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Smoke tainted wine – what now?

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Abstract. Wines made from grapes exposed to smoke from bushfires that burned during the 2019/20 Australian grape growing season were subjected to various amelioration techniques, including: the addition of activated carbons, molecularly imprinted polymers (MIPs), or a proprietary adsorbent resin (either directly, or following fractionation by membrane filtration); spinning cone column (SCC) distillation; and transformation into spirit or vinegar, via fractional distillation or fermentation by acetic acid bacteria, respectively. The efficacy of treatments was determined by comparing volatile phenols (VPs) and their glycoconjugates, as chemical markers of smoke taint and changes in the intensity of fruit and smoke-related sensory attributes in wines, distillate, and vinegar samples. In brief: activated carbons can remove free and glycosylated VPs from smoke-tainted wines to some extent, without stripping desirable wine aroma and flavour. MIPs were also effective in removing VPs but not VP glycoconjugates. In contrast, adsorbent resin removed both free (<90%) and bound VPs (<30%). However, membrane filtration followed by resin treatment of the resulting permeate removed >95% of VPs. SCC distillation alone cannot remediate smoke taint, but smoke-related attributes were significantly diminished when 'stripped wine' was treated with activated carbon and blended with its corresponding condensate. Fractional distillation yielded 'heart' distillate fractions that were considered suitable for spirit production. Lastly, the potential for smoke-tainted wine to be transformed into vinegar was also demonstrated. The choice (and success) of each treatment ultimately depends on the extent to which wine is tainted, but the cost of harvesting and processing smoke-affected grapes should be considered when evaluating the economic return of remediation.

1. Introduction

Wildfire smoke poses a significant threat to vineyards and wine production worldwide. As climate change intensifies the frequency and severity of wildfires, the wine industry faces growing challenges related to smoke exposure. Smoke from bushfires contains volatile phenols (i.e., guaiacol, 4-methylguaiacol, *o*-, *m*-, and *p*-cresol, syringol, and 4-methylsyringol) that can be taken up by grapevine leaves and fruit, whereby they undergo glycosylation, to accumulate as volatile phenol (VP) glycoconjugates [1-3]. While VP glycosides do not impart smoky or ashy characters themselves, they can hydrolyse during fermentation, ageing, and even in the mouth during wine consumption, releasing aglycone VPs and potentially contributing to the sensory perception of smoke taint [4-7]. Volatile phenols and their glycoconjugates are measured by gas chromatography-mass spectrometry (GC-MS) and liquid chromatography-tandem mass spectrometry (LC-MS/MS), respectively [8, 9]. Recent research has identified a class of sulfur-containing compounds, thiophenols, that may also contribute to the undesirable ashy flavours associated with wines made from smoke-affected grapes [10], which adds another layer of complexity to the compositional analysis of smoke taint.

Several factors influence the extent to which grapes absorb smoke-related compounds, such as the timing and duration of smoke exposure [11, 12], the density of smoke [13-15] and grape variety [16-18]. Once smoke-affected grapes arrive in the winery, several processing and/or winemaking techniques can be employed to mitigate the sensory outcomes in finished wine [19]. Whole-bunch pressing has been shown to reduce the extraction of smoke taint compounds [20]. Similarly, implementing cold maceration and limiting skin contact has been shown to give significantly lower levels of guaiacol and 4-methylguaiacol in rosé-style wines [21]. Different yeast strains can influence the level of smoke taint in finished wine, with some strains enhancing smoke attributes and others diminishing them, however, no yeast strain has been found to be capable of eliminating the perception of smoke taint entirely [21-23]. The addition of oak chips and ellagic tannin can partially mask the perception of smoke taint by increasing wine complexity [21, 22], but winemakers are advised to avoid oak toast profiles that enhance smoke characters. Remediation trials involving the addition of fining agents, particularly activated carbon, have demonstrated their ability to remove smoke taint compounds [24, 25]. However, different activated carbon products vary in their performance and sensory outcome, and because they are not selective, they can remove desirable wine constituents, therefore small-scale tests are usually recommended [25, 26]. Reverse osmosis/nanofiltration used in combination with adsorbent materials can help target the removal of smoke taint compounds [27], and with new developments in membrane technologies, this approach warrants further investigation. Spinning cone column (SCC) distillation is used primarily to adjust wine alcohol levels [28, 29] but can remove undesirable flavours without negatively affecting the overall sensory profile of the wine [30]. It operates through a combination of centrifugal force and low temperature steam distillation, allowing winemakers to selectively capture condensate enriched with aroma and flavour compounds. The potential for SCC technology to be used to mitigate smoke taint in juice and wine was recently demonstrated [31], but again, remediation requires the use of adsorbent additives. While SCC distillation may not completely eliminate all smoke taint compounds, it can decrease their concentrations to levels where the taint is less noticeable. Fractional distillation was also evaluated as a means of transforming smoke-tainted wine into a saleable product. Industrial stills are capable of achieving high levels of fractionation which could aid the removal of smoke taint compounds when producing spirits [32, 33].

More recent trials have evaluated the use of molecularly imprinted polymers (MIPs), tailor-made to selectively bind smoke-derived volatile phenols, and ameliorate smoke taint during or after fermentation [34]. The MIPs were capable of removing VPs from white or red fermentations and wines (by up to 60%), but less than 10% of VP glycoconjugates were removed, thus further optimisation of the MIP template may be required [34]. Other novel polymeric adsorbents, such as resins which are routinely used to purify water [35] are also being evaluated for amelioration of smoke taint in wine. However, it is important to recognise that many of these techniques are more effective when used in combination, rather than individually.

