COMPARING THE CHEMICAL AND SENSORY CONSEQUENCES OF GRAPEVINE SMOKE EXPOSURE IN GRAPES AND WINE FROM DIFFERENT CULTIVARS AND DIFFERENT WINE REGIONS IN AUSTRALIA

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Abstract

Aim: This study aimed to benchmark the chemical and sensory consequences of grapevine exposure to smoke, by comparing: (i) the concentration of volatile phenols and volatile phenol glycosides in control and smoke-affected grapes from different cultivars and different wine regions; and (ii) the chemical and sensory profiles of wines made from control and smoke-affected grapes, from different cultivars.

Methods and Results: Control and smoke-affected grapes and wines were sourced from a combination of: experimental trials (involving the application of smoke to different grapevine cultivars); and commercial vineyards located in Australian wine regions, some of which were exposed to bushfire smoke during the 2019/20 growing season. The concentrations of smoke taint marker compounds were determined in grapes and wine by gas chromatography-mass spectrometry and liquid chromatography-tandem mass spectrometry; while wine sensory profiles were determined by descriptive analysis.

Conclusions: Volatile phenols and volatile phenol glycosides remain useful chemical markers of smoke taint. Volatile phenol concentrations (in free and glycosylated forms) varied by cultivar and wine region, which likely reflects varietal differences in the naturally occurring ('background') levels of volatile phenols, and the density and duration of smoke exposure experienced in different regions.

Significance and Impact of the Study: Research findings provide an initial benchmark of the 'background' levels of free and glycosylated volatile phenols that can occur naturally in grapes from different cultivars, as well as the concentrations of smoke taint marker compounds present in smoke-affected grapes and wine. These results can be used by industry to inform decisions around harvesting vs. rejecting smoke-affected grapes, albeit a greater understanding of baseline volatile phenol levels by cultivar and region is needed.

Keywords: Cresol, guaiacol, smoke taint, syringol, volatile phenols, volatile phenol glycoconjugates

Introduction

When bushfires occur near wine regions, vineyard exposure to smoke can taint grapes, and therefore wine (Krstic *et al.,* 2015), depending on the timing and duration of smoke exposure (Kennison *et al.,* 2009; Kennison *et al.,* 2011). Wines made from smoke-affected grapes can exhibit unpleasant smoky and ashy characters (Kennison *et al.,* 2007; Kennison *et al.,* 2008; Parker *et al.,* 2012; Ristic *et al.,* 2016), now colloquially referred to as 'smoke taint'. Several volatile phenols (guaiacols, cresols and syringols) are routinely measured in free and glycosylated forms (Hayasaka *et al.,* 2010; Hayasaka *et al.,* 2013; Noestheden *et al.,* 2018; Szeto *et al.,* 2020), as markers of smoke taint. However, the occurrence of volatile phenols (and their glycoconjugates) as natural constituents of some grape cultivars, Shiraz in particular (Wilkinson *et al.,* 2011, Ristic *et al.,* 2016), can confuse the detection and quantification of smoke taint. Volatile phenols can also be extracted into wine during barrel fermentation and maturation (Pollnitz *et al.,* 2004), often at levels higher than those observed in wines made from smoke-affected grapes. Thus, oak treatment can also complicate smoke taint analysis.

Recent research suggests volatile phenols adsorbed by grapes during smoke exposure are rapidly metabolised (Szeto *et al.,* 2020). Whereas volatile phenols were detected in grapes 1 hour after smoke exposure, their concentrations had decreased by as much as 75% within 24 hours of smoke exposure, presumably due to glycosylation. Volatile phenol glycosides were detected in grapes sampled 1 and 7 days post-smoke exposure, but significant increases in glycoside concentrations were observed between 7 and 28 days post-smoke exposure. The phenological timing of smoke exposure might therefore affect the extraction (and detection) of volatile phenol glycosides. This can be overcome by acid hydrolysis of grape juice or homogenate (i.e. adjustment of pH to 1.0, followed by heating at 100°C for 1 hour), which cleaves glycoside bonds to release free volatile phenols (Kennison *et al.,* 2008; Noestheden *et al.,* 2018). However, sample preparation needs to be carefully considered to avoid incomplete hydrolysis and/or analyte losses; in particular, the use of PTFE tubes for acid hydrolysis (rather than borosilicate glass vials) is recommended (Noestheden *et al.,* 2018).

Around the world, commercial and research laboratories perform smoke taint analysis using combinations of either direct measurement of volatile phenols and volatile phenol glycosides (by gas chromatography-mass spectrometry (GC-MS) and liquid chromatography-mass spectrometry (LC-MS) respectively), and indirect measurement of bound volatile phenols (by GC-MS) following acid hydrolysis treatment of samples. It can be difficult to make direct comparisons between data reported from different laboratories, when different analytical methods are used to measure different smoke taint markers.

