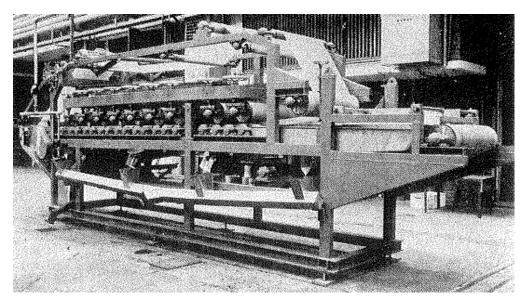


Figure 8.3: Ensink belt press for apple juice production (From: Ensink 1974)



Figure 8.4: Flottweg belt press for apple juice production (From: Flottweg 2008)

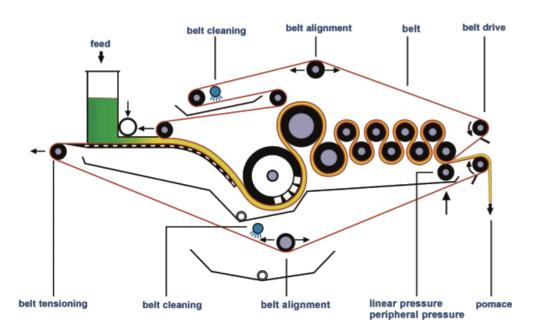


Figure 8.5: Schematic of Flottweg belt press for apple juice production (From: Flottweg 2008)

#### 8.4 Oilseed processing

Heavy duty screw presses have been widely used for expressing oil from appropriately prepared oilseeds. Oil in seeds is usually encased in small cells deeply embedded in fibrous structures. Preparation typically involves grinding or flaking to rupture the cells and heat treatment to help release the oil. The heat treatment also hardens pulverised plant tissue so it can withstand the pressure applied to squeeze out the oil (Williams 2005). Screw presses are used either to full press seeds or to pre-press seeds prior to solvent extraction (Bredeson 1978).

Continuous screw presses for oilseed processing were first successfully used around 1900 (Tindale and Hill-Haas 1976) and screw presses dominate the mechanical expression of oils (Williams 2005).

Notably, much higher pressures are reached in oilseed screw presses than in grape screw presses, with pressures sometimes reaching around 1000 bar (Bredeson 1978, Williams 2005). All high pressure oilseed screw presses feature an interrupted flight design together with interrupter bars protruding from the barrel (Williams 2005) unlike most grape screw presses, which have relatively continuous flights. An example of a partially disassembled oilseed screw press is presented in Figure 8.6. Williams (2005) describes the development and patenting (Anderson 1900) of the first (Tindale and Hill-Haas 1976) oilseed screw press in 1900 by Valerius Anderson. Anderson tested screw-conveyor-like devices with perforated walls, but whenever the discharge was restricted, the material would simply spin with the shaft. Anderson partially overcame this by the use of an interrupted flight design. The material would still tend to spin with the flighting but spinning would stop in the areas between flights, allowing the shaft to generate enough pressure to push the material against the discharge restriction. The introduction of interrupter bars that intermeshed with the flighting further helped prevent the material from spinning with the shaft.

To feed the pre-treated material to the main compression chamber and prevent return of material towards the hopper some oilseed screw presses feature a feed screw that force feeds the main screw (Ward 1976). Toothed anti-return wheels, conceptually similar to those used in many grape screw presses (see Figure 5.26), have also been employed (Bredeson 1978, Williams 2005).

One manufacturer's system for full pressing is presented in Figure 8.7. In this device a horizontal screw-conveyor controls the amount of material delivered to a vertical feed screw which then force feeds the main horizontal screw. The vertical screw rotates in the opposite direction to, and faster than, the main screw, and is sometimes equipped with a drainage cage (Williams 2005).

The widespread use of interrupted flights and interrupter bars in oilseed screw presses but not in grape screw presses, suggests that the rotation of the material with the screw shaft may be a bigger problem with screw pressing oilseeds than it is with grapes. Rotation of material with the screw could be a greater problem because of the more compact press cake, and faster screw press speed, which can apparently be in the order of 40 rpm (Bredeson 1978), compared with around 2 rpm in grape screw presses.

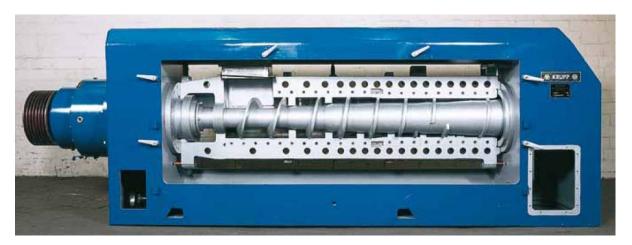


Figure 8.6: Partially disassembled oilseed screw press (From: Harburg-Freudenberger 2010)

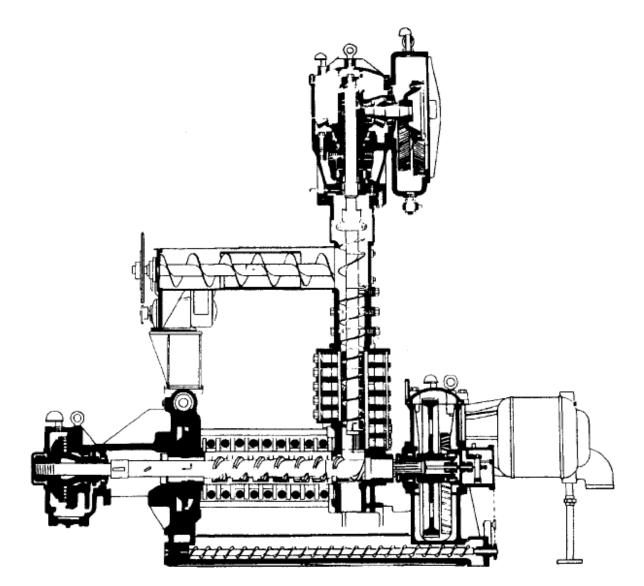


Figure 8.7: Anderson Super duo Expeller 55 with conditioner (From: Williams 2005)

#### 8.5 Sugar cane processing

Non-belt roller presses (referred to as mills) have been widely used to process sugar cane. Roller presses have not been employed in wine production, except for grape crushing. Unlike grapes, sugar cane has very strong cell walls requiring high pressures for rupture and pressures up to 400 bar may be reached in sugar cane mills (Schwartzberg 1983).

Sugar cane is prepared for milling (or for diffusion extraction) by size reduction, typically with rotating knives in the cane conveying system and/or passing the cane through a swing hammer shredder (Rein 2007). In earlier times, coarsely grooved roller crushers were also commonly used for cane preparation (Hugot 1986). The optimum preparation for subsequent milling is one where most of the sugar containing cells are ruptured, but long strands of fibre remain, not disintegration to a sawdust consistency (Rein 2007).

The objective of the milling process is to separate the sucrose-containing juices from the prepared cane. Milling is performed by passing the prepared cane through a tandem of around five mills. An example of one conventional three-roll mill is presented in Figure 8.8. Mills often have additional rollers to aid feeding (Rein 2007). At each mill, the sugar cane (also referred to as bagasse) passes between the feed and top roll and then out between the discharge roll and top roll. Juice is collected in a tray below.

Juice drainage is a key issue that influences sugar cane mill performance. If the juice is not drained sufficiently, lubrication of the rollers results, and there can be slipping and choking (Bonin and Govaert 1999). The necessity for good mill drainage is further enhanced by the common addition of fluid to increase the extraction of sugar, referred to as imbibition, which simultaneously increases the liquid load on the mill. The exit of liquid from pockets inside the layer of sugar cane itself also has a critical influence. Hugot (1972) and Rein (2007) report on extensive experiments performed with simple two-roll mills to better understand sugar cane milling. These have demonstrated that the sugar cane layer exiting the mill rolls has a larger volume than that generated at the pinch point by the two rolls. This phenomenon referred to as re-absorption, is illustrated in Figure 8.9, together with a typical pressure profile. As the sugar cane layer enters the rolls and approaches the pinch point, pockets of juice that cannot escape the sugar cane layer, will tend to flow back upstream through the sugar cane to the zones at lower pressure. Shortly before the pinch point, the free juice can no longer permeate backwards through the densely compacted fibre to escape. This juice in the tightly packed cane layer finds a zone of lower pressure ahead of it. From this point therefore, the juice flows forward through the fibre, travelling at a speed faster than the roll surface. The juice is quickly reabsorbed by the relatively dry expanding bagasse.

An interesting feature of mill rolls is their circumferential grooves. These grooves provide surface area for grip, assist in breaking up the cane and, as the cane can not pack into the bottom of the grooves, also provide a natural drainage channel for expressed juice (Rein 2007). Extending the concept of

juice draining through the bottom of the roller grooves, additional grooves have been explicitly included to provide passage for larger quantities of juice to drain. These juice drainage grooves, known as Messchaert grooves, are widely used on feed rolls (Hugot 1972). A diagram of mill roll surfaces illustrating this concept is presented in Figure 8.10. A further means of providing improved drainage is the provision of interior channels within a roller to transport juice from orifices at the base of the grooves on the roll surface. This concept was employed by Bouvet (1976, 1980) for a modified top roll, known as the Lotus roll (Figure 8.11), where Messchaert grooves could not work because juice expressed to Messchaert grooves in this roll would just drain back into the sugar cane. The Lotus roll has been tried in many facilities, however problems have been encountered with blockage of both the orifices and the interior channels, and as a result, there are few in service (Rein 2007).

A more recent configuration of sugar cane mill is the Bundaberg High Extraction mill as shown in Figure 8.12 (Batstone et al. 2001, Rein 2007). This uses two rolls on the same horizontal plane. Cane is fed vertically with juice draining co-currently on both rolls through deep Messchaert grooves. Notably, the configuration is somewhat similar to the roller crushers employed for grape crushing, the key difference being the simultaneous separation of juice during compression through the Messchaert grooves. This concept of co-current drainage through roller grooves is an interesting idea, which could possibly be adapted to grape processing equipment at a much smaller scale and with lower pressures than in sugar cane processing. Advantages of employing rollers for compression are that they can have a very small hold-up volume, can permit rapid processing and if arranged horizontally could allow several stages of rollers to be stacked one above the other giving a small footprint.

NOTE: This figure is included on page 137 of the print copy of the thesis held in the University of Adelaide Library.

# Figure 8.8: Traditional three-roll sugar cane mill (From: Rein 2007)

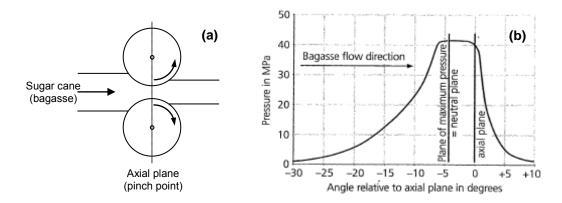


Figure 8.9: (a) Simple two-roll sugar cane mill model and (b) mill pressure profile (Adapted from Hugot 1972 and Rein 2007)

NOTE: This figure is included on page 138 of the print copy of the thesis held in the University of Adelaide Library.

Figure 8.10: Sugar cane mill roll surfaces with Messchaert grooves (From: Rein 2007)

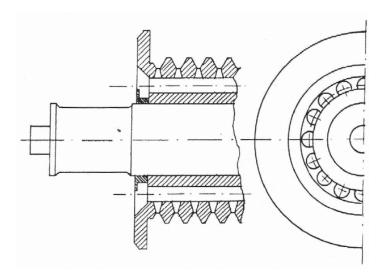


Figure 8.11: Sugar cane Lotus roller (From: Hugot 1986) NOTE: This figure is included on page 139 of the print copy of the thesis held in the University of Adelaide Library.

# Figure 8.12: Bundaberg High Extraction two-roll sugar cane mill (From: Rein 2007)

#### 8.6 Conclusions

Other i ndustries have generally employed similar techniques to those used (or at least trialled) in white wine production. One difference is a greater adoption of continuous equipment, other than screw presses. For example, belt presses have been much more widely used in apple juice production. Interestingly, screw presses used to process oilseeds and apples have generally featured interrupted as opposed to continuous flight designs. Roller presses with drainage grooves are widely employed in the processing of sugar cane. The concept of simultaneous drainage during compression between rollers is one that may possibly be adapted in some form to white grape processing.

One c ommon t heme in t he us e of c ontinuous ex pression equipment in d ifferent i ndustries is the importance of removing free liquid that can cause lubrication and choking. Another common theme is a trade-off in the degree of preliminary comminution. Small particles yield their contents more easily but larger particles provide greater structure to the cake.

# CHAPTER 9: FUNDAMENTAL STUDIES OF GRAPE JUICE EXPRESSION

The aim of the work presented in this chapter was to perform small-scale laboratory experiments in order to better understand grape juice expression. The longer-term goal was the development of expression equipment that could combine the throughput aspects of the continuous inclined drainer and screw press line with the quality obtained using a membrane press. Developments of this nature could provide a practical means by which wineries could manage phenolic levels while keeping production costs low and achieving the throughputs required.

To achieve high throughputs, the appropriate device is likely to be continuous. It was elected to perform constant rate compression experiments, in preference to constant pressure experiments as it was postulated that constant rate expression experiments were a better approximation to continuous devices with converging surfaces, such as roller or screw presses, that might ultimately be employed.

In order to achieve appropriate quality it was proposed that the most appropriate action would be one that mimicked a batch press; that is repeated cycles of compression and crumbling, ideally with minimal shearing action. Based on this reasoning, experiments were performed with repeated cycles of compression and crumbling.