In 2020, Australia experienced widespread wildfires, and vineyards in wine regions in South Australia, New South Wales, and Victoria were affected by fire and/or smoke, resulting in great economic losses [36]. In response, a series of trials were established to evaluate strategies for amelioration of smoke-tainted wines, including the addition of MIPs, adsorbent resin or activated carbon, individually, or following membrane filtration; spinning cone column (SCC) distillation, with and without the addition of activated carbon; fractional distillation to transform smoke tainted wine into spirit; and fermentation with acetic acid bacteria to transform smoke tainted wine into vinegar. Here, the outcomes of those trials are presented.

2. Materials and methods

2.1. Smoke tainted wines

Wines made from fruit harvested from vineyards (located in New South Wales and South Australia) that were exposed to smoke from bushfires that burned during the 2019/20 growing season were sourced from a commercial winery (Cassegrain Wines, Port Macquarie, NSW, Australia) or the University of Adelaide's winery (Waite Campus, Urrbrae, SA, Australia).

2.2. Remedial treatments

2.2.1. Addition of MIPs, adsorbent resin or activated carbon to smoke tainted wine

Smoke-tainted Chardonnay wine was treated (2L per treatment, in triplicate) with: a molecularly imprinted polymer (MIP) from amaea (New Zealand); a proprietary adsorbent resin from VAF Memstar (Australia); and activated carbons, Claril SMK from Enatris (Italy), FPS from Vason (Italy) or PC 1000 from Activated Carbon Technologies (Australia). MIPs (10 g/L) and resin (20 g/L) were added directly to the wine and removed after 2 h of contact; whereas activated carbons were dosed at 2 g/L, and wines were filtered after 24 h of contact.

2.2.2. Ultrafiltration and solid-phase adsorption treatment of smoke-tainted wine

A membrane filtration unit equipped with four ultrafiltration membranes with 5 KDa nominal molecular weight cut-off specifications (VAF Memstar, Nuriootpa, SA, Australia) was used to fractionate smoke-tainted Chardonnay wine (500 L per treatment, in duplicate) and the resulting permeate eluted through a column packed with adsorbent resin (60 L bed volume). Treated permeate and retentate were then returned to the feed tank. Treatments were performed to two distinct end-points, being generation (and subsequent elution through the resin column) of 1250 and 550 L of permeate. For comparison, the smoke-tainted Chardonnay wine (160 L, in duplicate) was also eluted through a column packed with MIPs (2 L bed volume).

2.2.3. Spinning cone column (SCC) distillation of smoke tainted wine

Part 1. Smoke-tainted Shiraz Sangiovese wine (~8000 L) was treated (by Australian Vintage Limited, Buronga Hill, NSW, Australia) using an industrial-scale SCC distillation system (SCC10000, Flavourtech, Griffith, NSW, Australia), as previously described (Puglisi et al. 2022), with samples of untreated wine, stripped wine and condensate collected after removal of 1%, 14% and 29% of the initial wine volume, for analysis.

Part 2. Smoke-tainted Chardonnay wine (75 L) was treated (by the NOLO research facility in the Hickinbotham Roseworthy Wine Science Laboratory, Urrbrae, SA, Australia) using a pilot-scale SCC distillation system (SCC100, Flavourtech). A 3% strip rate was applied (i.e., 3% of the initial wine volume was removed). The resulting stripped wine (50 L) was blended with condensate (3%, 600 mL, to restore the initial alcohol concentration), with and without the addition of activated carbon (2 g/L for 24 h, PC1000). For comparison, the smoke-tainted Chardonnay wine (5 L) was also treated directly with activated carbon (2 g/L for 24 h). The treatments were performed in triplicate.

2.2.4. Transformation of smoke-tainted wine into spirit via distillation

A smoke-tainted Pinot Noir wine (23 L) containing 14.8% alcohol by volume (abv), was distilled using a Grainfather G30 benchtop still without rectification. This yielded ~9 L of low wine containing 35.9% abv. The low wine was adjusted to 20% abv (by diluting with reverse osmosis water) and activated carbon (2 g/L for 48 h, FPS) was added. After racking and filtration to remove carbon lees, 20% abv low wine (~16.38 L) was fractionally distilled, using a 1.5” × 12” packed copper column. Ten sequential 400 mL distillate fractions (F1-F10) were collected with distillation temperatures of 85 °C (F1) to 99 °C (F10). Distillate alcohol levels decreased from 85.4% abv (F1) to 28.3 % abv (F10).

2.2.5. Transformation of smoke-tainted wines into vinegar

Smoke-tainted Chardonnay wine (2 L, 4 replicates) was diluted to 8.09% abv (by adding sterile MilliQ water); to remove excess SO_2 in wine, wines were aerated and treated with hydrogen peroxide. Acetic acid fermentation of the diluted wine was then conducted by inoculating with *Acetobacter pasteurianus* (AWRI B250) and *Gluconobacter oxydans* (AWRI B1905) (Australian Wine Research Institute, Australia). Fermentation (at ambient temperature, with stirring) occurred over 11 months and was deemed completed when the alcohol level decreased to <1 g/L.