This study therefore sought to share compositional data for smoke-affected grapes and wines generated following vineyard exposure to bushfires smoke in several Australian wine regions during the 2019/20 growing season. The natural (background) levels of volatile phenols (in free and glycosylated forms) detected in grapes which were not affected by smoke will also be reported. Additionally, the chemical and sensory profiles of smoke-affected wines of different cultivars, and their corresponding control wines, are included to enable a comparison of the volatile phenol levels detected in wines with perceivable smoke taint. It is hoped that these results can be used by industry to benchmark analytical data from commercial laboratories, thereby enabling more informed decisions to be made around harvesting vs. rejecting grapes suspected of being smoke tainted.

Materials and Methods

Control grapes (51 samples, comprising 13 different cultivars) were sourced from vineyards located in Western Australian wine regions that were not exposed to bushfire smoke during the 2019/20 growing season. Smoke-affected grapes (365 samples, comprising 29 different cultivars) were sourced from vineyards located in 20 Australian wine regions that were exposed to bushfire smoke (of unknown density and duration) during the 2019/20 growing season. Wines made from smoke-affected grapes (92 samples, predominantly Shiraz) grown in vineyards in the Barossa Valley that were exposed to bushfire smoke (again, of unknown density and duration) were also sourced.

The concentrations of seven volatile phenols (guaiacol, 4-methylguaiacol, *o*-, *m*- and *p*-cresol, syringol and 4-methylsyringol) and six volatile phenol glycosides (syringol gentiobioside, 4-methylsyringol gentiobioside, phenol rutinoside, cresol rutinoside, guaiacol rutinoside and 4-methylguaiacol rutinoside) were measured in grapes and wine by the Australian Wine Research Institute's Commercial Services Laboratory (Adelaide, SA, Australia) using stable isotope dilution analysis methods described previously (Hayasaka *et al.,* 2010; Hayasaka *et al.,* 2013). These publications describe the preparation of internal standards, method validation and instrumental operating conditions. The limit of quantification for volatile phenols and volatile phenol glycosides was 1–2 µg/L and 1 µg/L, respectively.

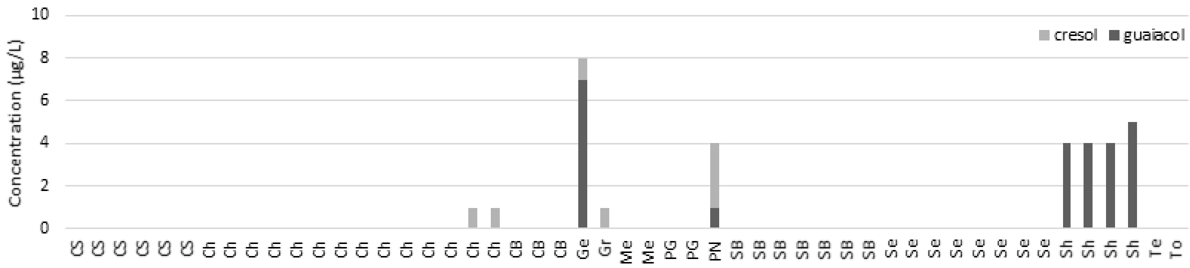
For comparative purposes, volatile phenol and sensory data for smoke-tainted Chardonnay, Sauvignon Blanc, Pinot Gris, Pinot Noir, Shiraz and Cabernet Sauvignon wines, their corresponding control wines and acid hydrolysates, are also reported. This data comes from previously published studies investigating: (i) the impact of grapevine exposure to smoke on vine physiology and the composition and sensory properties of wine (Ristic *et al.,* 2016); and (ii) the impact of bottle aging on smoke tainted wines from different grape cultivars (Ristic *et al.,* 2017). Data is reproduced with permission from Springer Nature and the American Chemical Society.

Data were analysed by analysis of variance using GenStat (19th Edition, VSN International Limited, Herts, UK). Mean comparisons were performed by least significant difference (LSD) multiple comparison test at $P<0.05$.

