

Novel use of activated carbon fabric to mitigate smoke taint in grapes and wine

K.L. WILKINSON^{1,2} , R. RISTIC¹ , C. SZETO^{1,2} , D.L. CAPONE^{1,2} , L. YU^{3,4}  and D. LOSIC^{3,4} 

¹ School of Agriculture, Food and Wine, Waite Research Institute, The University of Adelaide, Glen Osmond, SA 5064, Australia; ² The Australian Research Council Training Centre for Innovative Wine Production, Glen Osmond, SA 5064, Australia; ³ School of Chemical Engineering and Advanced Materials, The University of Adelaide, Adelaide, SA 5005, Australia; ⁴ The Australian Research Council Research Hub for Graphene Enabled Industry Transformation, The University of Adelaide, Adelaide, SA 5005, Australia

Corresponding author: Professor Kerry L. Wilkinson, email kerry.wilkinson@adelaide.edu.au

Abstract

Background and Aims: Grapegrowers and winemakers, around the world, are searching for strategies to mitigate the compositional and sensory consequences of grapevine exposure to smoke from wildfires. This study evaluated the use of activated carbon fabrics as protective coverings to mitigate the uptake of smoke-derived volatile phenols by grapes, and accordingly, the intensity of smoky, ashy characters in wine.

Methods and Results: Smoke was applied to Mataro grapes, with and without individual bunches being enclosed in bags made from three activated carbon fabrics (felt, light cloth and heavy cloth). Wine made from smoke-exposed grapes had an elevated concentration of volatile phenols, but the composition of wines made from grapes protected by activated carbon fabric was comparable to that of the Control wine; the difference in concentration of guaiacol, *o*- and *m*-cresol and/or syringol was only 1 µg/L. Wine made from smoke-exposed grapes had diminished fruit and prominent smoke characters, whereas the sensory profile of the wines corresponding to activated carbon fabric treatments could not be differentiated from that of the Control wine. Analysis by GC/MS of the activated carbon fabrics following repeated smoke exposure confirmed their adsorption of smoke volatiles.

Conclusions: The activated carbon fabrics successfully protected Mataro grapes and wine from being tainted by smoke exposure.

Significance of the Study: This study demonstrates a promising new technology for overcoming smoke taint, an issue of major concern for grape and wine producers worldwide.

Keywords: GC/MS, rate-all-that-apply, smoke taint, volatile phenols

Introduction

Climate change has become a major challenge for grape and wine producers around the world (van Leeuwen and Darriet 2016). Increased temperature during the growing season affects fruit composition and ripening (Kliwer and Torres 1972, Coombe 1987), while water stress (because of decreased rainfall and/or warmer temperature) impairs vine growth and photosynthesis, reduces yield and affects fruit composition and quality (Hsiao 1973, Mendez-Costabel et al. 2014, Gambetta 2016). The frequency of extreme weather events is also increasing (Intergovernmental Panel on Climate Change 2014), including the occurrence of bushfires in or near prominent wine regions (Larsen 2009, Mirabelli-Montan et al. 2021). In the last few years, wine regions in Australia, New Zealand, the USA, Canada, Chile and South Africa have experienced major fires (Walpole 2020, Mirabelli-Montan et al. 2021, Romano 2021), with devastating consequences. Where fires burn through vineyards, the radiant heat can scorch the leaves, fruit, trunks and buds of grapevines (Whiting 2012, Collins et al. 2014), and depending on the intensity of the fire, heat-damaged vines may require remedial pruning to aid recovery (Collins et al. 2014), or

removal and replanting (Whiting 2012). Vineyards are more likely, however, to be affected by smoke, which can taint grapes and therefore wine (Krstic et al. 2015, Ristic et al. 2016, Mirabelli-Montan et al. 2021), depending on the density of smoke (Szeto et al. 2020) and the timing and duration of smoke exposure (Kennison et al. 2009, 2011). Wine is deemed to be smoke tainted when it displays unpalatable smoky, burnt, medicinal, burnt rubber aromas and flavours, and a drying, ashy aftertaste (Kennison et al. 2008, Parker et al. 2012, Ristic et al. 2016, Szeto et al. 2020). In 2020, an estimated 4% of the Australian winegrape crop was discarded because of smoke taint (Walpole 2020), while the California harvest was reportedly down nearly 14% (Romano 2021). The resulting revenue losses are estimated to be in the hundreds of millions of dollars (Walpole 2020, Romano 2021).

Smoke taint occurs because volatile organic compounds present in smoke are absorbed by grapes when smoke from bushfires or prescribed burns drifts into vineyards (Krstic et al. 2015, Mirabelli-Montan et al. 2021), including volatile phenols derived from thermal degradation of lignin in plants (Wittkowski et al. 1992), which are thought to be responsible for the characteristic aromas of smoke (Kennison et al. 2008, Parker et al. 2012, Ristic et al. 2016, Szeto et al. 2020). Following exposure of grapevines to smoke, volatile phenols accumulate in grapes (and leaves) in glycoconjugate

*The authors declare no competing interests.

doi: 10.1111/ajgw.12548

© 2022 The Authors. *Australian Journal of Grape and Wine Research* published by John Wiley & Sons Australia, Ltd on behalf of Australian Society of Viticulture and Oenology Inc.