2.3. Compositional analysis

The ethanol content of wines, distillates and vinegars (g/L) was measured with an Agilent 1100 high-performance liquid chromatograph equipped with a reflective index detector (Agilent Technologies, Forest Hill, Vic., Australia) following previously reported instrumental conditions [37]. VPs were measured with an Agilent 6890 gas chromatograph coupled to an Agilent 5973 mass spectrometer (Agilent Technologies), according to published stable isotope dilution analysis (SIDA) methods [9, 38]. VP glycoconjugates were quantified (as syringol gentiobioside equivalents) with an Agilent 1200 high-performance liquid chromatograph fitted with a 1290 binary pump and turbo V™ ion source (Framingham, MA, USA), and coupled to an AB SCIEX Triple Quad 4500 tandem mass spectrometer Agilent Technologies), again using published SIDA methods [9]. Sample preparation, method validation, and instrumental parameters for SIDA methods were as previously reported [9, 38-40].

2.4. Sensory analysis

Sensory analysis was conducted with approval by the Human Research Ethics Committee of the University of Adelaide (Ethics Approval No. H-2019-095, approved 6 June 2019) and informed consent was obtained from all sensory panellists.

The sensory profiles of untreated and treated wines were determined using the Rate-All-That-Apply (RATA) sensory analysis method [41]. Sensory panels comprised 50 regular wine consumers, typically aged 21-80 years. Panellists rated the intensity of 19 aroma, flavour, taste and mouthfeel attributes adapted from previous smoke taint studies [42, 43] using a 7-point scale (where 0 = ‘not perceived’, 1 = ‘extremely low’ and 7 = ‘extremely high’). The sensory sessions were held under controlled conditions in a purpose-built sensory laboratory at the University of Adelaide. Wines (30 mL) were served in 315 mL transparent glasses, labelled with randomly generated 4-digit codes, and presented monadically in a randomised order across participants. A ~1 min break was enforced between samples so that participants could refresh their palates with water and plain crackers, to avoid sensory fatigue. Data were collected with Red Jade software (Redwood Shores, CA, USA).

Sensory analysis of distillate: An expert panel comprising 12 spirit producers, and 3 academic staff and 1 student from the University of Adelaide (with extensive sensory analysis experience) evaluated samples obtained from distillation of smoke-tainted wine. Distillation fractions were diluted to 20% abv. Samples (30 mL) were served in 315 mL transparent glasses, labelled with randomly generated 4-digit codes. Panellists provided tasting notes and rated quality using a 100-point scale. Water and plain crackers were provided as palate cleaners.

Sensory analysis of vinegar: Vinegar samples were evaluated by a panel of 12 experienced sensory panellists comprising staff and students from the University of Adelaide, using the RATA method and a published tasting procedure for sensory analysis of vinegar [44]. Vinegar samples (30 mL) were served in shot glasses, labelled with randomly generated 4-digit codes, and presented monadically in a randomised order across participants. Again, panellists rated sensory attributes adapted from previous smoke taint studies [42], with the addition of overall pungent aroma and flavour. Water and crackers were provided as palate cleaners.

2.5. Data analysis

Chemical data were analysed by one-way ANOVA with mean comparisons by Tukey's Honestly Significant Difference (HSD) post-hoc test at $p < 0.05$, while sensory data were analysed by two-way ANOVA (using participants as a random factor and wines as a fixed factor) with mean comparisons performed by Fischer's Least Significant Difference (LSD) test at $p<0.05$. All analyses, including Principal Component Analysis (PCA), were performed using XLSTAT (version 2022, Lumivero, New York, USA).

3. Results and Discussion

Following the 2019/20 bushfires in New South Wales and South Australia, a series of trials were undertaken to evaluate the efficacy of different strategies for amelioration of smoke-tainted wines. The chemical and sensory outcomes of these trials are highlighted below.

3.1. Addition of MIPs, adsorbent resin and activated carbon to smoke-tainted wine

Activated carbon is widely used for its adsorptive properties, making it effective at removing undesirable compounds from liquids, but its efficacy depends on the type of activated carbon used, the dosage rate and the sample matrix [25, 26]. In a preliminary trial, 11 activated carbons were evaluated (data not shown), from which the best performing carbons were identified, being Claril SMK, FPS and PC 1000.

Subsequent addition of the three activated carbons to a smoke-tainted Chardonnay wine demonstrated their ability to remove VPs. Guaiacol was removed by 10-15%, syringol by 30% and cresols by 29% (Figure 1A), but they did not bind significant amounts of VP glycoconjugates (Figure 1B). This was not unexpected, as a previous study reported that activated carbons remove free VPs more readily than VP glycoconjugates, from both grape juice and wine [26]. By comparison, the addition of MIPs or adsorbent resin to the same wine achieved the removal of 39% and 28% of guaiacol, 28% and 27% of syringol, and 50% and 22% of total cresols, respectively (Figure 1A). The capacity of MIPs to remove VPs during and after fermentation of Semillon juice and Merlot must have recently been demonstrated [34]. The addition of MIPs after yeast inoculation gave the best outcome; the absorption of VPs by MIPs was 10% higher than when MIPs were added after fermentation, likely reflecting competition with other constituents in the finished wine. Despite their ability to adsorb VPs, no adsorption of glycoconjugates by MIPs was observed [34]. In this respect, the adsorbent resin was superior, with small, but significant quantities of guaiacol, syringol and cresol glycosides removed (Figure 1B).