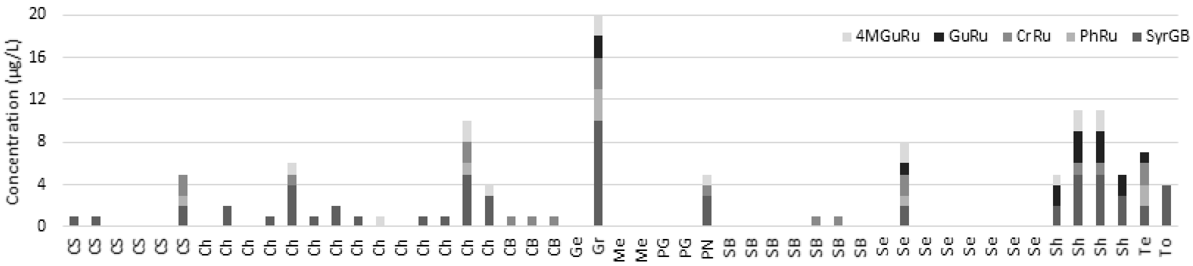
Results and Discussion

Of the 51 control (unsmoked) grape samples that were analysed, only nine contained detectable levels of volatile phenols (Figure 1a). Guaiacol was found at 1–7 µg/L in six samples, with 4–5 µg/L of guaiacol found in Shiraz samples, consistent with previous reports that guaiacol is a natural constituent of this cultivar (Wirth *et al.,* 2001; Ristic *et al.,* 2016). Surprisingly, the highest guaiacol concentration (7 µg/L) was detected in a Gewurztraminer grape sample, but given there was only one sample for this cultivar, it is not clear if this is representative of Gewurztraminer. Syringol was found at 1 µg/L in four samples and at 3 µg/L in a Pinot Noir samples. Again, there was only one Pinot Noir sample so it is not clear if this result is representative.

Volatile phenol glycosides were detected in 28/51 grape samples (Figure 1b), including eight of the samples which were found to contain volatile phenols. Collectively, these eight samples accounted for 60% of the total glycoside pool; with four additional samples (a Cabernet Sauvignon, a Chardonnay, a Semillon and a Tempranillo) contributing a further 22% of the glycoside pool. Syringol gentiobioside was the most abundant glycoside. It was detected in 22 grape samples and contributed ~50% of the total glycoconjugate pool. 4-Methylsyringol gentiobioside was not detected in any of the control grape samples.



(a) volatile phenols



(b) volatile phenol glycosides

Figure 1: Concentrations (µg/L) of (a) volatile phenols and (b) volatile phenol glycosides detected in grapes (of different cultivars) that were not exposed to bushfire smoke. 4MGuRu = 4-methylguaiacol rutinoside, GuRu = guaiacol rutinoside, CrRu= cresol rutinoside, PhRu = phenol rutinoside, SyrGB = syringol gentiobioside.

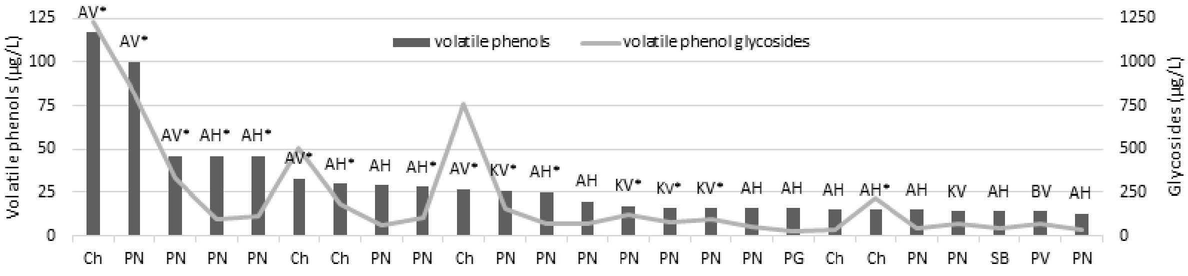
One or more volatile phenols were detected in 189 of the 365 smoke-affected grape samples analysed, while volatile phenol glycosides were detected in 333 samples (Table 1). Whereas guaiacol was detected in 164 samples, at up to 38 µg/L and 4 µg/L on average, cresols were detected in fewer samples, but at higher concentrations, i.e. at 7 µg/L on average and as high as 63 µg/L. Syringol and 4-methylsyringol were not detected in any smoke-affected grape samples and 4-methylguaiacol was detected in only 25 samples. Syringol gentiobioside was again the most abundant volatile phenol glycoside measured; it accounted for 53% of the total glycoside pool and on average, was present at several-fold higher concentrations than other glycosides.

Table 1: Concentrations of volatile phenols and volatile phenol glycosides detected in 365 grape samples (of different cultivars) that were exposed to bushfire smoke (of unknown density and duration) during the 2019/20 growing season. Gu = guaiacol, 4MGu = 4-methylguaiacol, Cr = cresols, Syr = syringol, 4MSyr = 4-methylsyringol, SyrGB = syringol gentiobioside, 4MSyrGB = 4-methylsyringol gentiobioside, PhRu = phenol rutinoside, CrRu= cresol rutinoside, GuRu = guaiacol rutinoside, 4MGuRu = 4-methylguaiacol rutinoside; nd = not detected.