## 9.1 Materials and methods

#### 9.1.1 Laboratory pressing apparatus

An Instron 1026 materials testing machine was used as the basis for construction of the pressing apparatus shown in Figure 9.1. The cross-head movement was used as intended by the manufacturer; however, a new 500 kg-F universal load cell (Dacell, South Korea) with a piston attachment was fitted in a bracket attached to the cross-head to allow for compression measurement. The load cell signal conditioning unit (Applied Measurement Australia, Australia) was connected to a data acquisition system, and monitored and recorded using a PC application written in Visual Designer 4.0 (Intelligent Instrumentation, USA).

The body of the unit consisted of a compression cylinder, which was located in a heavy duty mount. All metal parts in contact with grapes/juice were constructed from 316 stainless steel, while the mount was fabricated using carbon steel as a cost saving measure. The sieve plate was 6 mm thick and featured 2 mm  $\times$  20 mm slots with 5 mm spacing between the edges of each row and column of slots. An alternate sieve plate with 12 mm spacing between the edges of the rows of slots was also employed in a small number of experiments. Both sieve plates are shown in Figure 9.2. Gaskets were produced from 3 mm thick food grade nitrile rubber and a polyurethane piston sealing ring (Adelaide Seal Supplies, Australia) was employed. The main compression cylinder consisted of a 95 mm internal diameter acrylic tube, which enabled observations of juice channelling and cake condition. The use of a separate mount (Figure 9.3a) and compression cylinder (Figure 9.3b) ensured the compression cylinder was light and manoeuvrable to facilitate cake removal and crumbling.

During the 2008 vintage experiments, the volume of expressed juice was measured manually. To improve the apparatus, prior to the 2009 vintage a miniature 1 kg-F load cell (Dacell) was fitted in order to automatically weigh the expressed juice. This load cell was interfaced with the software in a similar manner to the piston load cell.

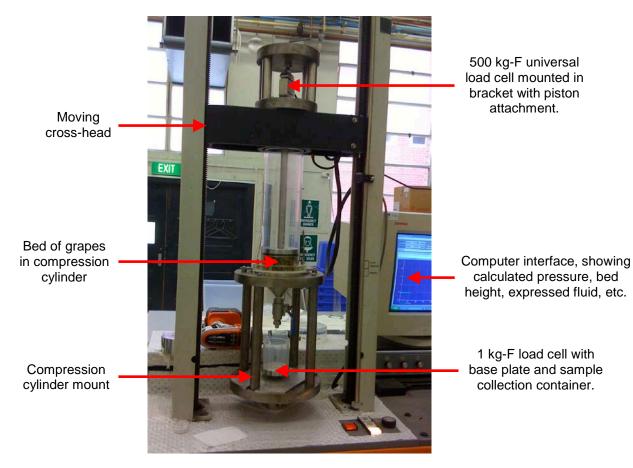


Figure 9.1: Laboratory pressing apparatus

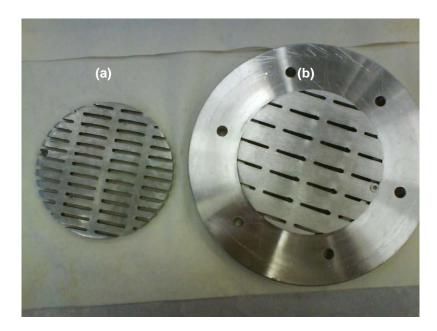


Figure 9.2: (a) Standard, and (b) alternate sieve plates



Figure 9.3: Laboratory pressing apparatus (a) mount, and (b) compression cylinder

#### 9.1.2 Experimental technique

The general procedure was as follows: First, a specific mass of grapes was manually destemmed. These grapes were then added to the compression cylinder, either whole or after a fraction of them had been manually pre-crushed in snap-lock LLDPE bags. The piston head was positioned to approximately 10 mm above the cake surface using a power-drill to rapidly drive the cross-head manual adjustment axle. The cross-head was then started at the constant speed set by the choice of Instron gears. Compression of the cake continued until there had been a rise in pressure of approximately 2 bar (based on the measured force, the known cross-sectional area and the frictional resistance estimated from the movement of the cross-head before engaging the cake). The piston head was then stopped, and the cake was allowed to relax for approximately 2 minutes before the piston was withdrawn. The juice valve was closed and the pomace was emptied from the cylinder into a PP container with lid. This was rotated several times in order to crumble the cake. The juice expressed during the cycle was treated with 100 mg/L potassium metabisulfite as a short-term preservative measure. The pomace was returned to the cylinder and the process of pressing, crumbling and juice sampling was repeated until insufficient juice could be obtained from the press cycle for sample analysis.

Experiments were performed over the 2008 and 2009 vintages with five different batches of grapes at commercial maturity (four batches of Chardonnay and one batch of Riesling). Grape picking and storage techniques were as outlined in Chapter 3. The 2008 vintage experiments were focussed on understanding the basic pressing phenomena, for which large bed heights and low pressing speeds were appropriate. The focus of the 2009 vintage experiments was on thinner press cakes and faster pressing speeds as it was hypothesised that this, to some extent, represented the operational conditions of an idealised continuous multi-stage expression device (i.e. low berry hold-up volume and high throughput).

Whilst numerous pressing experiments were performed, those intended for direct comparison were run in a randomised order soon after one another to mitigate any possible effects from grape deterioration during storage. The use of different bed heights was investigated by the use of different starting loads of grapes (125 – 2000 g), and pressing speeds were trialled between 10 and 100 mm/min. Pre-crushing all or a fraction of the berries was investigated as was the use of the two different sieve plates previously mentioned. After observation that pre-crushing produced an initial high solids juice fraction, back-addition and re-filtration of this juice through the pomace cake was also trialled.

#### 9.1.3 Sample analysis

On the conclusion of each pressing experiment, the pH and conductivity of the set of juice samples were determined as described in Chapter 3. The solids content and juice density were measured as summarised in Chapter 7. The remainder of each sample was clarified (5 minutes, 3,100 ×g, 4 °C) and the supernatant decanted into fresh 50 mL PP tubes. Juice samples were then treated with 900 mg/L potassium metabisulfite before distribution into 10 mL PP tubes and frozen storage (-20 °C) for later phenolic analysis. Later, thawed (4 minutes, 50 °C) samples were analysed for phenolic content by UV spectroscopy (Pharmaspec UV-1700; Shimadzu, Japan), following clarification by

micro-centrifugation (1.6 mL, 15 minutes,  $15,000 \times g$ ) or filtration (0.45 µm hydrophilic PP syringe filters with glass fibre pre-filters, Pall, USA). UV spectroscopy was performed with 1 mm path length quartz cells using an RO water reference. Spectral results were normalised to a 1 cm path length.

### 9.2 Results and discussion

#### 9.2.1 General observations

An example of typical pressure and expression profiles from a press cycle with 1 kg of whole berries is shown in Figure 9.4. Initially, there was little rise in pressure or juice expression as the advancing piston rearranged the berries. As it advanced further, some berries began to burst and the juice was observed to channel down through the cake and into the sample collection container. As the piston progressed even further, the channels increasingly narrowed, the speed of the channelling juice increased and the pressure rose rapidly, until the piston was stopped manually at approximately 2 bar. As soon as the piston was stopped, the pressure dropped quickly.

The behaviour observed was similar to that reported by Schwartzberg et al. (1977) for single pressing runs with spent coffee grounds, apple chunks and chopped alfalfa. Mechanical expression is a complex physical process (Saravcos and Kostaropoulos 2002), particularly so when it involves soft highly deformable biological tissues (Lebovka et al. 2003). The pressure applied during the compaction-induced expression of fluids from fluid-rich solids usually consists of compressive stresses, fluid pressure drop and sometimes friction with the compression cavity (Rebouillat and Schwartzberg 1986) and these forces interact (Schwartzberg et al. 1985). A diagrammatic representation of the forces on a grape or grape section in the initial stages of compression and in the later stages of compression, is presented in Figure 9.5. Initially, as shown in Figure 9.5a the cake was quite open with relatively few points of contact between the surrounding grape sections on section A. The space between the sections was occupied by air at atmospheric pressure. There can be relatively high local pressure differences applied across grape section A, without very high global pressure measurements at the piston surface. As compression proceeds the grape sections are deformed and there are more points of contact between surrounding grape sections and grape section A, which better support section A in all directions. Furthermore, any space between the grape sections is increasingly filled by liquid, which supports section A very evenly on all sides. Therefore to reach a sufficient local pressure difference across section A, to expel juice from it, there will need to be a much larger pressure drop across the entire cake, given the support of grape section A by the surrounding grape sections and fluid and the fluid pressure drop across the cake from the increasingly narrowing channels.

Figure 9.6 shows the pressure and juice expression profiles for the experiment presented in Figure 9.4 for repeated cycles of pressing and crumbling. With further press cycles the load increasingly consisted of already partially juiced grape sections and there was a less gradual rise in pressure than

there had been with the whole grapes, which had provided a cake structure with relatively open channels through which expelled juice could escape.

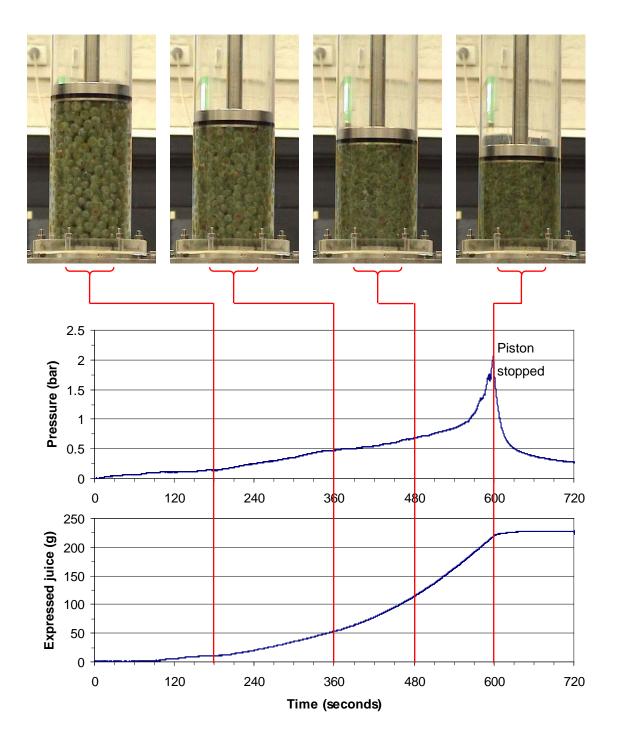


Figure 9.4: Pressure and juice expression profile during the first press cycle of 1 kg of whole 2009 vintage Barossa Valley Chardonnay grapes at 10 mm/min (with corresponding photos of pomace)

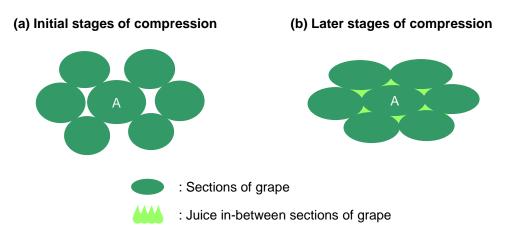


Figure 9.5: Representation of part of the cake structure during different stages of compression

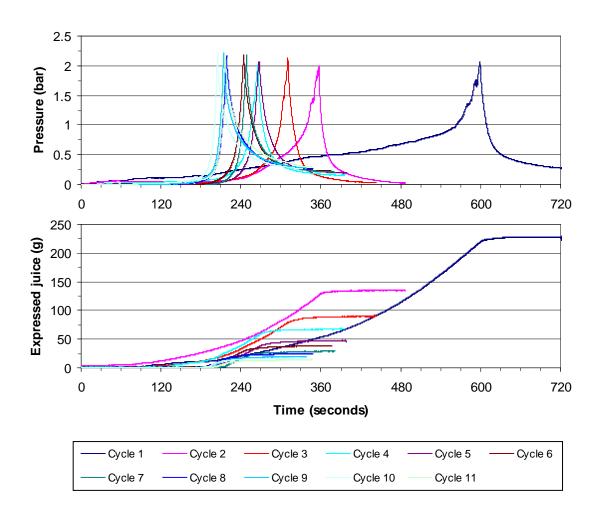


Figure 9.6: Pressure and juice expression profile during repeated press cycles of 1 kg of whole 2009 vintage Barossa Valley Chardonnay grapes at 10 mm/min

The analytical and yield results for the pressing experiment shown in Figures 9.4 and 9.6 are reported in Table 9.1. Diminishing juice yields with increasing press cycles was a feature seen in all experiments. This is to be expected as the juice easily expelled from the large weak intermediate pulp

cells has increasingly already been collected and further juice has to be collected from other zones of the grape.

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	210	210	4.73	2.95	3.31	3.74	0.8%	22.4
Cycle 2	128	338	5.67	3.33	3.60	3.82	1.6%	22.5
Cycle 3	83	421	6.42	3.70	3.80	3.93	1.4%	22.4
Cycle 4	60	481	7.22	4.17	3.97	3.97	1.6%	22.4
Cycle 5	43	524	7.77	4.55	4.29	3.91	1.4%	22.3
Cycle 6	36	560	8.15	4.71	4.38	4.05	1.3%	22.2
Cycle 7	27	587	8.15	4.61	4.54	4.05	1.5%	22.2
Cycle 8	23	610	8.23	4.53	4.64	4.08	1.3%	22.1
Cycle 9	19	629	8.34	4.36	4.76	4.12	n.d.	n.d.
Cycle 10	16	645	8.44	4.32	4.94	4.07	n.d.	n.d.
Cycle 11	14	659	8.16	4.02	4.97	4.15	n.d.	n.d.
-								

Table 9.1: Analytical results for samples collected during pressing of 1 kg of whole 2009vintage Barossa Valley Chardonnay grapes at 10 mm/min

n.d.: not determined.