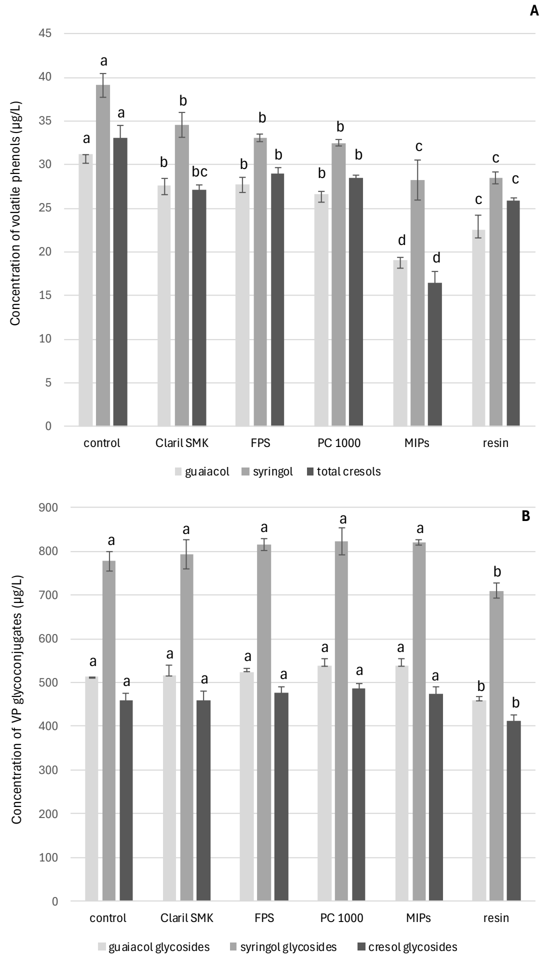

Figure 1. Concentration of (A) free and (B) glycosylated volatile phenols in smoke-tainted Chardonnay wine before (control) and after treatment with activated carbon, MIPs or adsorbent resin. Values are means (± standard error) of three replicates; letters indicate statistically significant differences ($p \leq 0.05$ one-way ANOVA).

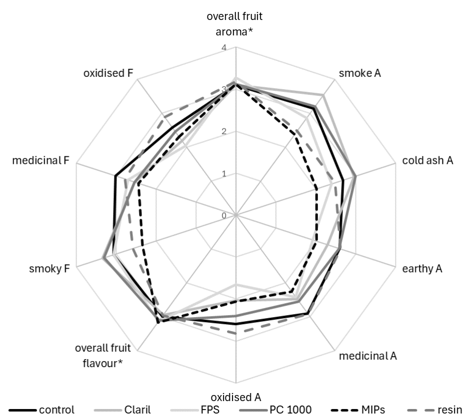

Figure 2. Sensory profiles of smoke-tainted Chardonnay wine before (control) and after treatment with activated carbons, MIPs and adsorbent resin. Values are mean intensity ratings from 3 wine replicates evaluated by 50 sensory panellists. * denotes attributes for which ratings were not significantly different. A = aroma; F = flavour; AT = aftertaste.

The effects of the above treatments on wine sensory profiles are presented in Figure 2. Wines treated with FPS were perceived as less smoky and ashy than wines treated with the other two activated carbons, Claril SMK and PC 1000. Importantly, however, none of the activated carbon treatments had any detrimental effects on overall fruit aroma and flavour. In this trial, the best outcome was achieved by MIP addition; the intensity of smoky and ashy attributes in MIP-treated wine was significantly lower than for any other treatment. Wines treated with resin were also significantly improved, with less apparent smoke-related sensory characters, but some oxidative characters were perceived.

3.2. Ultrafiltration and solid-phase adsorption treatment of smoke-tainted wine

An earlier study demonstrated the combined use of reverse osmosis and solid phase adsorption to mitigate the perception of smoke taint in wine [27]. The removal of VPs improved the sensory properties of wine, however, VP glycoconjugates were not passed through the RO membrane, so remained in treated wines, and are now understood to contribute to the sensory impact of smoke taint [6]. As such, the partitioning of free and glycosylated VPs by membranes of various nominal molecular weight cut-off (MWCO) specifications (i.e., from 20 kDa to 200 Da) was investigated. VPs were observed (at comparable concentrations) in the permeate and retentate generated with ultrafiltration (UF) and nanofiltration (NF) membranes, but permeation of VP glycoconjugates progressively decreased with the use of tighter membranes, such that VP glycoconjugates were concentrated in retentate (data not shown). To enable the removal of both free and glycosylated VPs, a smoke-tainted Chardonnay wine was fractionated (on a semi-commercial scale) using a UF membrane (with a 5 kDa MWCO) and the resulting permeate eluted through a column packed with absorbent resin.

The resin adsorbed up to 93% of VPs and 60% of VP glycoconjugates when 1250 L of UF-derived permeate was treated (i.e., 'resin 1') and up to 75% of VPs and 20% of VP glycoconjugates when 550 L of UF-derived permeate was treated (i.e., 'resin 2'). These results suggest the resin was saturated by VP glycoconjugates sooner than occurs for VPs (Figure 3). It should be noted that the first treatment ('resin 1') was deemed by the winemakers involved in the study to have overtreated the wine; the process was therefore repeated on a second volume of wine, to an earlier end-point (i.e., 'resin 2'). Elution of the wine through a column packed with MIPs resulted in 20% and 30% removal of guaiacol and cresols, respectively (Figure 3A), whereas VP glycoconjugates were not removed (Figure 3B), in agreement with previous trials (Figure 1).

The composition of untreated and treated wines reflected their sensory properties (Figure 4). Membrane filtration and resin treatments significantly diminished the perception of smoke-related sensory attributes. However, the wine corresponding to resin treatment of 1250 L of permeate (i.e., 'resin 1') also had diminished fruit aroma and flavour, and thus was considered to have been overtreated. Where resin treatment was scaled back to 550 L of permeate, greater fruit expression was retained. Despite the removal of some VPs, wine treated with MIPs still exhibited some smoky and ashy characters, suggesting further optimisation of the column treatment is required, i.e., increased MIP dose and/or slower flow rates to achieve greater VP removal, and thus smoke taint remediation.