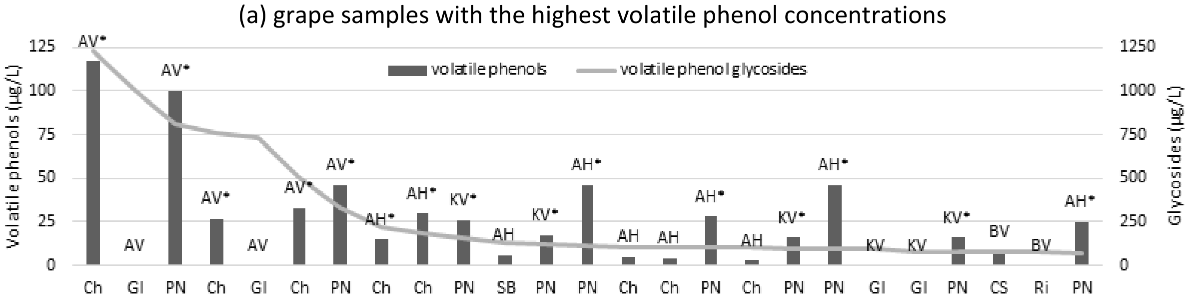
	volatile phenols (µg/L)					volatile phenol glycosides (µg/L)					
	Gu	4MGu	Cr	Syr	4MSyr	SyrGB	4MSyrGB	PhRu	CrRu	GuRu	4MGuRu
# samples	164	25	86	0	0	295	215	244	299	281	280
range	1–38	1–19	1–63	nd	nd	1–757	1–219	1–54	1–62	1–31	1–137
mean	4	3	7	nd	nd	28	8	4	5	5	6

A subset of 167 samples contained volatile phenol glycosides at concentrations comparable to or less than those observed in control grapes (i.e. ≤20 µg/L, data not shown), suggesting minimal, if any, smoke exposure. 47 of these samples (including 21 Shiraz, 11 Pinot Noir, 4 Chardonnay and 3 Cabernet Sauvignon samples) contained 1–7 µg/L of guaiacol, *o*-cresol and/or *p*-cresol, which might reflect natural (background) levels.

When the composition of samples with the highest volatile phenol and/or volatile phenol glycoside levels were compared (Figure 2), 35 grape samples accounted for 63% and 50% of the total volatile phenol and volatile phenol glycoside pools, respectively. This included 16 Pinot Noir, 9 Chardonnay, 3 Sauvignon Blanc, 2 Cabernet Sauvignon and 2 Shiraz samples, from four wine regions: the Adelaide Hills (18 samples), the Alpine and King Valleys (7 samples each), and the Barossa Valley (3 samples). While total volatile phenol and volatile phenol glycoside levels were strongly correlated (0.839) for the 25 samples containing the highest concentrations of volatile phenols (Figure 2a), a weaker correlation (0.550) was observed for the 25 samples containing the highest volatile phenol glycosides levels (Figure 2b). The proportion of smoke-derived volatile phenols present in grapes in free vs. glycosylated forms likely depends on how recently smoke exposure occurred. As indicated above, berry physiology, and therefore the phenological timing of smoke exposure, might also influence the extraction of volatile phenol glycosides from grapes, and as such, their direct quantification by LC-MS.



(a) grape samples with the highest volatile phenol concentrations



(b) grape samples with the highest volatile phenol glycoside concentrations

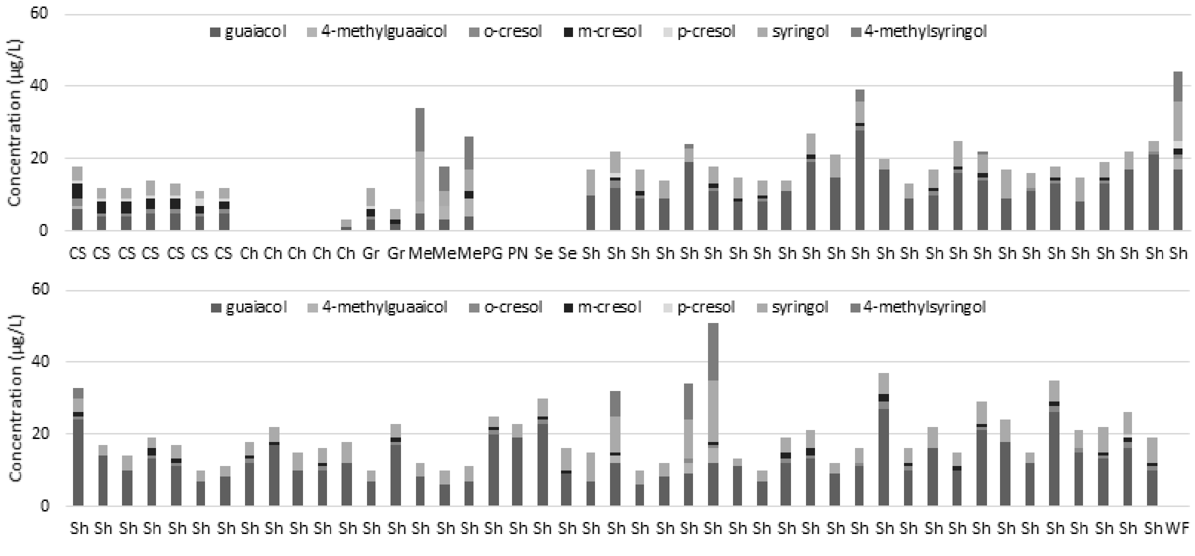
Figure 2: Composition of smoke-affected grapes (of different cultivars) found to contain the highest (a) volatile phenol and (b) volatile phenol glycoside concentrations. Samples came from various Australian wine regions, including: AV = Alpine Valley, AH = Adelaide Hills, KV = King Valley, BV = Barossa Valley. * denotes samples found to contain the highest levels of both volatile phenols and volatile phenol glycosides.

