#### Smoke taint: the latest research from the University of Adelaide Professor Kerry Wilkinson The University of Adelaide kerry.wilkinson@adelaide.edu.au

## Smoke taint research at the University of Adelaide



Sensors for real-time monitoring of vineyard exposure to smoke

# Smoke monitoring project:

Attentis Technologies develop environmental sensors that monitor air quality as particulate matter (PM) concentrations, accessible online, in real-time

#### www.attentistechnologies.com





# Smoke monitoring project: smoke density field trial









## Smoke monitoring project: smoke density field trial



















	guaiacol	cresols	syringol
Control	1	nd	6
Wheat A	3	3	12
Wheat B (upwind)	1	nd	6
Oats A	4	3	18
Oats B	1	nd	7



# Smoke monitoring project:

Latrobe Valley Information Network

www.lvin.org



#### Evaluation of strategies for mitigation of smoke taint

#### Strategies evaluated for mitigation of smoke taint

In the vineyard...

Defoliation

Washing/misting grapes

Hand-harvesting

Protective sprays (e.g. kaolin)\*

Protective coverings\*

🖄 molecules

MDPI

Review

Techniques for Mitigating the Effects of Smoke Taint While Maintaining Quality in Wine Production: A Review

Ysadora A. Mirabelli-Montan<sup>1</sup>, Matteo Marangon<sup>1,\*</sup>, Antonio Graça<sup>2</sup>, Christine M. Mayr Marangon<sup>1</sup> and Kerry L. Wilkinson<sup>3,4</sup>

In the winery...

Reduced skin contact

Different yeast strains

Oak/tannin addition

Post-harvest ozonation\*

Distillation\*

Adsorbents (e.g. carbon, MIPs) \*

Membrane filtration\*

Activated carbon fabric as a protective covering to prevent smoke contamination of grapes

#### Activated carbon fabric project: preliminary field trial



## Activated carbon fabric project: preliminary field trial

	guaiacol	cresols	syringol
Control	nd	nd	nd
Smoke	21	16	16
Plastic bag	11	5.0	5.3
Paper bag	10	8.5	2.0
AC fabric bag	1.3	nd	nd



## Activated carbon fabric project: 'box' trial (grapes)



# Activated carbon fabric project: 'box' trial (grapes)

	guaiacol	cresols	syringol
Control	nd	nd	nd
Smoke	231	194	80
Paper bag	75	48	1
AC fabric bag	5	5	nd
Kaolin	183	158	58
Anti-transpirant	239	224	88



# Activated carbon fabric project: 'box' trial (wine)

	guaiacol	cresols	syringol
Control	2	1	6
Smoke	16	22	21
AC fabric bag (felt)	3	3	7
AC fabric bag (light)	3	2	7
AC fabric bag (heavy)	3	3	6

## Activated carbon fabric project: 'box' trial (wine)

	guaiacol	cresols	syringol
Control	2	1	6
Smoke	16	22	21
AC fabric bag (felt)	3	3	7
AC fabric bag (light)	3	2	7
AC fabric bag (heavy)	3	3	6



-Control ----Smoke

## Activated carbon fabric project: 'box' trial (wine)





fruit A\*

Peter Michael winery now pursuing commercialisation

#### TASTING







#### Sample 1 Control Wine

Sample 2 Smoke Wine Sample 3 ACF Wine

# TASTING

guaiacol	cresols	syringol	guaiacol	cresols	syringol	guaiacol	cresols	syringol
1	nd	2	100	55	36	3	1	6
Sa	mple '	1	S	ample	2	S	ample	3
Cont	trol Wi	ne	Sm	oke W	line	A	CF Wi	ne





	guaiacol	cresols	syringol
Control	1	nd	3
Smoke	25	14	7
Smoke + Viscose	14	8	3
Smoke + ACF	7	5	4













Post-harvest ozone treatment of grapes to mitigate the intensity of smoke taint in wine