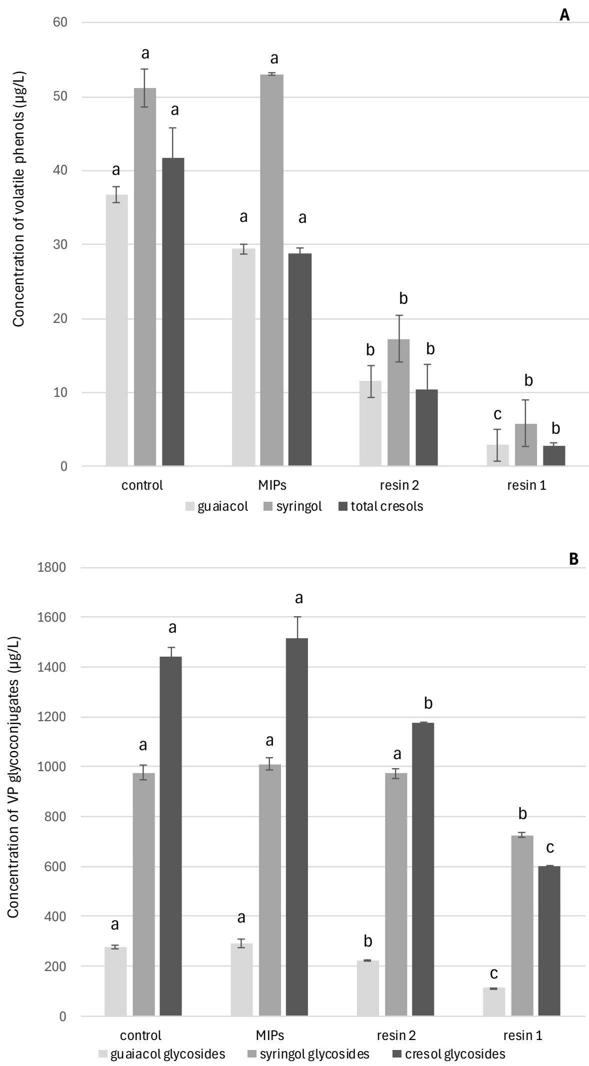

Figure 3. Concentration of (A) free and (B) glycosylated volatile phenols in smoke-tainted Chardonnay wine before (control) and after the addition of MIPs or combined membrane filtration and adsorbent resin treatment. Values are means (± standard error) of two replicates; letters indicate statistically significant differences ($p \leq 0.05$ one-way ANOVA).

Whilst the results from this study demonstrate the removal of smoke taint compounds from wine, and thus, the potential for mitigation of smoke taint, findings also highlighted several limitations associated with the remediation processes employed. Firstly, MIPs were designed to adsorb VPs, and the larger VP glycoconjugates do not seemingly fit into the VP binding site, so are not removed. In this study, the MIPs became saturated by VPs, so future studies could optimise regeneration (which was beyond the scope of this study). Saturation and carryover of VP glycoconjugates were also observed during remediation with the adsorbent resin, so optimisation of resin regeneration (or the use of tighter membranes that preclude permeation of VP glycoconjugates) may also be needed.

Figure 4. Sensory profiles of smoke-tainted Chardonnay wine before (control) and after the addition of MIPs or combined membrane filtration and adsorbent resin treatment. Values are mean intensity ratings from two wine replicates presented to 50 sensory panellists. A = aroma; F = flavour; AT = aftertaste.

3.3. Spinning cone column (SCC) distillation of smoke-tainted wine

Part 1. SCC distillation is a separation process typically used to dealcoholise wines, but it has also been evaluated as a strategy for removing VPs from smoke-tainted wine [31]. Analysis of the stripped wine and condensate fractions collected after removal of 1%, 14% and 29% of the initial wine volume demonstrated progressive removal of alcohol (Table 1), as expected while the concentration of anthocyanins (from 136 to 170 mg/L), pigmented polymers (from 1.5 to 2.1 mg/L), total phenolics (from 48 to 65 au) and acids (titratable acidity, from 6.3 to 8.5 g/L) increased [31]. Whereas VPs were expected to be found in condensate, they were largely retained in stripped wine (Table 1). This was attributed to their higher boiling points (e.g., 205 °C for guaiacol and 260 °C for syringol) relative to other wine volatile compounds. As expected, VP glycoconjugates were not detected in condensate but were instead concentrated in stripped wines (Table 1). The mild distillation conditions (i.e., low temperature) did not seem to result in hydrolysis of VP glycoconjugates.

Fruit expression was highest in the initial untreated wine (Figure 5), and as wine was progressively fractionated, stripped wines were found to become less fruity and more smoky, because alcohol and desirable wine volatile compounds were removed [28] and smoke-taint compounds were largely concentrated in the stripped wine [31, 45]. Stripped wines also became more acidic and salty, as organic acids and salts were concentrated. On its own, SCC distillation does not provide an effective strategy for the remediation of smoke-tainted wine, however, the retention of both VPs and their glycoconjugates in stripped wines presents an opportunity for SCC to be instead used in combination with an adsorbent material. For example, desirable wine volatile compounds can be captured in condensate, allowing the addition of activated carbon to stripped wine to remove smoke taint compounds (without the inherent loss of desirable aroma compounds). Treated stripped wine could then be blended with condensate to obtain treated wine, which should retain fruit expression. To test this theory, a subsequent trial involving both SCC distillation and activated carbon treatments was performed.