Chemical analysis of the 92 wines made from smoke-affected grapes led to the detection of volatile phenols at concentrations up to 51 µg/L (and 17 µg/L on average) in 85 samples (Figure 3a). With the exception of the one Pinot Noir wine, the samples which did not contain volatile phenols were all white wines, which likely reflects their reduced skin contact during winemaking (and thus limited extraction of any smoke taint compounds), as reported in previous studies (Ristic *et al.,* 2011; Ristic *et al.,* 2016). The remaining white wine (a Chardonnay) contained 1 µg/L of guaiacol and 2 µg/L of syringol, compared with volatile phenol levels ≥6 µg/L for the other red wines. Guaiacol and syringol were the most abundant volatile phenols and on average accounted for 60% and 26% of the volatile phenols observed, respectively. Interestingly, the Cabernet Sauvignon wines comprised the highest cresol levels (at 4–7 µg/L). However, it is not clear if this reflects increased smoke exposure of Cabernet Sauvignon grapes (since total volatile phenol levels were only 11–18 µg/L) or an effect of cultivar.

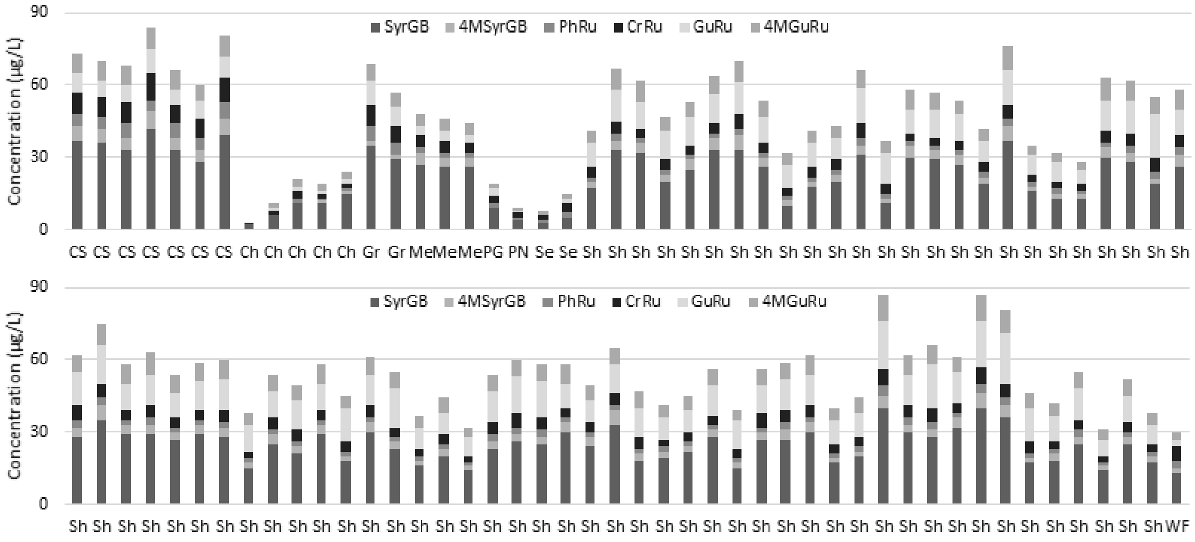
Volatile phenol glycosides were detected in all 92 wines (Figure 3b), but concentrations were ≤30 µg/L for the white and Pinot Noir wine. Again, this likely due to reduced skin contact during winemaking in the case of the white wines. In contrast, volatile phenol glycoside concentrations ranged from 28 to 87 µg/L (and averaged 55 µg/L) for the red wines (excluding the Pinot Noir). Cabernet Sauvignon wines were amongst the wines with the highest total glycoside levels (at ≥60 µg/L); again it's not clear if this reflects increased exposure to smoke, varietal susceptibility to smoke or naturally occurring background levels of volatile phenol glycosides. Of the volatile phenol glycosides that were measured, guaiacol rutinoside and syringol gentiobioside were again the most abundant, but in contrast to their free forms, syringol gentiobioside accounted for the lion share of total glycosides, i.e. 47%; while guaiacol rutinoside accounted for a further 20% of the pool of glycosides measured.