# Ozonation project: Merlot trial (moderate smoke exposure)

	guaiacol	cresols	syringol
Control	1.0	nd	3.0
Smoke	15	7.7	4.7
Smoke + 1 ppm $O_3$	12	5.7	4.0
Smoke + 3 ppm O <sub>3</sub>	14	7.4	4.3

	guaiacol glycosides	cresol glycosides	syringol glycosides
Control	9.4	20	3.4
Smoke	295	280	240
Smoke + 1 ppm O <sub>3</sub>	232	217	213
Smoke + 3 ppm O <sub>3</sub>	329	314	273

# Ozonation project: Merlot trial (moderate smoke exposure)

	guaiacol	cresols	syringol
Control	1.0	nd	3.0
Smoke	15	7.7	4.7
Smoke + 1 ppm $O_3$	12	5.7	4.0
Smoke + 3 ppm $O_3$	14	7.4	4.3

	guaiacol glycosides	cresol glycosides	syringol glycosides
Control	9.4	20	3.4
Smoke	295	280	240
Smoke + 1 ppm O <sub>3</sub>	232	217	213
Smoke + 3 ppm $O_3$	329	314	273
# Ozonation project: Merlot trial (moderate smoke exposure)

	guaiacol	cresols	syringol
Control	1.0	nd	3.0
Smoke	15	7.7	4.7
Smoke + 1 ppm O <sub>3</sub>	12	5.7	4.0
Smoke + 3 ppm O <sub>3</sub>	14	7.4	4.3

	guaiacol glycosides	cresol glycosides	syringol glycosides
Control	9.4	20	3.4
Smoke	295	280	240
Smoke + 1 ppm O <sub>3</sub>	232	217	213
Smoke + 3 ppm $O_3$	329	314	273



### Ozonation project: Cabernet trial (heavy smoke exposure)

	guaiacol	cresols	syringol
Control	~1.0	nd	2.3
Smoke	30	21	6.3
Smoke + 1 ppm $O_3$	23	18	5.3

	guaiacol glycosides	cresol glycosides	syringol glycosides
Control	9.8	4.3	12.2
Smoke	340	117	614
Smoke + 1 ppm $O_3$	258	100	473

# Ozonation project: Cabernet trial (heavy smoke exposure)

	guaiacol	cresols	syringol
Control	~1.0	nd	2.3
Smoke	30	21	6.3
Smoke + 1 ppm $O_3$	23	18	5.3
	guaiacol	crocol	
	glycosides	cresol glycosides	syringol glycosides
Control	U		
Control Smoke	glycosides	glycosides	glycosides



Control —— Smoke – – – Smoke + Ozone

Amelioration of smoke taint in wine using novel adsorbents and membrane filtration

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Amelioration of smoke taint in wine

#### Australian Journal of Grape and Wine Research 18, 302–307, 2012

#### Amelioration of smoke taint in wine by reverse osmosis and solid phase adsorption

#### A.L. FUDGE<sup>1</sup>, R. RISTIC<sup>1</sup>, D. WOLLAN<sup>2</sup> and K.L. WILKINSON<sup>1</sup>

#### Abstract

Background and Aims: Wines made from grapes harvested from vineyards exposed to bushfire smoke can exhibit objectionable 'smoky', 'cold ash', 'medicinal' and 'ashy' aroma and flavour characters. This study evaluated a combined reverse osmosis and solid phase adsorption process as a potential amelioration method for the treatment of smoke-tainted wines.

Methods and Results: Smoke-tainted wines were treated using either pilot or commercial scale reverse osmosis systems and the chemical composition and sensory properties of wine compared before and after treatment. The concentrations of smoke-derived volatile phenols, including marker compounds, gualacol and 4-methylgualacol, decreased significantly with treatment. As a consequence, diminished smoke-related sensory attributes enabled treated wines to be readily differentiated from untreated wines. However, the taint was found to slowly return with time, likely because of hydrolysis of glycoconjugate precursors, which were not removed during the treatment process.

Conclusions: Reverse osmosis and solid phase adsorption reduced the concentration of smoked-derived volatile phenols and improved the sensory attributes of smoke-tainted wines.