The phenolic content, as estimated from the spectral absorbance at 280 nm and 320 nm, tended to increase in each juice fraction, as did pH and conductivity. This was a typical pattern, irrespective of the grape variety and experimental conditions. The results presented in Table 9.1 should be considered in the context of the chemical and mechanical composition of the grape, as discussed in section 2.2. These analytical measurements correspond with increasing contribution of juice from towards the skins and/or from the skins. The overall trend of increasing phenolics, pH and conductivity with yield are the same as observed in previous large scale studies (Terrier and Blouin 1975, Lemperle and Kerner 1978, Desseigne et al. 2003).

In some of the laboratory pressing experiments, the  $A_{320}$  reached a maximum and then started to decrease with subsequent pressing cycles. This trend was also seen in the  $A_{280}$ , after a lag period. While this trend could potentially be a result of reduced extraction of hydroxycinnamates from the skins at higher yields, this appears unlikely given that it was occurring at too low a yield for all the skin cells to have been sufficiently mechanically damaged to release the hydroxycinnamates contained within. It seems more likely to be a result of increased oxidation and precipitation of hydroxycinnamates reducing the concentration.

#### 9.2.2 Crumbling

Figures 9.4 and 9.6 demonstrate the rapid rise in pressure with increasing compression, and suggest that crumbling needs to be performed to achieve sufficient juice yields without the application of very high pressures. However, the pressure did quickly dissipate when the piston was stopped, and therefore it was thought worthwhile for the future design of equipment to verify the importance of

complete cake crumbling as opposed to a period of relaxation before re-starting compression. This was investigated by comparing the juice yields for experiments with normal crumbling and without crumbling where the piston was just stopped for approximately 9 minutes before resuming compression. The results are presented in Figures 9.7 and 9.8 for whole grapes and for pre-crushed grapes, respectively. Much higher yields were obtained with crumbling. Without crumbling, as soon as the piston movement resumed, the pressure transient rose rapidly. The rapid reduction in pressure after stopping piston movement is probably related to the expression of a small mass of the essentially incompressible fluid that occupied the space in-between the grape sections. Only a small volume of fluid had to be expelled from grape sections on resumption of piston movement to replace that fluid.

Proper crumbling in constant-rate devices whereby juice saturating the pores between sections is removed will therefore be a practical requirement after a compression step, unless high pressures are to be applied. Crumbling can also serve to clear the sieve plate and rearrange the grape sections so that sections from which disproportionately little juice had been expelled from as a consequence of their previous location or orientation in the cake are re-positioned.

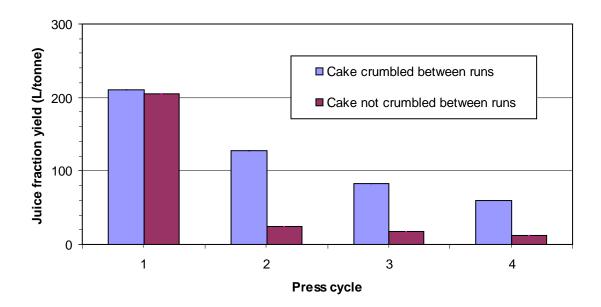


Figure 9.7: Yield comparison with and without crumbling for pressing of 1 kg of whole 2009 vintage Barossa Valley Chardonnay grapes at 10 mm/min

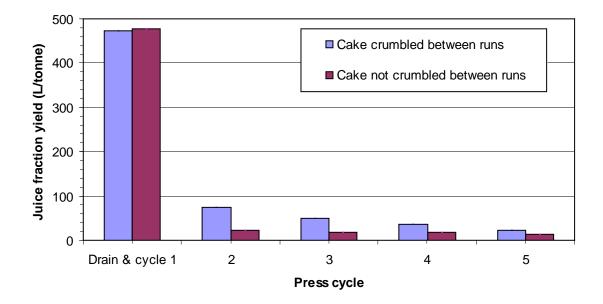


Figure 9.8: Yield comparison with and without crumbling for pressing of 1 kg of 100% precrushed 2009 vintage Barossa Valley Chardonnay grapes at 10 mm/min

#### 9.2.3 Bed height

Figure 9.9 explores the effect of bed height on solids and phenolic content for initially whole grapes. Variable width column charts have been used, with each column representing a pressing step. It should be noted that the final yield does not necessarily correspond with the end of the last column as for latter samples, there was often insufficient juice for solids or phenolic measurement. With smaller cakes, a higher yield was generally obtained in fewer steps. However, the solids content was also higher. While phenolic content always increased with increasing yield, there was no clear trend with different bed heights at the yields studied. Similar trends were observed with other grape lots.

Solids may be released as the juice is expelled from grape sections and they may be filtered from this expelled juice as the juice passes through the remainder of the cake towards the juice exit. With deeper bed heights juice is expelled from grape sections more gradually as the deformation of the grape sections in the cake cushions against the movement of the piston. This gradual expelling of juice from grape sections means that the juice is actually filtered as it leaves the relatively intact grape. For the earlier juice fractions, it would seem most likely that the dominant mechanism controlling juice solids content is related to the expelling of juice from grape sections as opposed to filtration of this juice by passage through the remainder of the cake. With relatively intact grapes the cushioning effect is maximised, but the open cake structure created by these relatively intact grapes would be expected to be a poor filter. For later juice fractions, filtration during passage through the cake could potentially start to become an important mechanism in controlling expressed juice solids content, given the decreased cake porosity.

The influence of bed height (for different pressing speeds) on juice yield, for experiments performed during the 2009 vintage with initially whole Langhorne Creek Chardonnay grapes is reported in Figure 9.10. With smaller press cakes, higher yields were derived in significantly fewer steps than with larger cakes – an important practical point with respect to the development of juice expression equipment. At a given pressure, a smaller cake results in a higher pressure applied per unit height and across individual grape sections, so it is not unexpected that that there is increased juice expression. Experiments with 125 g of berries (1.8 g/cm<sup>2</sup> cross-sectional area) showed that it may be possible to collect around 600 L/tonne (a typical yield for high-quality juice in current membrane presses) in three to five stages of high-speed constant-rate compression. However, as discussed previously, the trade-off is a higher suspended solids content in the juice when compared with deeper cakes.

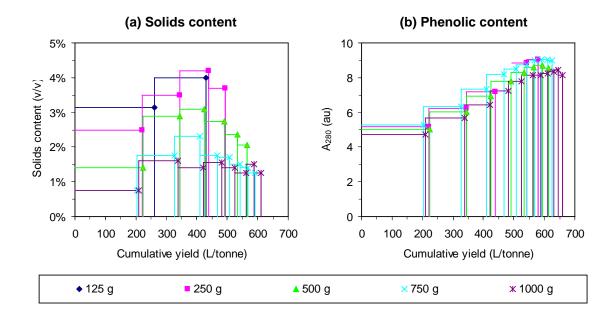


Figure 9.9: Effect of bed height on solids and phenolic content for whole 2009 vintage Barossa Valley Chardonnay grapes pressed at 10 mm/min

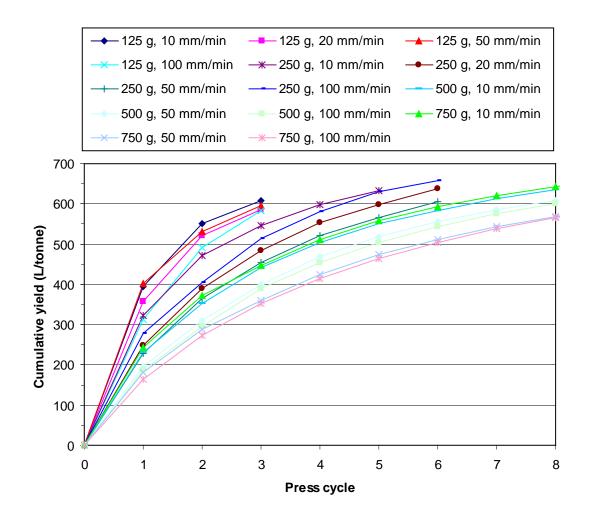


Figure 9.10: Effect of bed height and press speed on juice yield for whole 2009 vintage Langhorne Creek Chardonnay grapes

#### 9.2.4 Pressing speed and sieve plate design

Figure 9.11 explores the effect of pressing speed on solids and phenolic content for initially whole grapes. Faster pressing speeds resulted in higher solids levels and lower yields at each pressing step. While phenolic content always increased with increasing yield, no clear trend was evident with different pressing speeds at the yields studied. At higher pressing speeds the juice is expelled more violently from the grape sections. As well, some expelled juice was trapped in the cake as the channels quickly closed, and when the piston was removed, this juice that had not been subject to cake filtration trickled out.

Extrusion of solids through the sieve plate is also likely to contribute considerably to the juice solids content at faster pressing speeds, particularly with thinner cakes. This is a probable explanation of the varied solids measurements when compared with experiments performed at the relatively slow speed of 10 mm/min. The effect of pressing speed and sieve plate design on solids content for 125 g of initially whole Adelaide Hills Chardonnay grapes is presented in Figure 9.12. Once again, higher

pressing speeds produced significantly increased solids content. Generally, across a number of experiments with several different bed heights and pressing speeds, the type of sieve plate used (i.e. the standard one or the alternate one with half the open area) produced no consistent effect on solids content. However, the yields obtained at each pressing step were typically lower with the alternate sieve plate with only half the open area. The small number of slots in this sieve plate were likely blinded earlier than with the standard sieve plate, resulting in the cycle cut-off pressure of 2 bar being attained at a lower expressed yield.

The faster pressing speeds explored in this work and likely even higher speeds, would be required in a practical high speed juice expression device. The apparent large quantity of extruded pulpy solids with fast pressing speeds suggests that sieve plate apertures of this size would not be appropriate for rapid devices acting on small cakes. The sieve plates used in this experiment were designed after inspection of a number of membrane presses from several different manufacturers that typically featured slots approximately 2 mm wide. For future devices, it is recommended that smaller slots be trialled as these could provide greater support for the grape sections and perhaps limit extrusion through the sieve plate slots. This could however increase blinding. The use of apertures that increase in size from feed to exit as employed in many drainers and presses may partly assist in managing this problem. The extrusion of solids may potentially be further limited by not having the screen parallel to the advancing compression face (in this case, a piston). It seems that this acted to drive large solids through the sieve plate slots at fast pressing speeds. In traditional basket presses and horizontal moving plate presses, the draining screen is perpendicular to the direction of compression, and this general principle may be worth replicating in the construction of new juice expression devices to limit the extrusion of solids and screen blinding.

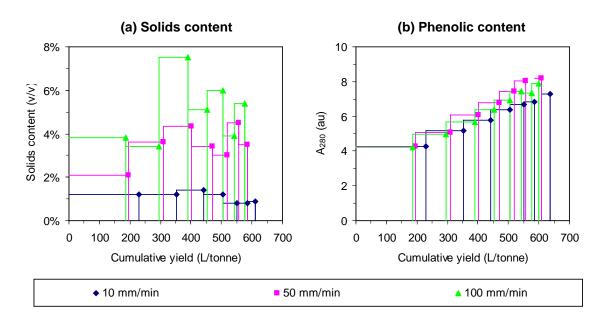


Figure 9.11: Effect of pressing speed on solids and phenolic content for 500 g of whole 2009 vintage Langhorne Creek Chardonnay grapes

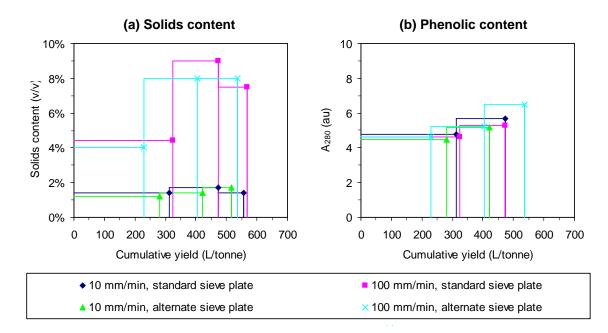


Figure 9.12: effect of pressing speed and sieve plate design on solids and phenolic content for 125 g of whole 2009 vintage Adelaide Hills Chardonnay grapes

#### 9.2.5 Whole or crushed berries

As discussed in chapter 4, grapes are typically crushed before draining and pressing (either explicitly or as a side-effect of machine harvesting, destemming, must pumping and chilling), to allow the collection of juice as quickly as possible. Figure 9.13 shows the cumulative yield profile for repeated pressing cycles of Riesling grapes where different fractions of the grapes had been pre-crushed. It reiterates that with pre-crushing, a higher yield of juice can be liberated more quickly. A comparison of

the influence of pressing whole or pre-crushed berries is presented in Figure 9.14. Pre-crushed berries exhibited significantly higher juice solids content initially that is not present if the juice is expressed from a bed of initially intact berries. The reason for this is illustrated in Figure 9.15. As shown in Figure 9.15a when crushing, the grape is contacted from two sides only so it will deform and then quite violently rupture releasing the contents and in this process pulpy solids. In comparison, as shown in 9.15b, the compression of whole grapes in a cake will be more gradual with several points of contact between the grape and the surrounding grapes and then very uniform support from the expelled fluid. These will act to provide small local pressure differences across the grape and the contents will be released gradually.