Correlation coefficients were calculated for the various smoke taint marker compounds, but few meaningful correlations were observed. 4-Methylguaiacol was detected in only eight wines, but it was usually detected in wines which also contained 4-methylsyringol, such that a strong correlation (0.918) was observed for these two volatile phenols. Guaiacol and guaiacol rutinoside concentrations were correlated (0.776), but otherwise, strong correlations (>0.700) were not observed between volatile phenols and their corresponding glycosides. The concentrations of phenol and cresol rutinosides were strongly correlated (0.912), while 4-methylsyringol gentiobioside and guaiacol rutinoside levels correlated with 4-methylguaiacol rutinoside concentrations (0.823 and 0.761, respectively). Correlation coefficients for syringol gentiobioside and other glycosides were ≥0.585, but were highest for 4-methylsyringol gentiobioside (0.895) and 4-methylguaiacol rutinoside (0.912).

In the absence of sensory data for these 92 wine samples, it is unclear whether the concentrations of smoke taint marker compounds observed in wines correspond to perceivable levels of smoke taint. However, chemical and sensory data for control and smoke-affected Chardonnay, Sauvignon Blanc, Pinot Gris, Pinot Noir, Shiraz and Cabernet Sauvignon wines has previously been reported in the literature (Ristic *et al.,* 2016) and can be used to benchmark grape and wine compositional data from the current study.



(a) volatile phenols



(b) volatile phenol glycosides

Figure 3: Concentrations of (a) volatile phenols and (b) volatile phenol glycosides detected in wines made from grapes (of different cultivars) that were exposed to bushfire smoke (of unknown density and duration) during the 2019/20 growing season. SyrGB = syringol gentiobioside, 4MSyrGB = 4-methylsyringol gentiobioside, PhRu = phenol rutinoside, CrRu= cresol rutinoside, GuRu = guaiacol rutinoside, 4MGuRu = 4-methylguaiacol rutinoside.

Control wines, irrespective of cultivar, were characterised by fruit aromas and flavours, and in the case of the full-bodied red wines, earthy notes also (Figure 4). In contrast, smoke-affected wines displayed varying degrees of smoke, cold ash and medicinal aromas, smoke flavour and an ashy aftertaste; in the most tainted wines, fruit aromas and flavours were diminished by smoke attributes. Small (but statistically significant) increases in the intensity of smoke aroma provided evidence of smoke taint in the Chardonnay and Sauvignon Blanc wines made from smoke-affected grapes. Chemical analysis supported this finding (Table 2); volatile phenols were not detected in any of the control white wines, but low levels of guaiacol and syringol (1–2 µg/L) were detected in smoke-affected Chardonnay and Sauvignon Blanc wines. Higher volatile phenol levels were detected in the smoke-affected Pinot Gris wine, which resulted in a perceivable smoke taint (Figure 4c). Background levels of guaiacol, cresol and syringol were detected in all three control red wines (at up to 9 µg/L), but Shiraz in particular. However, levels were two- to five-fold higher in the corresponding smoke-affected red wines. The Pinot Noir, Shiraz and Cabernet Sauvignon exhibited increasing intensities of smoke-related attributes (Figure 4), in good agreement with the difference in volatile phenols observed for control and smoke-affected wines.

Based on the volatile phenol levels reported in Table 2, the white wines shown in Figure 3 might reasonably be expected to exhibit minimal, if any, smoke taint; the red wines with elevated concentrations of volatile phenols and/or volatile phenol glycosides (e.g. the Merlot, Grenache and Cabernet Sauvignon wines), however, may well prove to be smoke tainted. Predicting the level of smoke taint in Shiraz wines is complicated by its high background levels of volatile phenols, especially guaiacol and syringol. Nevertheless, the levels of smoke taint markers observed in at least a few of the Shiraz wines look to be indicative of smoke taint.

The cresols may yet prove to be a better marker of smoke taint than guaiacol and syringol, since the cresols tend to occur at much lower background levels, even after acid hydrolysis (Table 2). Acid hydrolysis did not liberate volatile phenols from control white wines, but gave increased guaiacol and syringol concentrations for control red wines, particularly Shiraz and Cabernet Sauvignon. Acid hydrolysis of smoke-affected samples might therefore aid smoke taint detection (especially in the absence of LC-MS instrumentation and capability), pending the aforementioned sample preparation considerations.

Conclusions

Volatile phenols (and their glycosides) remain useful chemical markers of smoke taint in both grapes and wine, albeit a greater understanding of the baseline levels that occur naturally in grapes of different cultivars, and any regional variation, is needed to better support decision-making with regards to managing smoke-affected grapes and wine. Industry might benefit from establishing an archive of free and glycosylated volatile phenol levels for individual vineyards, especially grape and wine producers in smoke prone regions.