Significance of the Study: This is the first study to evaluate the amelioration of smoke taint in wine using reverse osmosis and solid phase adsorption.

Keywords: amelioration, guatacol, reverse osmosis, smoke taint, wine

#### Introduction

Aroma is an important aspect of wine quality and has therefore been the subject of considerable research. Indeed, several hundred volattle compounds have been identified in wine to date. These compounds contribute to the complexity and varietal character of wine and can originate from grapes, the action of yeast during fermentation, oak wood during barrel maturation or in the bottle with aging (Williams et al. 1981, Günata et al. 1985, Winterhalter et al. 1990, Pollnitz et al. 2004). However, not all volatile compounds make a destrable contributton to wine aroma. Some volatiles are indicative of winemaking faults; e.g. the 'bruised apple' and 'vinegar' characters associated with excessive concentrations of acetaldehyde and acetic acid due to oxidation and lactic bacteria spotlage (volatile acidity), respectively (Ribéreau-Gayon et al. 2000). In other cases, contamination by exogenous volatiles can lead to taints in wine: 2,4,6-trichloroanisole, for example, is considered to contribute to the 'musty' attribute associated with cork taint (Buser et al. 1982).

In recent years, the potential for smoke to taint grapes and wine has been a concern for some winemakers, following the occurrence of significant bushfires in the vicinity of wine grapegrowing regions. Kennison et al. (2007) demonstrated the presence of several smoke-derived volatile phenols, including guatacol and 4-methylgualacol, in wines made from smokeaffected grapes. These wines were found to exhibit objectionable 'smoky', 'cold ash', 'medicinal' and 'ashy' aroma and flavour characters (Kennison et al. 2007), with the intensity of smoke-

related sensory attributes dependent on the timing and duration of grapevine smoke exposure (Kennison et al. 2009). Vineyard exposure to smoke cannot be readily predicted or prevented, but can have a significant financial impact on grape and wine production. As such, methods which reduce the concentration of smoke-derived volatile compounds in wine, thereby mitigating the effects of smoke exposure, would be of benefit to grape growers and winemakers. Ristic et al. (2011) investigated the effect of different winemaking techniques on the extent of smoke taint in wine and found the duration of skin contact, choice of yeast strain and addition of oak chips or tannins influenced smoke-related sensory properties. These techniques can be applied by winemakers when processing smoke-affected grapes, but do not address the issue of smoke taint in wine.

Reverse osmosis is a filtration process involving diffusion across a semi-permeable membrane against a concentration gradient (Paulsen et al. 1985), in which separation efficiency relies on both size exclusion and solution-diffusion mechanisms (Cuperus and Nijhuis 1993). Reverse osmosis is routinely used for water purification and desalination (Madaeni 1999), with an increasing number of applications being reported within food and beverage industries, e.g. the preparation of milk (Glover 1971) and fruit juice concentrates (Paulsen et al. 1985, Kane et al. 1995). Within the wine industry, reverse osmosis has been used to manipulate wine alcohol content, volatile acidity and acidification through concentration of grape must (Duitschaever et al. 1991) and wine (But et al. 1988, Massot et al. 2008). Reverse osmosis has also been coupled with solid

#### Amelioration of smoke taint in wine by treatment with commercial fining agents

#### A.L. FUDGE<sup>1</sup>, M. SCHIETTECATTE<sup>1</sup>, R. RISTIC<sup>1</sup>, Y. HAYASAKA<sup>2</sup> and K.L. WILKINSON<sup>1</sup>

#### Abstract

Background and Aims: Fermentation of smoke-affected grapes can lead to wines that exhibit objectionable smoke-related sensory attributes, i.e. smoke taint. Fining agents are routinely used at different stages of the winemaking process to address constituents that are considered to adversely affect juice or wine quality. This study aimed to evaluate the efficacy of commercial fining agents in reducing the concentration of volatile phenols and the intensity of sensory attributes associated with smoke-tainted wine.