Given that pre-crushing is the easiest way to quickly collect a large volume of juice and the fact that the cake may act to some extent as a filter, it was postulated that a possible method of rapidly collecting low solids juice may be to pre-crush the grapes to release the juice, and then to filter this high solids juice back through the cake.

Experiments were performed with 500 g each of 2009 vintage Langhorne Creek and Adelaide Hills Chardonnay grapes to explore this concept. The drained juice was collected and was back-added after one pressing cycle. For the Langhorne Creek Chardonnay, this was performed with a crumbled cake and with a non-crumbled cake (piston just removed). As shown in Figure 9.16, the crumbled cake was an ineffective filter, whereas the intact non-crumbled cake was effective to some extent, reducing the solids content in that fraction from approximately 6% to 4% for the Langhorne Creek Chardonnay. For the Adelaide Hills Chardonnay under the same conditions, back-addition reduced the solids in this juice fraction from approximately 5% to 3%. The pressure required to drive the juice through the approximately 25 mm thick pomace cake in both instances was provided by compression of the air above the cake surface as the piston was moved into position. This confirmed the concept that juice could be separated and refiltered through the cake. However, the magnitude of solids reduction is unlikely to be sufficient with cake thicknesses of this height; and low cake heights of this order are desirable for rapid juice expression.

To achieve relatively low solids contents without large bed heights, the most effective means of expression may be to keep the berries relatively intact for as long as possible to take advantage of the structure of the grape to retain solids during juice expulsion as opposed to relying on cake filtration. This is the same concept as employed in whole cluster pressing of hand-picked grapes (to some extent; grapes still have stems in whole cluster pressing), which is widely acknowledged to produce lower solids contents (e.g. Jung and Seckler 1996). This is why the focus of the experiments performed in this chapter was on the use of whole as opposed to crushed grapes. One challenge in implementing this concept is whether machine harvested grapes would be sufficiently intact to filter the juice as it is expelled from the berry. The other major challenge is a configuration of device that allows pressure to be applied rapidly but sufficiently uniformly around the grape such that juice is actually expelled with low solids content, similar to traditional whole cluster pressing. This was not

achieved with small cake heights and fast pressing speeds in the current experiments. As discussed in section 9.2.4, configurations with alternate screen apertures and different relative orientation of screen and compression surface may help to rectify this problem.

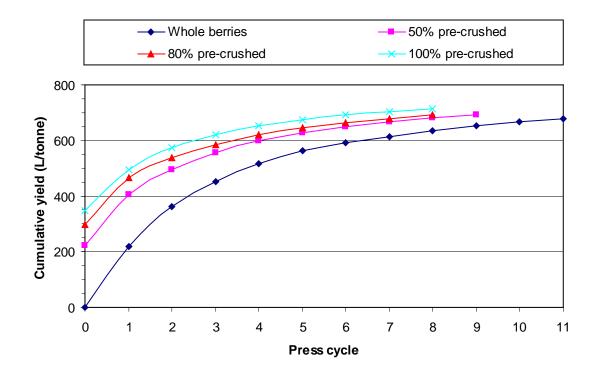
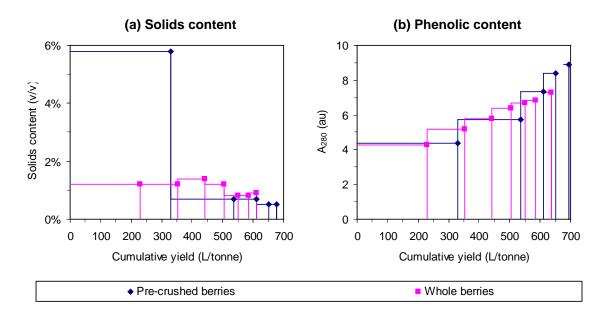
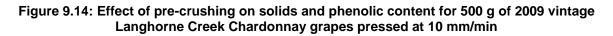


Figure 9.13: Influence of the fraction of pre-crushed berries on cumulative yield for 1 kg of 2008 vintage Eden Valley Riesling pressed at 10 mm/min





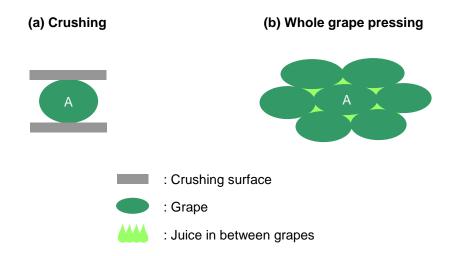
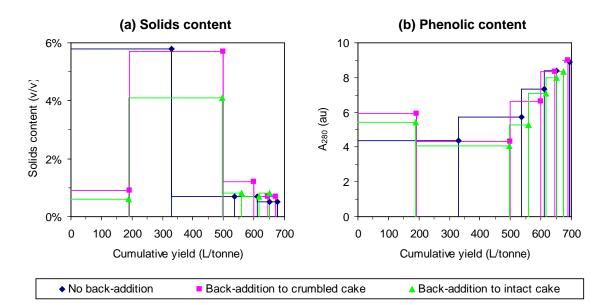
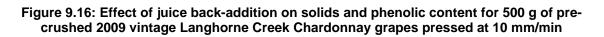


Figure 9.15: Representation of juice expellation under different regimes





#### 9.2.6 Constant rate and constant pressure compression

In comparison with membrane presses, it must be reiterated that these experiments were conducted at constant rate. Pressing conditions in winery membrane presses are probably better represented by constant pressure experiments. Constant rate experiments may partially represent their operation as the membrane fills with air to achieve a new pressure set point or to maintain the existing pressure set point as juice is evacuated from the chamber.

Generalised pressure and juice yield profiles under constant rate and constant pressure regimes are illustrated in Figure 9.17. Initially, the grape mixture is a three phase system as a consequence of the trapped air. As the air is removed, the juice expression rate eventually becomes constant under a constant rate regime, but diminishes with time for the constant pressure regime. The constant pressure regime is more effective at preventing the closure of channels in the press cake, provided the pressure set point is not excessively high. To prevent excessive pressing times with constant pressure regimes, the approach is to increase the pressure when the flow rate diminishes or to crumble the cake before further pressure application. Ultimately, stages of constant pressure compression means that more juice can be expressed with a lower maximum pressure. However, in terms of juice quality higher pressures do not necessarily result in lower quality juice as measured pressure across the cake is largely influenced by the cake thickness and the local stresses acting on the grape sections in the cake may in fact be quite low. However, given that there will be a distribution of components in the cake with different cellular structures, it is probably fair to say that with higher overall pressures there may be increased risk of damage to components and there can also be mechanical constraints when working with higher overall cake pressures as discussed in section 5.1.3 for horizontal plate presses.

Diminishing yields were observed with increasing numbers of constant rate press cycles (see Figures 9.10 and 9.13). While several stages of constant rate compression may provide a viable strategy for lower yields, it would be extremely difficult to achieve overall yields in the order of those typical of membrane press without the use of much higher pressures and/or many stages of compression, perhaps so many that the number may become impractical.

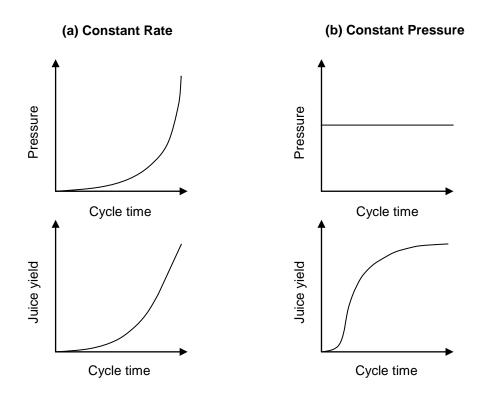


Figure 9.17: Generalised pressure and juice expression profile under constant rate and constant pressure compression for an individual press cycle

# 9.2.7 Analytical implications for drainer/press development, optimisation and control

As presented in Table 9.1, the trend of the results indicated that the phenolic content (as shown by  $A_{280}$ ), conductivity and pH increased with increasing yield fractions.

Simplistically, during the operation of membrane presses or other juice expression devices, key analytical parameters are the solids and phenolics contents, because of their influence on quality as well as downstream processing costs for solids removal and the amelioration of phenolic flavours. For practical use, analytical tools need to be rapid. As discussed in Chapter 7, assessment of phenolic content simply by UV absorbance at 280 nm is one of the more rapid techniques available, but it still involves sample clarification and possibly dilution before spectral analysis. Given that the pH and conductivity were shown in this work to increase with phenolic concentration, they could potentially be used as an indirect indication of phenolic content. Plots of  $A_{280}$  against conductivity and pH for juice samples from laboratory pressing experiments are presented in Figures 9.18 and 9.19 respectively, and a summary of the coefficients of determination for these plots is given in Table 9.2.

Relationships between  $A_{280}$  and conductivity and pH differ for the different grape lots, reflecting the results presented in Chapter 7. The coefficients of determination demonstrate a similar or slightly better relationship of  $A_{280}$  with conductivity than with pH. Desseigne et al. (2003) considered different

analytical parameters for use in drainer/press optimisation. They noted some difficulty in the practical implementation of pH measurement including the need for regular calibration of probes. They advocated the use of conductivity in the investigation of press operations as it is simple, inexpensive and has a fast response time. They also found a strong correlation ( $R^2 \sim 0.9$ ) between conductivity and pH, potassium and total phenolics.

The results presented in this chapter further support the idea that conductivity is a useful tool in optimising press operation and for investigating different juice expression equipment.

It must be cautioned that the relationship with phenolics can vary, even between batches of the same variety, and thus the use of absolute measurements for drainer/press control will not generally be possible and instead changes in conductivity would likely need to be used. Additionally, one must be aware of the direct contribution of additives to conductivity. For example, whilst typical additions of potassium metabisulfite appear unlikely to contribute drastically to results (100 mg/L potassium metabisulfite additions to juice samples contributed < 0.10 mS/cm in lab experiments), large poorly distributed additions of potassium metabisulfite may cause conductivity spikes, as seen at Winery A in Chapter 7 at the destemmer/crusher run-off.

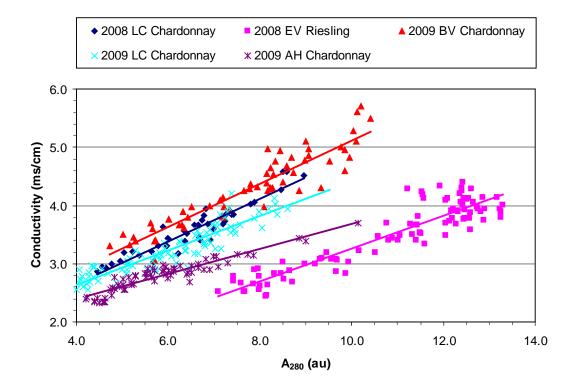


Figure 9.18: Relationship between conductivity and A<sub>280</sub> for juice samples collected during 2008 and 2009 vintage laboratory pressing experiments

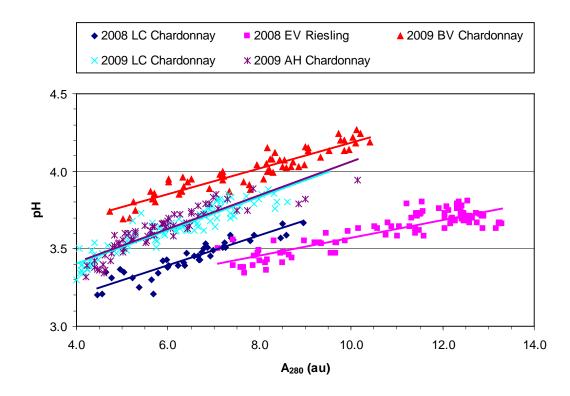


Figure 9.19: Relationship between pH and A<sub>280</sub> for juice samples collected during 2008 and 2009 vintage laboratory pressing experiments

Table 9.2: Coefficient of determination (R <sup>2</sup> ) between A <sub>280</sub> and conductivity or pH for juice
samples collected during 2008 and 2009 vintage laboratory pressing experiments

Grape lot	n	A <sub>280</sub> vs. conductivity	A <sub>280</sub> vs. pH	
2008 Langhorne Creek Chardonnay	41	0.90	0.85	
2008 Eden Valley Riesling	94	0.84	0.76	
2009 Barossa Valley Chardonnay	59	0.86	0.86	
2009 Langhorne Creek Chardonnay	86	0.84	0.86	
2009 Adelaide Hills Chardonnay	68	0.87	0.80	

#### 9.3 Conclusions

Repeated constant rate pressing of grapes produced fractions with increased phenolic levels, conductivity and pH. Crumbling was necessary to achieve reasonable yields without the application of high pressures. With whole grapes, thinner press cakes allowed more rapid collection of comparable yields, but unfortunately this was accompanied by increased juice solids contents. Faster pressing speeds, a likely practical requirement for high throughput devices, also resulted in higher solids in the juice.

Juice was more rapidly collected if grapes were crushed; however this strategy produced a large initial fraction of juice with high solids content. Back-addition and re-filtration of this juice through the pomace cake reduced the solids content of this fraction to some extent, but for cakes sizes of the

order required for practical operation, this re-filtration is unlikely to be sufficient. A better means for achieving low solids may be to maintain the structure of the grape in order to expel low solids juice directly from the berry so that cake filtration is not required. However, for this to be successful, pressure would need to be applied more uniformly on grapes than was achieved in these experiments when using small cakes coupled with high pressing speeds.