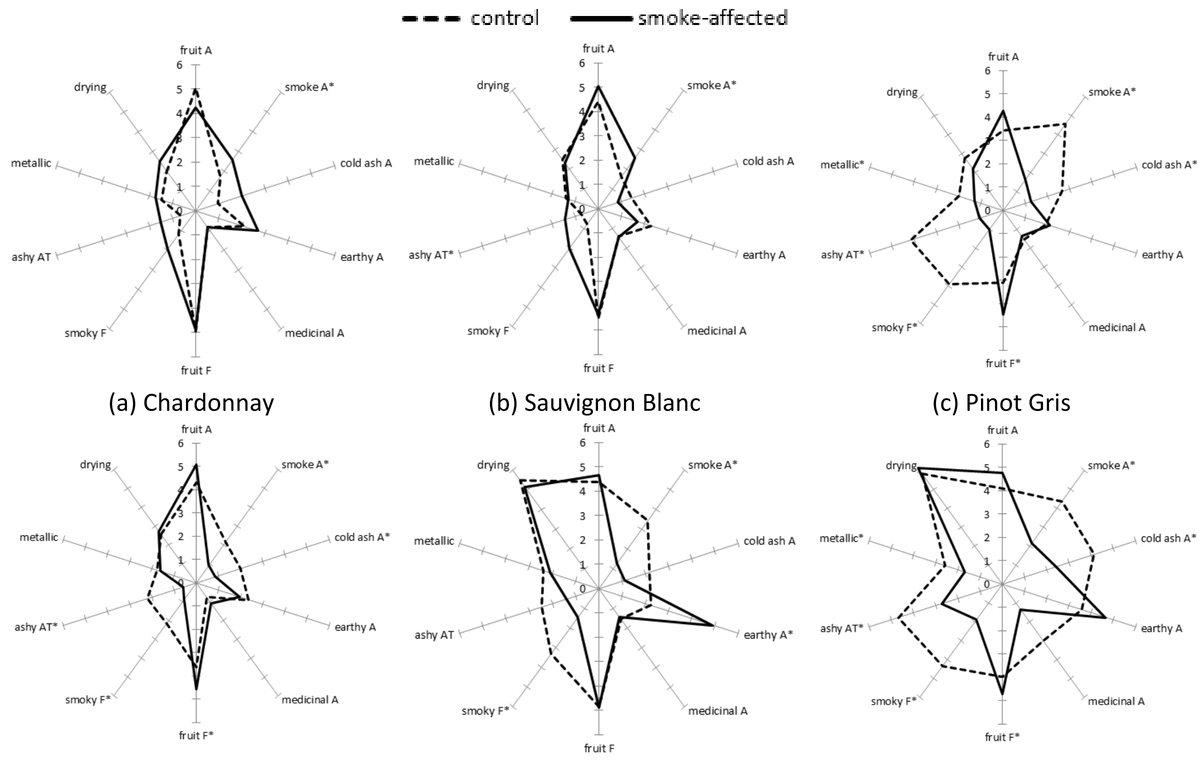


Figure 4: Sensory profiles of control and smoke-affected (a) Chardonnay, (b) Sauvignon Blanc, (c) Pinot Gris, (d) Pinot Noir, (e) Shiraz and (f) Cabernet Sauvignon wines. Adapted with permission from Springer Nature: Theor. Exp. Plant Physiol., Ristic et al., 2016. A = aroma, F = flavour, AT = aftertaste, * denotes statistical significance (*P = 0.05*, one-way ANOVA).

Table 2: Concentrations of volatile phenols in control (C) and smoke-affected (S) Chardonnay, Sauvignon Blanc, Pinot Gris, Pinot Noir, Shiraz and Cabernet Sauvignon wines, and in acid hydrolysates of wines (acid hydrolysis was performed after wines had been bottle aged for 6 years).

		wine volatile phenols† (µg/L)				hydrolysate volatile phenols‡ (µg/L)			
		Gu	4MGu	Cr	Syr	Gu	4MGu	Cr	Syr
Chardonnay	C	nd	nd	nd	nd	nd	nd	nd	nd
	S	1	nd	nd	nd	13	4	4	22
Sauvignon Blanc	C	nd	nd	nd	nd	nd	nd	nd	nd
	S	2	nd	nd	1	15	4	5	30
Pinot Gris	C	nd	nd	nd	nd	nd	nd	nd	nd
	S	10	nd	8	2	25	6	9	44
Pinot Noir	C	nd	nd	1	2	3	nd	1	2
	S	6	nd	8	3	14	3	6	35
Shiraz	C	9	nd	3	8	19	nd	3	38
	S	26	2	10	10	106	17	19	153
Cabernet Sauvignon	C	2	nd	5	7	3	nd	2	26
	S	20	nd	17	10	36	7	13	120

Gu = guaiacol, 4MGu = 4-methylguaiacol, Cr = cresols, Syr = syringol, 4MSyr = 4-methylsyringol; nd = not detected.
† Adapted with permission from Springer Nature: Theor. Exp. Plant Physiol., Ristic et al., 2016.
‡ Adapted with permission from the American Chemical Society: J. Agric. Food Chem., Ristic et al., 2018.