Methods and Results: Smoke-affected wines were treated with a range of fining agents, two of which, an activated carbon and a synthetic mineral, were found to appreciably ameliorate the taint. Treated wines contained a significantly lower level of smoke-derived volatile phenois and exhibited less intense 'smoke' and 'cold ash' aromas, 'smoky' flavour and 'ashy' aftertaste, compared with that of untreated (control) wines; with little or no impact on wine colour. Conclusions: Selected fining agents can ameliorate smoke taint in wine. Whereas most fining agents showed poor specificity towards the wine components responsible for smoke taint, some, an activated carbon in particular, were highly effective.

Significance of the Study: This research identifies a treatment that can be used to mitigate the impact of grapevine exposure to smoke on wine composition and sensory properties.

Keywords: activated carbon, amelioration, fining agent, gualacol, smoke taint, wine

#### Introduction

Fining agents are routinely used at different stages of winemaking to address constituents that are considered to adversely affect juice or wine quality. For example, gelatine, isinglass, silicon dioxide and bentonite have been used to facilitate the clarification of juice and wine, while egg albumin, casein, polyvinylpolypyrrolidone (PVPP) and activated carbon can remove the phenolic compounds associated with bitterness, astringency and browning (Iland et al. 2004b). Fining involves the addition of one or more adsorptive substrates to juice or wine to bind certain components, thus reducing their concentration (Castellari et al. 2001). Binding occurs as a result of complex van der Waals, resonance, electrostatic and hydrogen bonding interactions (Furuya et al. 1997) between the adsorbent (i.e. the fining agent) and the adsorbate (i.e. the component in juice or wine to be removed). Following treatment, fining agents will either settle or precipitate, allowing clear juice or wine to be separated by racking, filtration or centrifugation (Iland et al. 2004b).

Fining agents have also been used to remove volatile compounds responsible for the occurrence of various off-odours and flavours in wine. The remedial treatment of wine affected by *Harmonia axyridis* (multicoloured Astan lady beetle) taint was reported by Pickering and co-workers (2006). The concentration of 2-isopropyl-3-methoxypyrazine, derived from lady beetles present in grape bunches at the time of harvest, was reduced in white wine following the addition of activated carbon. Activated carbon and PVPP have also been shown to reduce the concentration of 4-ethylphenol and 4-ethylguatacol in wine (Lisanti et al. 2008): i.e. the volatile phenols associated with *Bretianomyces/Dekkera* spollage (Chatonnet et al. 1992). The adsorptive properties of oenological fining agents have even been exploited for the removal of ochratoxin A from wines (Castellari et al. 2001, Gambuti et al. 2005, Kurtbay et al. 2008).

In the last 5 years, an increasing number of studies concerning grapevine exposure to smoke and the occurrence of smoke taint in grapes and wine have been published in the scientific literature. Several studies have described the compositional and sensory implications of grapevine smoke exposure for grapes and wine (Kennison et al. 2007, 2008, Sheppard et al. 2009, Hayasaka et al. 2010a,b,c) and the influence of grapevine phenology on the extent of smoke taint in wine (Kennison et al. 2009, 2011). Additionally, a range of chromatographic and spectroscopic methods has been developed for the identification and screening of smoke-tainted grapes and wine (Dungey et al. 2011, Singh et al. 2011, Wilkinson et al. 2011, Fudge et al. 2012). To date, however, only two studies have investigated methods by which the impact of grapevine exposure to smoke on the chemical and sensory profiles of wine can be mitigated. Ristic and co-workers reported the influence of winemaking techniques, such as the duration of skin contact, yeast selection and the use of oak and tannin additives, on the concentration of smoke-derived volatile phenols and intensity of smoke-related sensory attributes in wines made from smoke-affected grapes (Ristic et al. 2011). The capacity of reverse osmosis and solidphase adsorption for the amelioration of smoke-tainted wine has also been demonstrated (Fudge et al. 2011); with treated wines shown to contain a reduced level of volatile phenols and dimin-Ished 'smoke' and 'ash' aromas and flavours, compared with that of untreated wines. While these studies give winemakers some options for managing smoke-affected grapes and wine, further