Fractional juice yields diminished with each pressing step. It was difficult to obtain high yields with repeated constant rate expression, much more so than it is with repeated constant pressure expression in batch membrane presses. If issues regarding high juice solids contents could be overcome, it may be possible to develop improved continuous equipment to express juice with total yields in the order of 600 L/tonne (typically the high-value juice fraction in any case), using three to five stages of rapid approximately constant rate expression. To achieve higher yields, either some form of staged constant pressure expression, like the membrane press or an essentially constant rate device that employs much higher pressures, such as a screw press, would likely be required.

In the development, optimisation and control of draining and pressing equipment, conductivity is a useful rapid analytical tool. A significant advantage of conductivity is that it is relatively inexpensive, well established in many industries and can be trialled without long analytical technique research lead times.

## **CHAPTER 10: KEY FINDINGS AND RECOMMENDATIONS**

Based on the work performed in this study, key findings and recommendations in three broad areas were developed. In section 10.1, recommendations with respect to harvesting and transportation are made based principally on work performed in Chapter 3. In section 10.2, recommendations related to the operation of existing juice expression equipment are presented. In section 10.3, recommendations and considerations for the development of new juice expression equipment are made. Recommendations presented in sections 10.2 and 10.3 are drawn primarily from the work presented in Chapters 4 to 9.

#### 10.1 Harvesting and transport prior to winery processing

- (a) The wine industry is correct to be cautious about the elapsed times between machine harvesting and winery processing. Where possible, time between harvesting and winery processing should be minimised in order to prevent increased phenolic levels in the highvalue low yield juice.
- (b) The fraction of grapes damaged during machine harvesting has a large influence on phenolic extraction into the high-value low yield juice. It is therefore desirable to minimise grape damage during harvesting and transportation.
- (c) Given the uncertainty surrounding the level of berry breakage in industrial practice, wineries should perform full-scale trials where economic advantage could be gained from relaxing restrictions on times between harvesting and winery processing.
- (d) Where possible, harvesting should be performed when it is cooler, as expression from warmer grapes appears to produce higher phenolic levels in the high-value, low yield juice.
- (e) Must chilling should be systematically investigated at full scale to evaluate whether chilling at the winery is a worthwhile remedial action for already warm grapes.

#### **10.2 Operation of existing juice expression equipment**

(a) Wineries should spend time and effort optimising their membrane press programmes to obtain the correct balance between quality and productivity for different types of grapes. "Intelligent programmes" that adapt their programmes in real time based principally on assessment of juice flow rate are one tool that should be considered to manage variations in feedstock.

- (b) Given the often stark differences between the commercial value of low-yield ("free-run") and high yield ("pressings") juices, the correct division of these fractions is economically critical and should be considered carefully. Division of fractions by real-time tasting of juice directly from press troughs is likely to lead to overly conservative divisions of juice and lost revenue.
- (c) The large differences in the value of free-run and pressings juice, suggest that press programmes and the extent of expression should be considered carefully for different grades of raw material. There may be merit in the use of different programmes for different grades of grapes.
- (d) Electrical conductivity measurement is a simple, readily available tool that can be very useful in the investigation and quality control of draining and pressing operations. Tracking of conductivity trends across many batches of grapes can aid in membrane press programme optimisation and assist in the determination of yield press cut points for different types, sources and conditions of grapes.
- (e) Wineries should be mindful of the validity of equipment comparisons, particularly when financed by manufacturers. The yield in each fraction and the analytical results for that particular fraction are ultimately the key juice quality performance indicators. Associations of results with press pressures can be extremely misleading. Furthermore, in membrane press use, the specific programme has a significant influence on quality and productivity, and as such, comparison between models can be erroneous if the operation of one model is optimised to a greater extent than the other.

#### **10.3 Development of new juice expression equipment**

- (a) Modern pneumatic presses are technically mature pieces of equipment. They are produced by many manufacturers and are widely employed for quality white wine production. Their main disadvantage is low throughput, which is why there remains room for innovation. The ideal press would be one that combines the throughput of the continuous inclined drainer and screw press line with the quality (particularly low solids and low phenolic levels) obtained with a membrane press.
- (b) Juice solids content is a critical issue in the design of new equipment. It is easiest to express low solids juice in devices with large bed heights and exploiting stages of constant pressure compression, similar to the operation of a membrane press. However, this combination of operations is not generally consistent with high throughputs and low hold-up volumes.
- (c) Thin press cakes and fast compression speeds will likely be necessary for small footprint, rapid juice expression, but these actions will tend to result in higher levels of juice solids. Refiltration of high solids juice through the pomace may allow for some solids removal, but it is

unlikely to be sufficient. A better means of obtaining low solids juice may be to maintain the structure of the berry for as long as practicable such that low solids juice is expelled directly from the berry in the first instance without requiring cake filtration to remove solids, in a manner conceptually similar to whole cluster pressing. This will potentially allow for the use of smaller cakes, but will have associated difficulties in applying pressure sufficiently gently to expel low solids juice directly from the berry, particularly if a substantial fraction of the grapes are fractured following machine harvesting.

- (d) Crumbling is an important means to achieve reasonable yields without high pressures or processing times.
- (e) If issues regarding high juice solids contents could be overcome/managed, it may be possible to develop continuous equipment to express juice in the order of 600 L/tonne (typically the high-value juice fraction in any case), using three to five stages of rapid, approximately constant rate expression. To achieve higher yields, either some form of staged constant pressure expression, like a membrane press or a device that employs much higher pressures, such as a screw press, would likely be required.
- (f) Screens with smaller slots than the common 2 mm wide slots found in membrane presses, may be useful in continuous devices to provide greater support for the grapes and prevent their extrusion through the screen apertures. However, with smaller slots, issues such as blinding will need to be appropriately managed. Furthermore, it may be advantageous if the screens are not located parallel to compression faces, as this may promote blinding or extrusion of grape solids through the apertures.
- (g) In devices involving high speed moving parts such as rollers or screws, it may be advantageous to remove free juice as soon as possible. Free juice is likely to lubricate surfaces and cause slipping and choking that acts to reduce throughput and generate suspended solids.
- (h) In devices involving high speed moving parts, such as roller presses, the development of an appropriate seal on each compression will be critical to ensure sufficient pressure can develop such that juice is actually expressed and not just expelled and immediately reabsorbed.
- (i) One possible means of applying several stages of compression is an adaptation of the roller mills used for sugar cane processing, which employ drainage grooves. In one plausible implementation, several stages of sets of horizontal rollers could be arranged above one another and integrated with a destemmer (in a similar manner to the current integration of destemmers and roller crushers) potentially allowing rapid continuous collection of the high-

value juice fraction (perhaps 600 L/tonne). In the development of any device of this nature, the specific roll design will be critical. There are also likely to be issues with solids contents, slipping and choking of rollers and cleaning that would need to be overcome and which would ultimately determine its success.

- (j) Development of devices with a very small footprint and low-holdup volume may ultimately allow for their mounting directly on machine harvesters, potentially simultaneously eliminating problems with phenolic extraction during transport between the vineyard and winery.
- (k) Despite quality-related problems, particularly high solids contents, the configuration of inclined drainers and screw presses are very convenient. High throughputs can be processed and material is simply loaded into the hopper with juice and dry marc being produced with little operator intervention. It is possible that the quality level obtained with current designs may be further improved upon. The use of different screw configurations and multiple antireturn devices to try and mimic the multiple stages of essentially non-shearing compression and crumbling of batch membrane presses is of interest. This subject should be further investigated. Initially the preferred approach would be laboratory studies with a purpose-built laboratory screw press that could be easily reconfigured to investigate the influence of potential design modifications.

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### **APPENDIX A: ADDITIONAL DATA FOR CHAPTER 3**

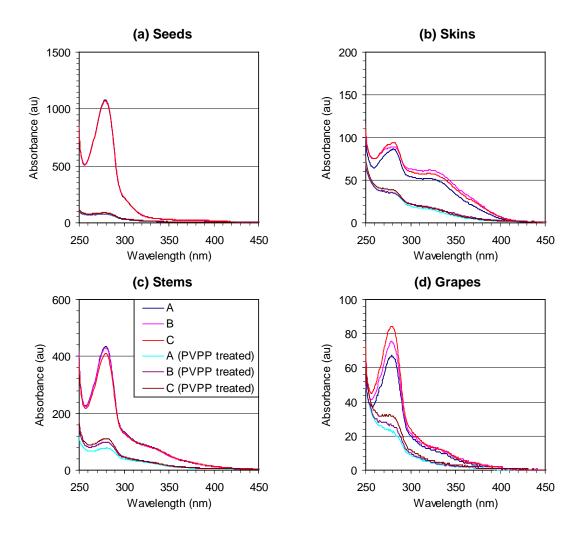


Figure A.1: UV/Vis spectra of Barossa Valley Chardonnay component extracts (with and without PVPP treatment) on a component basis (Replicates A, B and C)

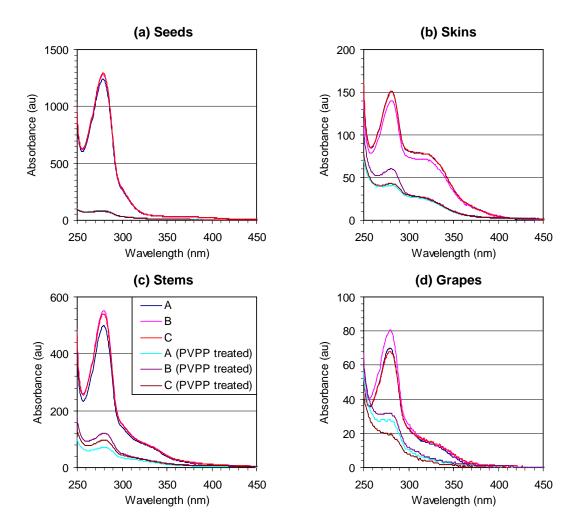


Figure A.2: UV/Vis spectra of Langhorne Creek Chardonnay component extracts (with and without PVPP treatment) on a component basis (Replicates A, B and C)

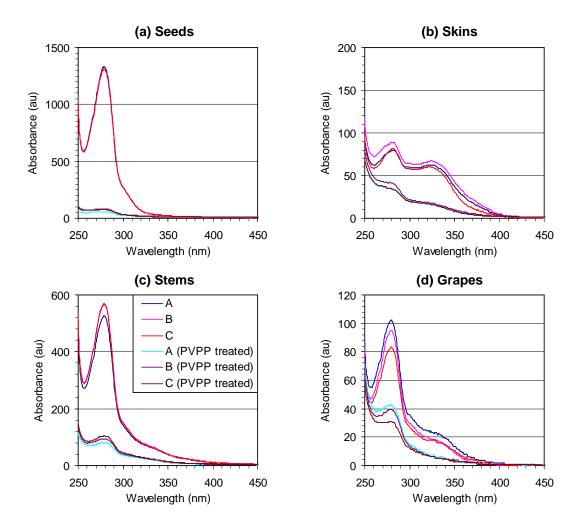


Figure A.3: UV/Vis spectra of Eden Valley Riesling component extracts (with and without PVPP treatment) on a component basis (Replicates A, B and C)

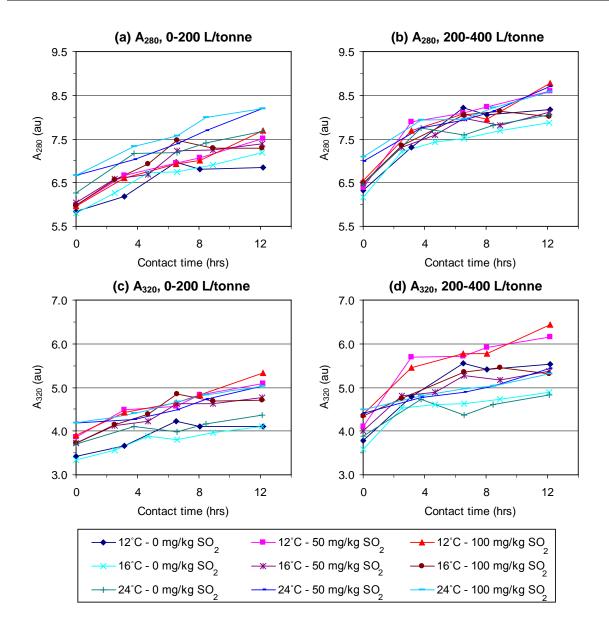


Figure A.4: Extraction kinetics for Barossa Valley Chardonnay, 100% crushed (Replicate 1)

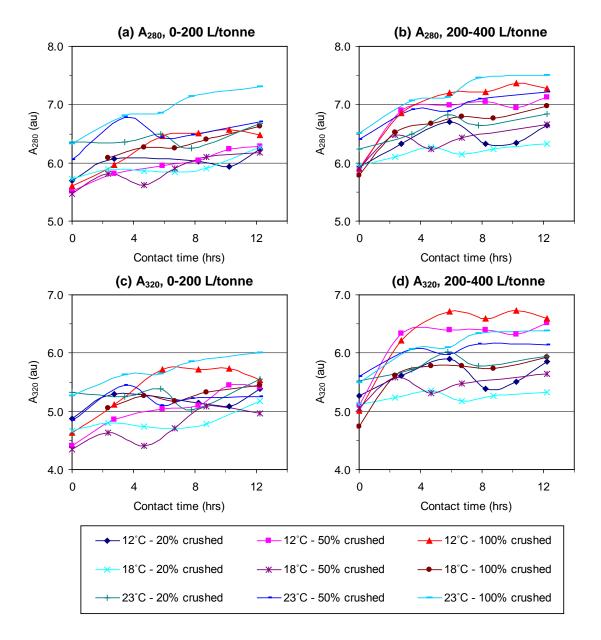


Figure A.5: Extraction kinetics for Langhorne Creek Chardonnay, 50 mg/kg SO<sub>2</sub> (Replicate 2)