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References

Hayasaka, Y., Baldock, GA., Parker, M., Pardon, KH., Black, CA., Herderich, MJ., Jeffery, DW., 2010. Glycosylation of smoke derived volatile phenols in grapes as a consequence of grapevine exposure to bushfire smoke. Journal of Agricultural and Food Chemistry, 58: 10989-10998.

Hayasaka, Y., Parker, M., Baldock, GA., Pardon, KH., Black, CA., Jeffery, DW., Herderich, MJ., 2013. Assessing the impact of smoke exposure in grapes: development and validation of an HPLC-MS/MS method for the quantitative analysis of smoke-derived phenolic glycosides in grapes and wine. Journal of Agricultural and Food Chemistry, 61: 25-33.

Kennison, KR., Wilkinson, KL., Williams, HG., Smith, JH., Gibberd, MR., 2007. Smoke-derived taint in wine: Effect of postharvest smoke exposure of grapes on the chemical composition and sensory characteristics of wine. Journal of Agricultural and Food Chemistry, 55: 10897-10901.

Kennison, KR., Gibberd, MR., Pollnitz, AP., Wilkinson, KL., 2008. Smoke-derived taint in wine: The release of smoke-derived volatile phenols during fermentation of Merlot juice following grapevine exposure to smoke. Journal of Agricultural and Food Chemistry, 56: 7379-7383.

Kennison, KR., Wilkinson, KL., Pollnitz, AP., Williams, HG., Gibberd, MR., 2009. Effect of timing and duration of grapevine exposure to smoke on the composition and sensory properties of wine. Australian Journal of Grape and Wine Research, 15: 228-237.

Kennison, KR., Wilkinson, KL., Pollnitz, AP., Williams, HG., Gibberd, MR., 2011. Effect of smoke application to field-grown Merlot grapevines at key phenological growth stages on wine sensory and chemical properties. Australian Journal of Grape and Wine Research, 17: S5-S12.

Krstic, MP., Johnson, DL., Herderich, MJ., 2015. Review of smoke taint in wine: smoke-derived volatile phenols and their glycosidic metabolites in grapes and vines as biomarkers for smoke exposure and their role in the sensory perception of smoke taint. Australian Journal of Grape and Wine Research, 21: 537-553.

Noestheden, M., Dennis, EG., Romero-Montalvo, E., DiLabio, GA., Zandberg, WF., 2018. Detailed characterization of glycosylated sensory-active volatile phenols in smoke-exposed grapes and wine. Food Chemistry, 259: 147-156.

Parker, M., Osidacz, P., Baldock, GA., Hayasaka, Y., Black, CA., Pardon, KH., Jeffery, DW., Geue, JP., Herderich, MJ., Francis, IL., 2012. Contribution of several volatile phenols and their glycoconjugates to smoke-related sensory properties of red wine. Journal of Agricultural and Food Chemistry, 60: 2629-2637.

Pollnitz, AP., Pardon, KH., Sykes, M., Sefton MA., 2004. The effects of sample preparation and gas chromatograph injection techniques on the accuracy of measuring guaiacol, 4-methylguaiacol and other volatile oak compounds in oak extracts by stable isotope dilution analyses. Journal of Agricultural and Food Chemistry, 52: 3244-3252.

Ristic, R., Osidacz, P., Pinchbeck, K., Hayasaka, Y., Fudge, A., Wilkinson, K., 2011. The effect of winemaking techniques on the intensity of smoke taint in wine. Australian Journal of Grape and Wine Research, 17: S29-S40.

Ristic, R., Fudge, AL., Pinchbeck, KA., De Bei, R., Fuentes, S., Hayasaka, Y., Tyerman, SD., Wilkinson, KL., 2016. Impact of grapevine exposure to smoke on vine physiology and the composition and sensory properties of wine. Theoretical and Experimental Plant Physiology, 28: 67-83.

Ristic, R., van der Hulst, L., Capone, DL., Wilkinson, KL., 2017. Impact of bottle aging on smoke-tainted wines from different grape cultivars. Journal of Agricultural and Food Chemistry, 65: 4146-4152.

Szeto, C., Ristic, R., Capone, D., Puglisi, C., Pagay, V., Culbert, J., Jiang, W., Herderich, M., Tuke, J., Wilkinson, K., 2020. Uptake and glycosylation of smoke-derived volatile phenols by Cabernet Sauvignon grapes and their subsequent fate during winemaking. Molecules, 25: 3720.

Wirth, J., Guo, W., Baumes, R., Günata, Z., 2001. Volatile compounds released by enzymatic hydrolysis of glycoconjugates of leaves and grape berries from *Vitis vinifera* Muscat of Alexandria and Shiraz cultivars. Journal of Agricultural and Food Chemistry, 49: 2917-2923.