# Novel adsorbents: molecularly imprinted polymers (MIPs)

MIPs are mm-sized polymerisation products, with high surface area:size and adsorptive properties

Designed for selective removal of target molecules, e.g. volatile phenols



#### Novel adsorbents project: screening trial

2020 smoke-affected Chardonnay treated with adsorbents for 1, 3 or 7 days



# Novel adsorbents project: adsorption trial

Adsorbent	dose	guaiacol	cresols	syringol
Chardonnay	2 g/L, 24 hours	31	33	39
Chardonnay + Carbon 1	2 g/L, 24 hours	28	27	35
Chardonnay + Carbon 2	2 g/L, 24 hours	28	29	33
Chardonnay + Carbon 3	2 g/L, 24 hours	27	29	33
Chardonnay + VAF Resin	20 g/L, 2 hours	23	25	29
Chardonnay + MIPs	10 g/L, 6 hours	19	17	28

### Novel adsorbents project: adsorption trial

Adsorbent	guaiacol	cresols	syringol
Chardonnay	31	33	39
Ch + Carbon 1	28	27	35
Ch + Carbon 2	28	29	33
Ch + Carbon 3	27	29	33
Ch + VAF Resin	23	25	29
Ch + MIPs	19	17	28



#### TASTING







#### Sample 1 Smoke Wine

Sample 2 Smoke Wine + MIPs Sample 3 Smoke Wine + Resin

# Novel adsorbents project: ongoing R&D Optimisation of resin and MIP treatments direct addition vs column dose rate/bed volumes duration/flow rate regeneration

Additionally, use of adsorbents in combination with membrane filtration



Adsorbent	guaiacol	cresols	syringol
Chardonnay	32	40	41
UF5 permeate	30	39	38
UF5 retentate	29	39	38
UF10 permeate	28	36	35
UF10 retentate	29	39	37
UF20 permeate	29	37	38
UF20 retentate	29	37	37

Adsorbent	guaiacol	cresols	syringol	GuRu	4MGRu	PhRu	CrRu	SyrGB	4MSyrGB
Chardonnay	32	40	41	8	17	2	3	468	37
UF5 permeate	30	39	38	6	12	2	2	280	20
UF5 retentate	29	39	38	24	52	6	7	1467	138
UF10 permeate	28	36	35	7	14	2	2	368	26
UF10 retentate	29	39	37	16	35	4	5	910	79
UF20 permeate	29	37	38	7	16	2	2	423	32
UF20 retentate	29	37	37	12	25	3	4	681	57

Adsorbent	guaiacol	cresols	syringol	GuRu	4MGRu	PhRu	CrRu	SyrGB	4MSyrGB
Chardonnay	32	40	41	8	17	2	3	468	37
UF5 permeate	30	39	38	6	12	2	2	280	20
UF5 retentate	29	39	38	24	52	6	7	1467	138
UF10 permeate	28	36	35	7	14	2	2	368	26
UF10 retentate	29	39	37	16	35	4	5	910	79
UF20 permeate	29	37	38	7	16	2	2	423	32
UF20 retentate	29	37	37	12	25	3	4	681	57
UF5+NF permeate	28	34	35	0	0	0	0	3	0
UF5+NF retentate	27	33	42	46	110	13	18	1835	191
UF10+NF permeate	26	32	28	0	0	0	0	2	0
UF10+NF retentate	25	29	37	63	152	18	24	2450	269
UF20+NF permeate	28	34	34	0	0	0	0	4	0
UF20+NF retentate	27	32	41	60	147	17	23	2498	289