#### **APPENDIX B: ADDITIONAL DATA FOR CHAPTER 7**

Sample <sup>a</sup>	A <sub>280</sub> (au)	A <sub>320</sub> (au)	Conductivity (mS/cm)	Solids (v/v)	RI (°Brix
	(au)	(au)	(mo/cm)	(V/V)	
Destemmer-crusher run-off (DS-CR)					
Set 1	6.8	4.2	4.23	1.3%	21.4
Set 2	6.9	4.4	4.17	2.0%	21.8
Set 3	10.4	8.0	4.36	1.6%	23.2
Set 4	7.6	4.5	4.44	0.8%	21.9
Set 5	7.5	4.4	4.31	1.0%	21.5
Set 6	8.1	5.0	4.53	3.8%	21.4
Average	7.9	5.1	4.34	1.8%	21.9
	(17%)	(28%)	(3%)	(62%)	(3%)
nclined drainer (IN-DR)					
Set 1	9.0	7.0	4.06	2.2%	21.4
Set 2	7.5	5.5	3.62	3.8%	21.8
Set 3	11.2	9.0	3.84	1.4%	22.4
Set 4	9.3	7.3	3.60	2.0%	21.5
Set 5	9.2	7.4	3.45	3.3%	21.4
Set 6	8.4	5.7	3.56	3.3%	21.4
Average	9.1	7.0	3.69	2.7%	21.7
-	(13%)	(18%)	(6%)	(35%)	(2%)
Screw press (P <sub>a</sub> )					
Set 1	11.7	9.9	4.80	1.9%	22.2
Set 2	10.4	8.4	4.85	2.0%	21.3
Set 3	12.8	9.7	4.74	2.4%	23.3
Set 4	n.d. <sup>b</sup>	n.d.	4.53	n.d.	n.d.
Set 5	11.4	8.4	5.02	2.6%	21.4
Set 6	n.d.	n.d.	n.d.	n.d.	n.d.
Average	11.6	9.1	4.79	2.2%	22.05
	(9%)	(9%)	(4%)	(15%)	(4%)
Screw press (P <sub>b</sub> )					
Set 1	n.d.	n.d.	4.80	1.8%	21.5
Set 2	12.8	11.6	4.96	10.0%	21.2
Set 3	13.7	11.3	4.71	4.6%	23.5
Set 4	12.3	9.7	4.63	2.7%	21.7
Set 5	13.4	11.2	5.07	6.0%	21.3
Set 6	13.2	10.4	4.78	2.6%	21.5
Average	13.1	10.8	4.83	4.6%	21.8
	(4%)	(7%)	(3%)	(66%)	(4%)
	( )	( /	()	(	· · /

Table B.1: Individual results for Winery A

31.2 30.4	26.0 26.3	6.40	2.3%	21.1 23.3
21.2	28.8	8.08	0.6%	04.4
n.d.	n.d.	7.56	0.9%	21.2
(39%)	(31%)	(14%)	(47%)	(4%)
23.4	19.9	6.34	2.8%	22.2
				21.7
23.3	21.4	7.26	2.6%	21.9
16.8	14.8	5.61	4.2%	21.7
39.5	27.8	6.06	1.3%	24.1
		7.61		21.4
17 2	16 1	5.96	4 2%	22.3
(20%)	(27%)	(14%)	(39%)	(5%)
15.9	14.0	5.52	2.7%	22.1
				21.2
				21.5
				21.7
				24.2
				22.6 21.3
	(20%) 17.2 28.0 39.5 16.8 23.3 15.6 23.4 (39%) n.d.	18.7       18.0         18.7       16.6         12.6       10.1         19.2       17.9         13.5       10.6         15.9       14.0         (20%)       (27%)         17.2       16.1         28.0       26.1         39.5       27.8         16.8       14.8         23.3       21.4         15.6       13.0         23.4       19.9         (39%)       (31%)	18.7 $18.0$ $6.11$ $18.7$ $16.6$ $5.31$ $12.6$ $10.1$ $4.90$ $19.2$ $17.9$ $6.80$ $13.5$ $10.6$ $4.91$ $15.9$ $14.0$ $5.52$ $(20%)$ $(27%)$ $(14%)$ $17.2$ $16.1$ $5.96$ $28.0$ $26.1$ $7.61$ $39.5$ $27.8$ $6.06$ $16.8$ $14.8$ $5.61$ $23.3$ $21.4$ $7.26$ $15.6$ $13.0$ $5.54$ $23.4$ $19.9$ $6.34$ $(39%)$ $(31%)$ $(14%)$	18.7 $18.0$ $6.11$ $4.8%$ $18.7$ $16.6$ $5.31$ $2.8%$ $12.6$ $10.1$ $4.90$ $2.0%$ $19.2$ $17.9$ $6.80$ $2.3%$ $13.5$ $10.6$ $4.91$ $2.1%$ $15.9$ $14.0$ $5.52$ $2.7%$ $(20%)$ $(27%)$ $(14%)$ $(39%)$ $17.2$ $16.1$ $5.96$ $4.2%$ $28.0$ $26.1$ $7.61$ $1.3%$ $39.5$ $27.8$ $6.06$ $1.3%$ $16.8$ $14.8$ $5.61$ $4.2%$ $23.3$ $21.4$ $7.26$ $2.6%$ $15.6$ $13.0$ $5.54$ $3.3%$ $23.4$ $19.9$ $6.34$ $2.8%$ $(39%)$ $(31%)$ $(14%)$ $(47%)$

<sup>a</sup>Coefficient of variation reported in parentheses. <sup>b</sup>n.d.: not determined.

Sample	A <sub>280 nm</sub> (au)	A <sub>320</sub> (au)	Conductivity (mS/cm)	Solids (v/v)	RI (°Brix)
Drainer (ST-DR)					
Set 1	8.1	6.6	3.14	4.5%	22.8
Set 2	8.2	7.5	3.57	2.5%	22.6
Set 3	10.4	10.7	3.14	1.3%	25.1
Set 4	9.2	7.5	3.97	4.9%	22.8
Set 5	9.4	8.1	3.73	2.5%	24.4
Set 6	8.6	7.3	3.57	3.7%	23.0
Set 7	10.7	8.2	4.23	4.8%	22.2
Set 8	8.8	7.8	3.47	3.4%	22.4
Set 9	9.6	7.8	3.76	3.0%	23.7
Set 10	8.2	8.2	3.39	2.1%	21.1
Average	9.1 (10%)	8.0 (13%)	3.60 (10%)	3.3% (37%)	23.0 (5%)
Screw Press (P₁)					
	0.7		0.50	4.00/	00.0
Set 1	9.7	8.0	3.56	4.8%	22.6
Set 2	10.8	10.2	3.59	4.2%	24.1
Set 3	11.8	11.3	3.51	3.7%	24.7
Set 4	11.7	10.4	4.09	4.1%	24.5
Set 5	10.1	9.5	4.14	4.6%	22.9
Set 6	10.5	9.1	3.89	5.4%	23.4
Set 7	11.7	10.2	3.86	5.3%	23.9
Set 8	9.5	9.6	3.49	5.0%	21.1
Set 9 Set 10	10.0 10.6	9.0 8.8	3.76 3.67	5.9% 5.7%	23.4 22.7
Average	10.6	9.6	3.76	4.9%	23.3
Average	(8%)	(10%)	(6%)	(15%)	(5%)
Screw Press (P <sub>2</sub> )					
Set 1	19.6	18.4	5.65	4.0%	22.6
Set 2	19.2	20.0	4.54	7.8%	24.4
Set 3	17.6	17.6	4.03	5.5%	24.9
Set 4	16.4	16.0	4.54	3.3%	25.3
Set 5	17.7	18.5	5.12	6.9%	22.5
Set 6	16.7	15.6	4.80	4.9%	24.0
Set 7	18.4	18.0	4.73	5.0%	23.9
Set 8	14.0	15.6	3.81	6.1%	21.5
Set 9	13.5	12.8	3.98	5.9%	23.8
Set 10	16.2	15.2	4.47	5.3%	23.1
Average	16.9	16.8	4.57	5.4%	23.6
	(12%)	(13%)	(12%)	(24%)	(5%)

<sup>a</sup>Coefficient of variation reported in parentheses.

	100 g/L PVPP treatment				
Sample	-				
	A <sub>280</sub> (au)	A <sub>320</sub> (au)			
Winery A					
DS-CR, Set 5	5.3	1.7			
IN-DR, Set 5	4.6	0.8			
Pa, 4:30, Set 5	7.8	3.9			
Pb, 4:30, Set 5	9.3	5.3			
Pc, 4:30, Set 5	12.9	9.0			
Pd, 4:30, Set 5	13.6	10.1			
Pe, 4:30, Set 5	15.1	11.8			
Winery B – Process 1					
ST-DR, Set 2	4.0	1.5			
P1, 1:30, Set 2	5.5	3.1			
P2, 1:30, Set 2	8.8	6.4			
ST-DR, Set 4	5.0	1.9			
P1, Set 4	6.3	3.6			
P2, Set 4	8.3	5.6			
ST-DR, Set 10	3.3	1.2			
P1, Set 10	6.2	3.4			
P2, Set 10	9.1	5.8			

# Table B.3: Spectral results after 100 g/L PVPP treatment for selected samples (Results show<br/>higher residual absorbance after PVPP treatment for samples that exhibited higher<br/>absorbance without PVPP treatment)

#### **APPENDIX C: ADDITIONAL DATA FOR CHAPTER 9**

Table C.1: Spectral results with and wihtout 100 g/L PVPP treatment from juice samples collected during a preliminary laboratory pressing trial with 2009 vintage Barossa Valley Chardonnay grapes (Results show higher residual absorbance after PVPP treatment for samples that exhibited higher absorbance without PVPP treatment.)

Somolo	No PVPP	treatment	100 g/L PVP	P treatment
Sample	A <sub>280</sub> (au)	A <sub>320</sub> (au)	A <sub>280</sub> (au)	A <sub>320</sub> (au)
1	5.48	2.86	4.64	1.68
2	6.32	3.30	5.33	1.98
3	7.24	4.02	5.72	2.19
4	8.09	4.58	6.26	2.45
5	8.49	4.78	6.45	2.48
6	8.60	4.70	6.55	2.50
7	8.56	4.48	6.56	2.44
8	8.61	4.35	6.69	2.42
9	8.33	3.64	6.75	2.22

Table C.2: Analytical results for samples collected during pressing of 1 kg of 100% precrushed 2009 vintage Barossa Valley Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Drain	434	434	5.86	3.61	3.50	3.88	10.2%	n.d.
Cycle 1	54	487	7.19	4.15	4.14	4.00	1.7%	22.3
Cycle 2	77	565	8.23	4.47	4.31	4.00	4.4%	22.4
Cycle 3	52	617	9.51	5.31	4.76	4.13	3.2%	22.4
Cycle 4	37	653	10.10	5.58	5.11	4.18	1.6%	22.3
Cycle 5	23	676	10.04	5.36	5.28	4.22	1.3%	22.2
Cycle 6	17	693	10.41	5.34	5.49	4.19	0.7%	22.1
Cycle 7	13	706	10.20	4.95	5.70	4.24	n.d.	n.d.
Cycle 8	10	717	10.11	4.74	5.61	4.27	n.d.	n.d.

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	210	210	4.73	2.95	3.31	3.74	0.8%	22.4
Cycle 2	128	339	5.67	3.33	3.60	3.82	1.6%	22.5
Cycle 3	83	422	6.42	3.70	3.80	3.93	1.4%	22.4
Cycle 4	60	482	7.22	4.17	3.97	3.97	1.6%	22.4
Cycle 5	43	525	7.77	4.55	4.29	3.91	1.4%	22.3
Cycle 6	36	561	8.15	4.71	4.38	4.05	1.3%	22.2
Cycle 7	27	588	8.15	4.61	4.54	4.05	1.5%	22.2
Cycle 8	23	611	8.23	4.53	4.64	4.08	1.3%	22.1
Cycle 9	19	630	8.34	4.36	4.76	4.12	n.d.	n.d.
Cycle 10	16	646	8.44	4.32	4.94	4.07	n.d.	n.d.
Cycle 11	14	660	8.16	4.02	4.97	4.15	n.d.	n.d.

 Table C.3: Analytical results for samples collected during pressing of 1 kg of whole 2009

 vintage Barossa Valley Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Table C.4: Analytical results for samples collected during pressing of 750 g of whole 2009 vintage Barossa Valley Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	203	203	5.28	3.34	3.38	3.75	0.8%	23.3
Cycle 2	124	327	6.31	3.73	3.66	3.87	1.8%	23.3
Cycle 3	83	410	7.35	4.45	3.92	3.87	2.3%	23.3
Cycle 4	57	467	8.17	5.05	4.25	4.00	1.8%	23.2
Cycle 5	42	509	8.50	5.19	4.40	4.03	1.7%	23.2
Cycle 6	33	542	8.72	5.17	4.56	4.06	1.5%	23.1
Cycle 7	27	569	8.98	5.15	4.77	4.04	1.4%	23.1
Cycle 8	23	592	9.06	4.99	4.86	4.13	1.3%	23.2
Cycle 9	19	610	9.06	4.74	4.97	4.15	n.d.	n.d.
Cycle 10	15	625	8.99	4.55	5.10	4.16	n.d.	n.d.

n.d.: not determined.