Table 1. Composition of smoke-tainted Shiraz Sangiovese wine before (control) and after spinning cone column distillation (following 1%, 14% and 29% stripping).

	control	stripped wine		
		1%	14%	29%
Alcool (%)	15.1	14.5	7.9	0.3
pH	3.69	3.68	3.59	3.50
TA (g/L)	6.3	6.4	7.2	8.5
WCD (au)	6.5	6.6	7.7	9.2
A (mg/L)	136	136	148	170
PP (au)	1.5	1.5	1.8	2.1
P (au)	48	49	55	65
VPs (µg/L)	101	100 (*30*)	104 (*40*)	103 (*115*)
VP gly (µg/L)	250	232 (*nd*)	279 (*nd*)	338 (*nd*)

WCD = wine colour density; A = Anthocyanins; PP = pigmented polymers; P = total phenolics. Volatile phenols (VPs) = sum of guaiacol, 4-methylguaiacol, *o*-, *m*-, and *p*-cresols, phenol, syringol and 4-methylsyringol; Total glyconjugates are the sum of guaiacol rutinoside; 4-methylguaiacol rutinoside; phenol rutinoside; cresol rutinoside; syringol gentiobioside and 4-methylsyringol gentiobioside. Values in italics denotes the corresponding condensate concentration. nd = not detected.

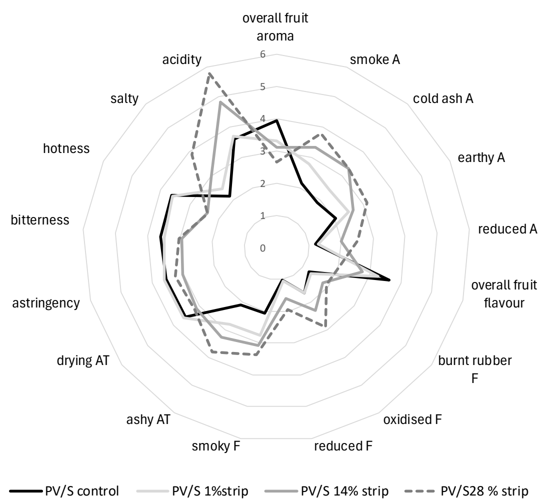

Figure 5. Sensory profiles of smoke-tainted Shiraz Sangiovese wine before (control) and after spinning cone column distillation (i.e., stripped wines sampled after 1%, 14% and 28% stripping). Values are mean intensity ratings from one wine replicate presented to 50 sensory panellists. A = aroma; F = flavour; AT = aftertaste.

Part 2. To evaluate the use of SCC distillation in combination with activated carbon addition, a follow up experiment was performed. Smoke-tainted Chardonnay wine was subjected to several treatments: (i) direct addition of PC 1000 activated carbon (C); fractionation by SCC distillation (3% strip) with condensate and stripped wine immediately blended (SCC); and fractionation by

SCC distillation (3% strip), followed by addition of PC 1000 activated carbon to stripped wine, before blending of treated stripped wine and condensate (SCC+C). The alcohol content, pH, titratable acidity and total phenolics of control and treated wines were measured to determine any impacts on wine quality (Table 2). Minimal (0.2–0.4%) decreases in alcohol were observed and these differences were not anticipated to have any effect on wine sensory profiles. Minimal changes in pH (<0.02) and TA (< 0.1g/L) were also observed but again were not expected to impact wines in a perceivable way. The most notable shifts in the basic composition of wines were phenolic measurements. Wines treated with activated carbon had significantly decreased phenolic measurements (i.e., decreases of 4.0 to 4.4 au), which should be considered when determining activated carbon dose rates so as not to compromise the desired style of wine. Importantly, significant decreases in VPs were achieved following treatments involving the addition of activated carbon (i.e., VPs decreased from 227 to 46 and 39 µg/L, for Carbon and SCC+C treatments, respectively), demonstrating the efficacy of treatments in removing smoke-derived volatile phenols. Only ~7% decreases in VP glycoconjugates were observed, but these changes were not statistically significant (Table 2).

Table 2. Composition of smoke-tainted Chardonnay wine before (control) and after spinning cone column (SCC) distillation (3% strip) and activated carbon treatments (applied individually and in combination).

	Control	SCC	C	SCC+C
Alcohol (%)	13.8a	13.6c	13.6c	13.4d
pH	3.49	3.47	3.49	3.45
TA (g/L)	6.1a	5.9b	5.9b	5.8b
P (au)	6.3a	6.0b	2.3c	1.9d
VPs (µg/L)	227a	223a	46b	39b
VP gly (µg/L)	3579	3764	3556	3327

C = activated carbon. Values are means of three replicates; letters indicate statistically significant differences ($p \leq 0.05$ one-way ANOVA). P = total phenolics; Volatile phenols (VPs) = sum of guaiacol, 4-methylguaiacol, *o*-, *m*-, and *p*-cresols, phenol, syringol and 4-methylsyringol; VP glycoconjugates (VP gly) = sum of 18 different VP glycosides (each measured as syringol gentiobioside equivalents).

From a sensory perspective, the control and SCC-treated wines both exhibited obvious smoke characters, i.e. smoke aromas and flavours, and an ashy aftertaste. However, the wines treated with activated carbon exhibited much less intense smoke characters. The wine pre-treated with SCC before activated carbon addition showed increased fruit expression compared with wine treated via direct addition of activated carbon. This finding demonstrates the value in remediating smoke-tainted wine using a combined SCC distillation and solid phase adsorbent treatment.