Amelioration of smoke tainted wine via spinning cone column distillation

#### Spinning Cone Column distillation project:



#### Spinning Cone Column distillation project:



	guaiacol	cresols	syringol	GuR	MGuR	CrR	PhR	SyGB	MSyGB
ShS wine	48	29	12	41	37	27	26	112	7
							fruit A		
						acidity	6 5	smoke A	
					motallia AT		- /	/	old ash
					metallic AT		3		olu asti
							$^{2}$		
				d	rying AT				— burnt rubbe
						$\times$			
					woody AT				medicinal A
								$\overline{\}$	
					ashy AT	/		fruit F	
						/ medicinal F	\ smo	oky F	
						_	<ul> <li>Untreated</li> </ul>		

	guaiacol	cresols	syringol
ShS wine	48	29	12
1% strip	50	28	13
14% strip	52	30	15
28% strip	46	29	18
1% strip condensate	6	nd	nd
14% strip condensate	7	1	nd
28% strip condensate	16	5	nd

	guaiacol	cresols	syringol	GuR	MGuR	CrR	PhR	SyGB	MSyGB
ShS wine	48	29	12	41	37	27	26	112	7
1% strip	50	28	13	40	34	24	26	107	7
14% strip	52	30	15	47	42	28	32	127	8
28% strip	46	29	18	55	51	35	38	152	11
1% strip condensate	6	nd	nd	nd	nd	nd	nd	nd	nd
14% strip condensate	7	1	nd	nd	nd	nd	nd	nd	nd
28% strip condensate	16	5	nd	nd	nd	nd	nd	nd	nd

	guaiacol	cresols	syringol
ShS wine	48	29	12
1% strip	50	28	13
14% strip	52	30	15
28% strip	46	29	18
1% strip condensate	6	nd	nd
14% strip condensate	7	1	nd
28% strip condensate	16	5	nd





RED JUICE PROCESSING	ED JUICE PROCESS	ING	
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	guaiacol	cresols	syringol	GuR	SyrGB
red juice	10	6	nd	19	85
clarified red juice	9	6	nd	19	84
condensate (pre-IEX)	42	23	5	nd	nd
condensate (post-IEX)	1	nd	nd	nd	nd
reconstituted red juice	2	1 [	22	20	62
red wine	4	3	30	na	na



Transformation of smoke tainted wine into spirit via distillation

# Distillation project: base wine

	guaiacol	cresols	syringol	GuR	MGuR	CrR	PhR	SyGB	MSyGB
Wine	53	53	10	7	9	8	5	35	3
						fı	ruit A		
					bitternes	5		, smoke A	
						4		/	
				met	allic AT	3		cc	ld ash
						2		$\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{\mathbf{$	
				druing A	-	$\mathbf{X}^{\mathbf{A}}$			— burnt rubbe
				drying A					
						$\sim$			
				woody	AT				medicinal A
								$\backslash$	
					ashy AT		$\checkmark$	fruit F	
					asity AT			nuit i	
					m	edicinal F	smoky	۲F	

# Distillation project: batch distillation

	guaiacol	cresols	syringol	GuR	MGuR	CrR	PhR	SyGB	MSyGB
Wine (2 L)	53	53	10	7	9	8	5	35	3
Low wine (1.5 L)	93	62	11	nd	nd	nd	nd	nd	nd
Stillage (0.5 L)	38	10	505	83	110	96	59	194	23

### Distillation project: batch distillation & addition of carbon

	guaiacol	cresols	syringol	GuR	MGuR	CrR	PhR	SyGB	MSyGB
Wine	53	53	10	7	9	8	5	35	3
Low wine	93	62	11	nd	nd	nd	nd	nd	nd
Stillage	38	10	505	83	110	96	59	194	23
Low wine + AC	2	nd	nd	nd	nd	nd	nd	nd	nd

# Distillation project: fractional distillation trial

	guaiacol	cresols	syringol
Wine	53	53	10
Low wine	93	62	11
Fraction 1 (>93% abv)	9	26	26
Fraction 2 (>94% abv)	16	32	24
Fraction 3 (>94% abv)	20	29	22
Fraction 4 (~10% abv)	1182	218	23

(with rectification, without vacuum)















#### Sample 7 Base Wine

Sample 8 Hearts Blend

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Wine Australia

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