 Table C.5: Analytical results for samples collected during pressing of 250 g of whole 2009

 vintage Barossa Valley Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
• • •								
Cycle 1	221	221	5.16	3.33	3.32	3.70	2.5%	23.2
Cycle 2	125	346	6.25	3.78	3.67	3.85	3.5%	23.2
Cycle 3	92	438	7.20	4.38	3.80	3.94	4.2%	23.4
Cycle 4	56	494	n.d.	n.d.	4.17	3.93	3.7%	23.4
Cycle 5	48	542	8.86	5.17	4.26	4.03	n.d.	n.d.
Cycle 6	36	579	9.04	5.04	n.d.	n.d.	n.d.	n.d.

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	223	223	5.01	2.98	3.50	3.69	1.4%	22.0
Cycle 2	120	343	6.01	3.37	3.77	3.95	2.9%	22.1
Cycle 3	82	425	6.91	3.88	4.00	3.89	3.1%	22.2
Cycle 4	64	489	7.79	4.46	4.36	3.88	2.8%	22.1
Cycle 5	44	533	8.30	4.61	4.53	3.99	2.4%	22.1
Cycle 6	32	566	8.58	4.53	4.68	4.02	2.1%	22.1
Cycle 7	28	594	8.68	4.48	4.82	4.03	n.d.	n.d.
Cycle 8	21	615	8.55	4.20	n.d.	4.07	n.d.	n.d.

 Table C.6: Analytical results for samples collected during pressing of 500 g of whole 2009

 vintage Barossa Valley Chardonnay grapes pressed at 10 mm/min, standard sieve plate

 Table C.7: Analytical results for samples collected during pressing of 125 g of whole 2009

 vintage Barossa Valley Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	261	261	n.d.	n.d.	3.51	3.61	3.2%	22.4
Cycle 2	169	430	n.d.	n.d.	3.94	3.83	4.0%	22.6

n.d.: not determined.

Table C.8: Analytical results for samples collected during pressing of 1 kg of whole 2009vintage Barossa Valley Chardonnay grapes pressed at 10 mm/min, without crumbling,<br/>standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	205	205	5.24	3.25	3.46	3.80	0.9%	22.5
Cycle 2	25	231	5.62	3.49	3.69	3.87	1.8%	22.2
Cycle 3	17	248	6.02	3.70	3.76	3.93	0.7%	22.3
Cycle 4	12	260	n.d.	n.d.	3.88	3.96	0.6%	22.4

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Drain	410	410	5.80	3.74	3.47	3.82	8.6%	21.9
Cycle 1	83	492	7.14	4.40	4.09	3.98	1.0%	22.0
Cycle 2	24	516	7.66	4.50	4.25	3.93	0.4%	21.9
Cycle 3	19	535	7.94	4.59	4.32	4.04	0.4%	22.0
Cycle 4	18	552	8.21	4.73	4.29	4.01	0.5%	22.1
Cycle 5	14	566	8.28	4.69	4.30	4.08	n.d.	n.d.

Table C.9: Analytical results for samples collected during pressing of 1 kg of 100% precrushed 2009 vintage Barossa Valley Chardonnay grapes pressed at 10 mm/min, without crumbling, standard sieve plate

## Table C.10: Analytical results for samples collected during pressing of 250 g of whole 2009 vintage Langhorne Creek Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Overla 1	200	200	4.00	0.54	0.05	0.00	4 00/	04.4
Cycle 1	322	322	4.28	3.51	2.85	3.39	1.8%	21.4
Cycle 2	151	473	5.54	4.17	2.90	3.55	2.3%	21.6
Cycle 3	72	545	6.58	4.87	3.17	3.62	1.8%	21.5
Cycle 4	52	597	n.d.	n.d.	3.37	3.72	1.6%	21.7
Cycle 5	36	634	7.72	5.32	n.d.	3.84	n.d.	n.d.

n.d.: not determined.

 Table C.11: Analytical results for samples collected during pressing of 250 g of whole 2009

 vintage Langhorne Creek Chardonnay grapes pressed at 100 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	277	277	4.08	3.79	2.73	3.34	6.0%	20.8
Cycle 2	128	405	5.12	3.80	2.99	3.52	6.6%	20.9
Cycle 3	108	514	6.16	4.51	3.15	3.61	8.0%	21.2
Cycle 4	68	582	6.78	4.63	3.34	3.62	6.6%	21.3
Cycle 5	48	630	n.d.	n.d.	3.58	3.77	9.0%	21.3
Cycle 6	28	658	7.99	5.51	n.d.	n.d.	n.d.	n.d.

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	229	229	3.99	3.83	2.56	3.30	4.3%	21.4
Cycle 2	136	365	4.88	3.88	2.67	3.47	4.9%	21.4
Cycle 3	88	453	5.74	4.60	2.87	3.51	3.9%	21.5
Cycle 4	68	521	6.43	5.27	2.93	3.62	4.9%	21.6
Cycle 5	44	566	n.d.	n.d.	3.09	3.69	2.9%	21.9
Cycle 6	40	606	7.43	5.81	n.d.	n.d.	n.d.	n.d.

 Table C.12: Analytical results for samples collected during pressing of 250 g of whole 2009

 vintage Langhorne Creek Chardonnay grapes pressed at 50 mm/min, standard sieve plate

 Table C.13: Analytical results for samples collected during pressing of 250 g of whole 2009

 vintage Langhorne Creek Chardonnay grapes pressed at 20 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	249	249	4.11	3.64	2.66	3.39	2.9%	20.9
Cycle 2	141	390	5.10	3.95	2.84	3.49	3.9%	21.0
Cycle 3	94	484	6.17	4.97	3.02	3.62	2.2%	21.0
Cycle 4	68	553	7.03	5.57	3.25	3.64	2.1%	21.2
Cycle 5	46	599	n.d.	n.d.	3.42	3.73	1.9%	21.2
Cycle 6	38	637	7.87	5.89	n.d.	n.d.	n.d.	n.d.

n.d.: not determined.

 Table C.14: Analytical results for samples collected during pressing of 125 g of whole 2009

 vintage Langhorne Creek Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield (L/tonne)	Cumulative yield (L/tonne)	A <sub>280</sub> (au)	A <sub>320</sub> (au)	Cond. (mS/cm)	рН	Solids (v/v)	RI (°Brix)
Cycle 1	394	394	4.48	4.08	2.71	3.43	2.3%	21.2
Cycle 2	157	551	6.66	5.25	3.17	3.64	2.4%	21.4
Cycle 3	56	607	8.04	6.22	n.d.	n.d.	n.d.	n.d.

n.d.: not determined.

 Table C.15: Analytical results for samples collected during pressing of 125 g of whole 2009

 vintage Langhorne Creek Chardonnay grapes pressed at 50 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	403	403	4.38	3.96	2.76	3.39	5.8%	20.7
Cycle 2	129	532	5.99	4.27	3.17	3.65	4.9%	20.7
Cycle 3	64	596	7.59	5.51	n.d.	n.d.	n.d.	n.d.

Fraction yield (L/tonne)	Cumulative yield (L/tonne)	A <sub>280</sub> (au)	A <sub>320</sub> (au)	Cond. (mS/cm)	рН	Solids (v/v)	RI (°Brix)
314	314	4.25	4.28	2.55	3.37	3.9%	21.1
177	490 582	5.63 7.16	4.48 5.72	2.93	3.61	6.0%	21.2 n.d.
	yield (L/tonne) 314	yield         yield           (L/tonne)         (L/tonne)           314         314           177         490	yield         yield         A280           (L/tonne)         (L/tonne)         (au)           314         314         4.25           177         490         5.63	yieldyieldA280A320(L/tonne)(L/tonne)(au)(au)3143144.254.281774905.634.48	yield         yield         A280         A320         Cond.           (L/tonne)         (L/tonne)         (au)         (au)         (mS/cm)           314         314         4.25         4.28         2.55           177         490         5.63         4.48         2.93	yield         yield         A280         A320         Cond.         pH           (L/tonne)         (L/tonne)         (au)         (au)         (mS/cm)           314         314         4.25         4.28         2.55         3.37           177         490         5.63         4.48         2.93         3.61	yield         yield         A <sub>280</sub> A <sub>320</sub> Cond.         pH         Solids           (L/tonne)         (L/tonne)         (au)         (au)         (mS/cm)         (v/v)           314         314         4.25         4.28         2.55         3.37         3.9%           177         490         5.63         4.48         2.93         3.61         6.0%

 Table C.16: Analytical results for samples collected during pressing of 125 g of whole 2009

 vintage Langhorne Creek Chardonnay grapes pressed at 100 mm/min, standard sieve plate

n.d.: not determined.

 Table C.17: Analytical results for samples collected during pressing of 125 g of whole 2009

 vintage Langhorne Creek Chardonnay grapes pressed at 20 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	357	357	4.14	4.20	2.60	3.34	2.0%	21.0
Cycle 2	164	521	5.72	4.71	2.91	3.59	2.7%	21.4
Cycle 3	64	585	7.31	6.07	n.d.	n.d.	n.d.	n.d.

n.d.: not determined.

 Table C.18: Analytical results for samples collected during pressing of 750 g of whole 2009

 vintage Langhorne Creek Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	243	243	4.38	3.69	2.95	3.41	0.9%	20.8
Cycle 2	128	371	5.11	3.70	3.03	3.59	1.2%	20.7
Cycle 3	75	446	5.81	4.25	3.19	3.63	2.5%	20.7
Cycle 4	65	512	6.27	4.59	3.37	3.63	1.4%	20.7
Cycle 5	48	560	6.70	4.76	3.51	3.72	0.9%	20.7
Cycle 6	33	593	6.96	4.88	3.68	3.74	0.9%	20.7
Cycle 7	27	620	7.24	5.31	3.80	3.81	1.1%	20.8
Cycle 8	23	643	7.31	5.21	3.96	3.85	1.0%	20.7
Cycle 9	19	663	7.39	5.13	4.21	3.83	n.d.	n.d.

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	163	163	4.12	3.83	2.78	3.40	1.9%	20.9
Cycle 2	111	274	4.57	3.44	2.91	3.47	4.3%	21.0
Cycle 3	79	353	5.11	3.65	3.04	3.59	6.5%	21.1
Cycle 4	63	415	5.84	4.37	3.15	3.62	5.3%	21.2
Cycle 5	49	465	6.24	4.43	3.38	3.69	3.2%	21.1
Cycle 6	40	505	6.60	4.74	3.48	3.68	4.6%	21.1
Cycle 7	33	538	6.91	4.93	3.58	3.78	4.0%	21.1
Cycle 8	28	566	6.97	5.05	3.68	3.78	n.d.	n.d.
Cycle 9	25	592	7.13	5.08	3.84	3.82	4.2%	21.2

 Table C.19: Analytical results for samples collected during pressing of 750 g of whole 2009

 vintage Langhorne Creek Chardonnay grapes pressed at 100 mm/min, standard sieve plate

## Table C.20: Analytical results for samples collected during pressing of 750 g of whole 2009 vintage Langhorne Creek Chardonnay grapes pressed at 50 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	182	182	4.10	3.79	2.72	3.36	1.6%	20.7
Cycle 2	106	287	4.68	3.43	2.84	3.52	2.4%	20.8
Cycle 3	73	361	5.23	3.85	2.97	3.68	3.1%	20.9
Cycle 4	63	423	5.83	4.47	3.07	3.64	3.2%	20.9
Cycle 5	50	473	6.36	4.69	3.23	3.65	2.2%	21.0
Cycle 6	39	512	6.77	5.21	3.37	3.74	2.2%	20.9
Cycle 7	32	544	6.89	5.25	3.42	3.76	2.4%	21.0
Cycle 8	25	569	7.08	5.36	3.52	3.81	n.d.	n.d.
Cycle 9	22	591	7.08	5.20	3.63	3.82	1.9%	20.9

n.d.: not determined.

 Table C.21: Analytical results for samples collected during pressing of 500 g of whole 2009

 vintage Langhorne Creek Chardonnay grapes pressed at 50 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	196	196	4.29	4.40	2.68	3.42	2.1%	21.1
Cycle 2	114	311	5.10	4.17	2.90	3.55	3.6%	21.2
Cycle 3	90	401	6.07	5.09	3.12	3.67	4.4%	21.4
Cycle 4	69	470	6.80	5.58	3.31	3.71	3.4%	21.3
Cycle 5	49	519	7.46	5.79	3.52	3.74	3.0%	21.4
Cycle 6	38	557	8.05	6.20	3.61	3.85	4.5%	21.5
Cycle 7	29	586	n.d.	n.d.	3.79	3.84	3.5%	21.5
Cycle 8	22	608	8.19	6.13	3.95	3.88	n.d.	n.d.

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	186	186	4.22	4.12	2.66	3.37	3.8%	20.8
Cycle 2	108	294	5.00	3.83	2.89	3.49	3.4%	21.0
Cycle 3	95	389	5.67	4.36	3.04	3.67	7.5%	21.1
Cycle 4	64	454	6.39	4.91	3.22	3.62	5.1%	21.1
Cycle 5	50	504	6.92	4.96	3.36	3.68	6.0%	21.1
Cycle 6	39	543	7.44	5.42	3.59	3.71	3.9%	21.1
Cycle 7	34	577	7.36	5.54	3.63	3.73	5.4%	21.1
Cycle 8	23	600	7.89	5.84	3.78	3.79	n.d.	n.d.