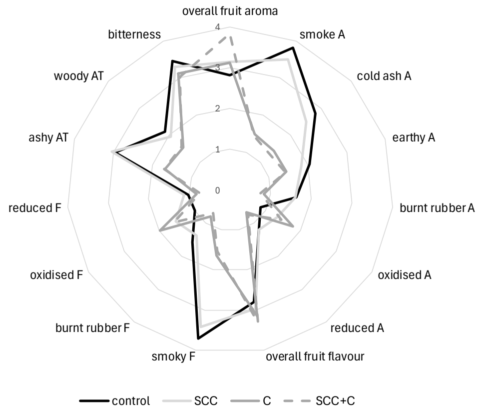

Figure 6. Sensory profiles of smoke-tainted Chardonnay wine before (control) and after spinning cone column distillation (3% strip) and activated carbon treatments (applied individually and in combination). Values are mean intensity ratings from two wine replicates presented to 50 sensory panellists. A = aroma; F = flavour; AT = aftertaste.

3.4. Transformation of smoke-tainted wine into spirit via distillation

A heavily smoke-tainted Pinot Noir wine with elevated VP concentrations (Figure 7) was first batch distilled to give a low wine which was then treated with activated carbon (FPS), resulting in only a small amount of guaiacol (3 µg/L) being detected. Fractional distillation was then performed, and 10 fractions were collected; no VPs were detected in the first two fractions and < 3 µg/L of VPs was observed in fractions F3 to F8. The last 2 fractions, being the ‘tails’, had much higher VP levels: with F10 comprising 14 µg/L of guaiacol, 2 µg/L of syringol and 5 µg/L of cresols. These ‘tail’ fractions are comparatively lower in alcohol content (Figure 7) and thus would usually be discarded or redistilled in typical spirit production. The alcohol content decreased from 85.0% abv in F1 to 28% abv in F10, or 0.34 mg/L (F1) to 0.11 mg/L (F10) of absolute ethanol. Distillate fractions were combined as ‘heads’ (F1+F2), ‘hearts 1’ (F3+F4), ‘hearts 2’ (F5+F6), ‘hearts 3’ (F7+F8) and ‘tails’ (F9+F10) and diluted (to 20% abv) for sensory evaluation by an expert panel. The ‘heads’ fraction was described as clean, fresh, and simple with caramel and fruity notes; ‘hearts 1’ exhibited floral, caramel, and butterscotch notes, with some smoke notes; ‘hearts 2’ had sweet spice, fruity, savoury and malt notes; while ‘hearts 3’ was described as having honey, caramel, cherry/plum and dry fruit, with hints of woody and oaky notes. Finally, the ‘tails’ fraction was defined by woodfire, ashy and savoury notes with bruised apple and honey/caramel characters. Sensory results suggest distillation of smoke-tainted wine can achieve distillate fractions that are suitable for spirit production; where some residual smoke characters remain, these fractions could potentially be further developed in barrels for use in brandy, whisky, or cognac.

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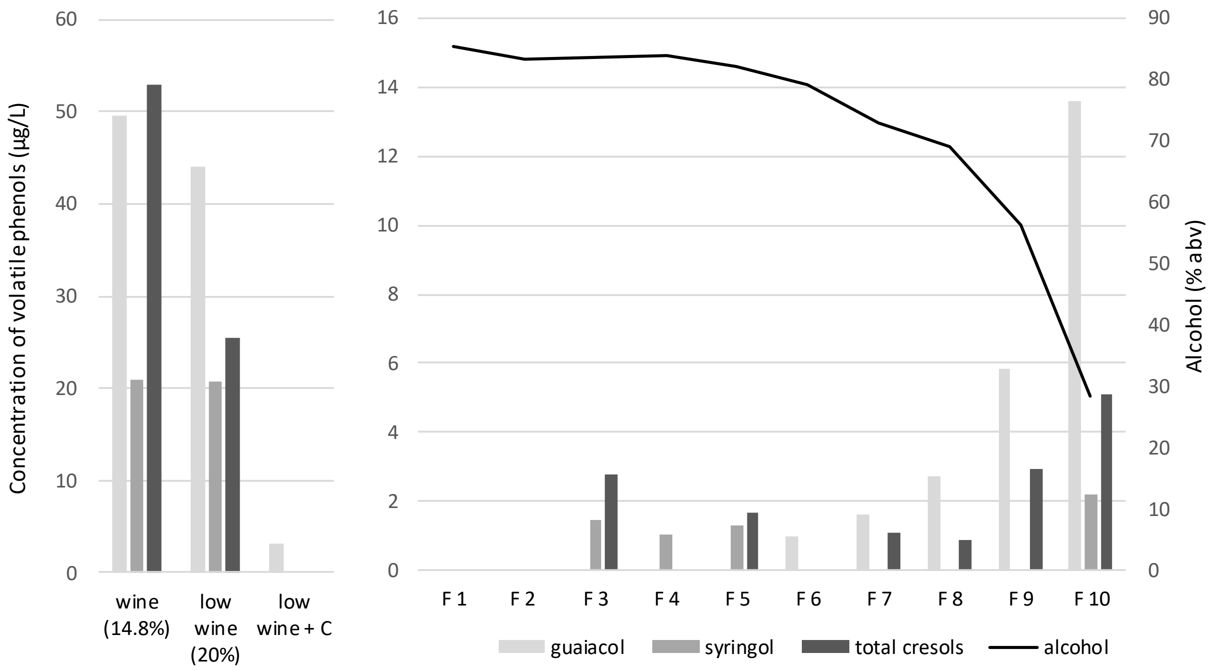

Figure 7. The concentration of volatile phenols in smoke-tainted Pinot Noir wine (14.8% abv), low wine after batch distillation (20% abv) and activated carbon treatment (C), and distillate fractions (F1-F10) obtained after fractional distillation.

3.5. Transformation of smoke-tainted wine into vinegar

Following appropriate sugar and alcohol adjustments, a smoke-tainted Chardonnay wine was inoculated with acetic acid bacteria (*Acetobacter pasteurianus* + *Gluconobacter oxydans*) and fermented for 11 months, until alcohol decreased to <1 g/L due to conversion to acetic acid (~70 g/L), i.e., wine was transformed into wine vinegar. Interestingly, the concentration of most VPs increased by ~70%, (and by ~120% for syringol) (Figure 8). This corresponded to a decrease in VP glycoconjugates (of > 60%) (Figure 8) presumably due to enzyme and/or acid hydrolysis.

Sensory evaluation of vinegar samples revealed that the overall aroma and flavour of the wine persisted (and was often rated higher in vinegar than in wine), while the intensity of smoke-related sensory attributes diminished, but pungent aromas and flavours were dominant (their mean ratings were 4.4 to 4.7 on a 7-point scale) (Figure 9). The vinegar exhibited complexity, even though it was not aged in barrels or with oak chips, suggesting smoke-tainted wines can be transformed into saleable vinegar. Further research is needed to understand to what extent the pungent aroma and flavour of vinegar can mask smoke-related sensory characteristics and to further optimise the fermentation process, but results to date are promising.

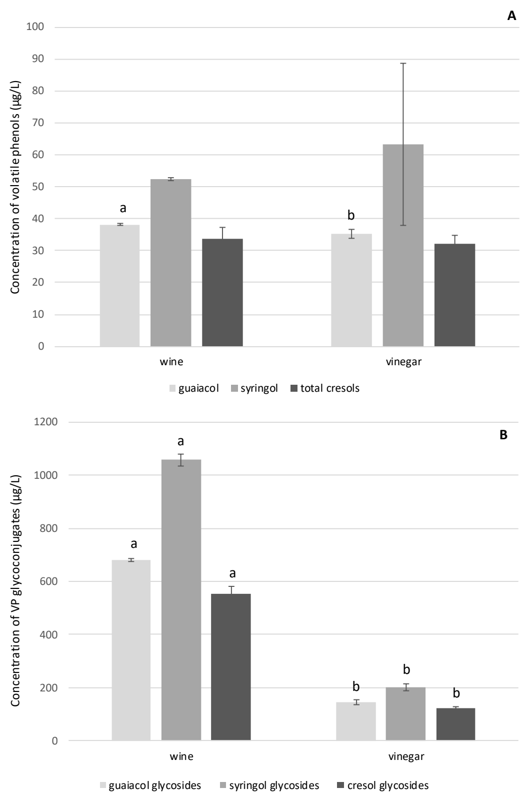

Figure 8. The concentration of (A) free and (B) glycosylated volatile phenols in smoke-tainted Chardonnay wine and the vinegar obtained after fermentation with acetic acid bacteria.

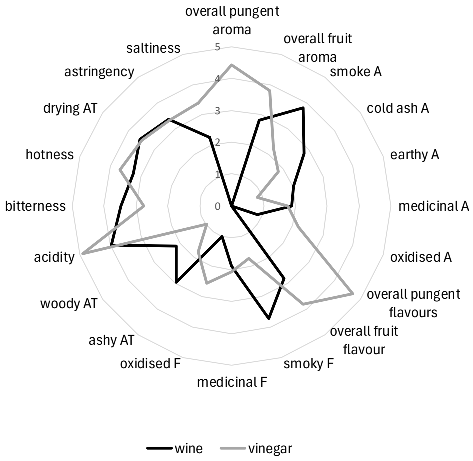

Figure 9. Sensory profiles of smoke-tainted Chardonnay wine and the vinegar obtained after fermentation with acetic acid bacteria. Values are the mean ratings from four vinegar replicates presented to 12 sensory panellists and two wine samples presented to 50 sensory panellists. A = aroma; F = flavour; AT = aftertaste.

4. Conclusion

The studies reported in this paper highlight the potential, and the shortcomings of several novel strategies for the amelioration of smoke-tainted wine. The use of adsorbent materials, not only activated carbon, but MIPs and resin, were found to be effective at removing VPs (but not so much their glycoconjugates), especially when coupled with other separation technologies, i.e., membrane filtration or spinning cone column distillation. Where the severity of smoke taint in wine is high, these amelioration methods may not be able to adequately treat wine or may inadvertently strip too much of the desirable wine colour, aroma and flavour during treatment, such that the overall quality of wine cannot be improved. In these instances, it may be more appropriate to consider other uses for smoke-tainted wines, and distillation and spirit production offer one alternate pathway. Preliminary results suggest smoke-tainted wine can also be transformed into wine vinegar, but the volumes at which vinegar is consumed (relative to wine and spirits) will likely see this avenue saturate fairly quickly. With all treatments, the cost of remediation needs to be carefully considered, and it should be recognised that the most economical solution to managing smoke-affected grapes may well still be to discard fruit, especially heavily tainted fruit. Nevertheless, results from the studies presented here should give winemakers greater insight into the efficacy of amelioration strategies that are available to them.

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