 Table C.22: Analytical results for samples collected during pressing of 500 g of whole 2009

 vintage Langhorne Creek Chardonnay grapes pressed at 100 mm/min, standard sieve plate

 Table C.23: Analytical results for samples collected during pressing of 500 g of whole 2009
 vintage Langhorne Creek Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	230	230	4.28	3.80	2.78	3.39	1.2%	20.5
Cycle 2	122	353	5.16	3.91	3.10	3.55	1.2%	20.6
Cycle 3	90	443	5.76	4.52	3.14	3.55	1.4%	20.6
Cycle 4	61	504	6.38	5.19	3.30	3.66	1.2%	20.6
Cycle 5	46	550	6.70	5.39	3.44	3.71	0.8%	20.7
Cycle 6	34	584	6.85	5.42	3.53	3.73	0.8%	20.7
Cycle 7	28	612	n.d.	n.d.	3.76	3.76	0.9%	20.7
Cycle 8	24	636	7.30	5.50	3.89	3.82	n.d.	n.d.

n.d.: not determined.

Table C.24: Analytical results for samples collected during pressing of 500 g of 100% precrushed 2009 vintage Langhorne Creek Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Drain	330	330	4.38	4.01	2.76	3.46	5.8%	21.3
Cycle 1	205	535	5.73	5.24	3.12	3.56	0.7%	21.4
Cycle 2	76	611	7.36	6.23	3.54	3.70	0.7%	21.4
Cycle 3	40	651	8.37	7.12	3.84	3.80	0.5%	21.5
Cycle 4	25	676	n.d.	n.d.	4.21	3.83	0.5%	21.5
Cycle 5	18	694	8.90	7.13	n.d.	n.d.	n.d.	n.d.

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Drain	(333)	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Cycle 1	192	192	5.91	4.84	3.35	3.60	0.9%	20.9
Back-								
addition	306	499	4.34	3.14	2.97	3.49	5.7%	20.7
of Drain								
Cycle 2	102	601	6.64	5.24	3.57	3.66	1.2%	20.9
Cycle 3	44	645	8.33	6.01	4.02	3.76	0.7%	21.0
Cycle 4	27	671	n.d.	n.d.	4.18	3.88	0.7%	21.1
Cycle 5	18	690	8.99	6.61	n.d.	n.d.	n.d.	n.d.

Table C.25: Analytical results for samples collected during pressing of 500 g of 100% pre-crushed 2009 vintage Langhorne Creek Chardonnay grapes pressed at 10 mm/min, standardsieve plate, Drain fraction back-added to crumbled cake

n.d.: not determined.

Table C.26: Analytical results for samples collected during pressing of 500 g of 100% precrushed 2009 vintage Langhorne Creek Chardonnay grapes pressed at 10 mm/min, standard sieve plate, Drain fraction back-added to intact cake

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Drain	(346)	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Cycle 1	188	188	5.42	4.64	3.16	3.73	0.6%	20.8
Back- addition of Drain	307	495	4.07	3.21	2.90	3.50	4.1%	20.8
Cycle 2	63	558	5.28	4.28	3.22	3.59	0.8%	20.9
Cycle 3	57	616	7.07	5.22	3.70	3.70	0.7%	21.1
Cycle 4	35	650	7.98	6.29	3.90	3.82	0.8%	21.0
Cycle 5	24	674	8.32	6.51	4.12	3.86	n.d.	n.d.

n.d.: not determined.

 Table C.27: Analytical results for samples collected during pressing of 250 g of whole 2009

 vintage Adelaide Hills Chardonnay grapes pressed at 100 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	180	180	4.50	5.44	2.32	3.36	4.0%	22.4
Cycle 2	156	336	5.03	4.54	2.56	3.50	5.5%	22.6
Cycle 3	104	441	5.70	4.79	2.70	3.59	6.3%	22.7
Cycle 4	72	513	6.42	5.01	2.85	3.63	4.6%	22.8
Cycle 5	54	567	n.d.	n.d.	2.90	3.75	3.6%	22.9
Cycle 6	40	607	7.24	4.92	n.d.	n.d.	n.d.	n.d.

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	114	114	4.39	5.01	2.35	3.38	1.9%	22.7
Cycle 2	138	252	4.93	4.39	2.61	3.54	5.4%	22.9
Cycle 3	106	358	5.53	4.45	2.83	3.55	4.5%	22.8
Cycle 4	76	435	6.12	4.78	2.87	3.65	4.0%	23.0
Cycle 5	68	503	6.65	4.96	2.99	3.71	6.0%	23.1
Cycle 6	46	549	6.92	4.73	n.d.	n.d.	n.d.	n.d.

 Table C.28: Analytical results for samples collected during pressing of 250 g of whole 2009

 vintage Adelaide Hills Chardonnay grapes pressed at 100 mm/min, alternate sieve plate

n.d.: not determined.

 Table C.29: Analytical results for samples collected during pressing of 250 g of whole 2009

 vintage Adelaide Hills Chardonnay grapes pressed at 10 mm/min, alternate sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	213	213	4.23	4.40	2.43	3.39	1.5%	22.5
Cycle 2	128	341	5.15	4.27	2.77	3.60	1.7%	n.d.
Cycle 3	88	429	5.55	4.40	2.91	3.58	1.1%	22.9
Cycle 4	70	499	5.89	4.54	2.98	3.66	1.2%	22.9
Cycle 5	48	547	n.d.	n.d.	3.12	3.76	1.2%	23.1
Cycle 6	40	587	6.73	4.72	n.d.	n.d.	n.d.	n.d.

n.d.: not determined.

 Table C.30: Analytical results for samples collected during pressing of 250 g of whole 2009

 vintage Adelaide Hills Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	224	224	4.21	4.76	2.39	3.32	1.4%	21.9
Cycle 2	142	367	5.01	4.61	2.64	3.48	1.3%	22.0
Cycle 3	104	471	5.52	4.83	2.76	3.54	1.2%	22.0
Cycle 4	68	539	6.11	5.12	2.92	3.58	1.0%	22.1
Cycle 5	42	581	6.34	4.72	n.d.	n.d.	n.d.	n.d.
Cycle 6	34	615	6.61	4.69	n.d.	n.d.	n.d.	n.d.

Fraction	Fraction yield (L/tonne)	Cumulative yield (L/tonne)	A <sub>280</sub> (au)	A <sub>320</sub> (au)	Cond. (mS/cm)	рН	Solids (v/v)	RI (°Brix)
		(Litonne)	լսսյ	(au)			(•/•)	
Cycle 1	229	229	4.60	5.46	2.34	3.34	4.0%	22.7
Cycle 2	177	405	5.21	4.32	2.64	3.56	8.0%	23.0
Cycle 3	132	538	6.46	5.22	2.69	3.65	8.0%	23.0

 Table C.31: Analytical results for samples collected during pressing of 125 g of whole 2009

 vintage Adelaide Hills Chardonnay grapes pressed at 100 mm/min, alternate sieve plate

n.d.: not determined.

 Table C.32: Analytical results for samples collected during pressing of 125 g of whole 2009

 vintage Adelaide Hills Chardonnay grapes pressed at 100 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	325	325	4.62	5.55	2.41	3.36	4.4%	22.5
Cycle 2	148	473	5.29	4.50	2.79	3.60	9.0%	22.5
Cycle 3	96	569	n.d.	n.d.	2.86	3.72	7.5%	22.9

n.d.: not determined.

 Table C.33: Analytical results for samples collected during pressing of 125 g of whole 2009

 vintage Adelaide Hills Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	313	313	4.75	5.63	2.55	3.42	1.4%	22.7
Cycle 2	160	473	5.70	4.80	2.88	3.64	1.7%	22.9
Cycle 3	84	557	n.d.	n.d.	3.03	3.71	1.4%	22.7

n.d.: not determined.

 Table C.34: Analytical results for samples collected during pressing of 125 g of whole 2009

 vintage Adelaide Hills Chardonnay grapes pressed at 10 mm/min, alternate sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	281	281	4.46	5.34	2.41	3.37	1.2%	22.6
Cycle 2	140	421	5.20	4.46	2.67	3.56	1.4%	22.8
Cycle 3	96	518	n.d.	n.d.	2.76	3.67	1.7%	22.9

			•					
Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	124	124	4.58	5.26	2.35	3.40	3.0%	22.4
Cycle 2	102	226	4.78	4.14	2.58	3.51	5.5%	22.5
Cycle 3	82	308	5.25	4.32	2.66	3.56	5.3%	22.5
Cycle 4	72	380	5.64	4.58	2.77	3.59	8.0%	22.6
Cycle 5	57	438	6.05	4.79	2.80	3.63	8.0%	22.6
Cycle 6	50	488	6.41	5.08	2.82	3.67	6.0%	22.6
Cycle 7	40	528	6.77	5.14	2.89	3.72	5.8%	22.7
Cycle 8	31	559	7.10	5.17	2.94	3.76	6.7%	22.8

Table C.35: Analytical results for samples collected during pressing of 500 g of whole 2009 vintage Adelaide Hills Chardonnay grapes pressed at 100 mm/min, alternate sieve plate, replicate 1

## Table C.36: Analytical results for samples collected during pressing of 500 g of whole 2009 vintage Adelaide Hills Chardonnay grapes pressed at 100 mm/min, standard sieve plate, replicate 1

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	124	124	4.77	5.62	2.45	3.47	2.1%	22.4
Cycle 2	104	228	5.01	4.60	2.73	3.56	3.0%	22.5
Cycle 3	87	315	5.49	4.62	2.84	3.65	5.3%	22.7
Cycle 4	72	387	5.87	4.88	2.86	3.66	3.6%	22.7
Cycle 5	60	447	6.26	5.14	2.96	3.72	3.9%	22.7
Cycle 6	50	497	6.50	5.21	2.96	3.74	7.2%	22.7
Cycle 7	41	539	6.99	5.32	3.13	3.83	6.5%	22.7
Cycle 8	34	573	7.06	5.17	3.13	3.85	2.7%	22.7

n.d.: not determined.

## Table C.37: Analytical results for samples collected during pressing of 500 g of whole 2009 vintage Adelaide Hills Chardonnay grapes pressed at 100 mm/min, standard sieve plate, replicate 2

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	144	144	4.62	5.41	2.35	3.46	2.2%	22.4
Cycle 2	112	256	5.05	4.54	2.55	3.60	5.5%	22.6
Cycle 3	89	346	5.62	4.69	2.81	3.62	7.0%	22.5
Cycle 4	78	424	5.99	5.01	2.75	3.68	7.0%	22.6
Cycle 5	58	482	6.51	5.36	2.85	3.73	8.0%	22.5
Cycle 6	49	531	6.91	5.47	2.90	3.79	3.7%	22.5
Cycle 7	40	571	7.29	5.62	3.01	3.82	4.6%	22.7
Cycle 8	34	605	7.85	5.82	3.15	3.89	3.5%	22.8

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Cycle 1	132	132	4.39	5.12	2.36	3.42	2.6%	22.2
Cycle 2	100	232	4.84	4.15	2.77	3.59	3.2%	22.3
Cycle 3	85	318	5.28	4.38	2.80	3.61	6.5%	22.5
Cycle 4	64	382	5.66	4.57	2.91	3.64	3.7%	22.5
Cycle 5	52	434	6.01	4.91	2.88	3.70	4.9%	22.4
Cycle 6	52	486	6.13	4.90	2.91	3.72	10.0%	22.4
Cycle 7	38	524	6.66	5.21	2.93	3.78	3.9%	22.4
Cycle 8	34	558	6.82	5.06	3.08	3.79	2.6%	22.5

Table C.38: Analytical results for samples collected during pressing of 500 g of whole 2009 vintage Adelaide Hills Chardonnay grapes pressed at 100 mm/min, alternate sieve plate, replicate 2

Table C.39: Analytical results for samples collected during pressing of 500 g of 100% precrushed 2009 vintage Adelaide Hills Chardonnay grapes pressed at 10 mm/min, standard sieve plate

Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Drain	287	287	4.84	5.29	2.57	3.52	5.0%	22.5
Cycle 1	187	474	5.90	5.90	2.88	3.67	1.1%	22.6
Cycle 2	87	561	7.51	6.79	3.16	3.75	1.2%	22.7
Cycle 3	49	610	8.86	7.71	3.43	3.79	0.6%	22.6
Cycle 4	33	643	n.d.	n.d.	3.53	3.89	0.6%	22.6
Cycle 5	23	666	10.14	8.30	3.70	3.94	n.d.	n.d.

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Fraction	Fraction yield	Cumulative yield	A <sub>280</sub>	A <sub>320</sub>	Cond.	рН	Solids	RI
	(L/tonne)	(L/tonne)	(au)	(au)	(mS/cm)		(v/v)	(°Brix)
Drain	(287)	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Cycle 1	181	181	5.96	5.82	2.94	3.64	0.4%	22.7
Back-								
addition	293	474	4.91	4.81	2.63	3.53	2.8%	22.6
of Drain								
Cycle 2	39	513	5.96	5.56	2.81	3.64	0.5%	22.7
Cycle 3	70	583	7.74	6.82	3.16	3.75	0.7%	22.9
Cycle 4	43	626	9.00	7.68	3.39	3.82	0.7%	22.9
Cycle 5	30	656	n.d.	n.d.	3.54	3.92	0.6%	23.0

Table C.40: Analytical results for samples collected during pressing of 500 g of whole 2009vintage Adelaide Hills Chardonnay grapes pressed at 10 mm/min, standard sieve plate, Drainfraction back-added to intact cake