This is an open access article under the terms of the Creative Commons Attribution-NonCommercial-NoDerivs License, which permits use and distribution in any medium, provided the original work is properly cited, the use is non-commercial and no modifications or adaptations are made.

precursor forms (Hayasaka et al. 2010, Ristic et al. 2016, Noestheden et al. 2018a, van der Hulst et al. 2019, Szeto et al. 2020); their glycosylation by endogenous enzymes mitigates toxicity and the potential for cellular damage (Song et al. 2018). The presence of these volatile phenols and their glycoconjugates is measured, respectively, by GC/MS and liquid chromatography/MS methods (Pollnitz et al. 2004, Dungey et al. 2011, Hayasaka et al. 2013, Noestheden et al. 2018b), and used as compositional markers of smoke taint in grapes and wine.

Various strategies for mitigating the chemical and sensory effects of grapevine exposure to smoke have been evaluated, both preventative measures applied in the vineyard (Ristic et al. 2013, Favell et al. 2019, 2021, van der Hulst et al. 2019, Mirabelli-Montan et al. 2021) and remedial measures applied in the winery (Fudge et al. 2011, 2012, Ristic et al. 2011, Culbert et al. 2021, Mirabelli-Montan et al. 2021, Modesti et al. 2021a,b). Among them, the removal of smoke-derived volatile compounds (and their glycosides) using adsorbent materials such as activated carbon (AC), either from juice (Culbert et al. 2021) or wine (Fudge et al. 2011, 2012), remains the most effective amelioration strategies. Nevertheless, preventing the initial uptake of smoke volatiles by grapes would be preferable to strategies that ameliorate smoke-tainted grapes or wine, because of the potential loss of desirable wine constituents. Several recent studies involving the application of protective sprays to grapevine fruit and foliage (Favell et al. 2019, 2021, van der Hulst et al. 2019) and postharvest ozonation of smoke-affected grapes (Modesti et al. 2021a,b) report promising results, that is a significantly reduced concentration of smoke taint marker compounds in grapes or wine and/or less intense smoke-related sensory attributes in wine. The efficacy of these methods, however, is still under investigation, because divergent results were obtained when field trials were repeated in different growing seasons and/or with different cultivars. The need for vineyard-based strategies that mitigate the risk of smoke taint in grapes therefore remains an imperative.

Activated carbon is a popular adsorbent used by various industries for environmental remediation, including solvent recovery, wastewater treatment and air and water purification (Chen 2017). It is produced in different forms (powders, granules, pellets) from waste organic materials, such as wood, coal and coconut shells via thermochemical processes (carbonisation and activation) (Foo and Hameed 2009, Chen 2017). Activated carbon can also be mixed and extruded with fibre polymers to produce AC fabrics, including felt and cloth, which are used for air filtration and in personal protective clothing (Chen 2017). The high surface area, pore volume and adsorption capacity of AC are well established (Chen 2017), along with the adsorption affinity of different contaminants for AC, including phenolic compounds (Dabrowski et al. 2005). The current study sought to exploit these physicochemical properties and validate the potential for AC fabrics to be used as protective coverings to mitigate the risk of smoke taint in grapes and wine, that is their efficacy for preventing the uptake of smoke-derived volatile phenols by grapes, and accordingly, the perception of smoky, ashy characters in wine.

Materials and methods

Mitigation trials

Mataro grapes (50 kg) were hand-harvested at commercial maturity (TSS 26°Brix) from a vineyard at The University of

Adelaide's Waite Campus (in Urrbrae, SA, Australia, 34°58'02.5"S, 138°38'01.0"E). Grape bunches were randomly allocated to five replicate fruit parcels (~50 bunches, 10 kg each). One parcel was set aside as a Control (i.e. fruit was not exposed to smoke), while four parcels were exposed to smoke (for 15 min) using a purpose-built smoke chamber (Figure S1), with smoke generated by burning barley straw (100 g) in a commercial fire box smoker. Immediately prior to smoke exposure, bunches from three of the fruit parcels were enclosed in bags made from three different AC fabrics (Nature Technologies, Hangzhou, China); a 1 mm felt and two 0.4 mm cloths of light and heavy weave, with a nominal surface area of 1000, 900 and 1200 m²/g, respectively. The ~20 × 25 cm bags were made (in-house) by folding rectangular pieces of AC fabric in half and stitching the two adjacent sides together. Bags were then positioned over individual bunches and the fabric at the top of the bag gathered tightly around the rachis stem and held in place with a bulldog clip that was then used to suspend the bunch on a rack inside the smoke chamber. Following smoke exposure, grapes (30 berries from three replicate bunches per treatment, chosen randomly) were sampled for GC/MS analysis. The remaining bunches from each fruit parcel were then randomly allocated to three winemaking replicates and fermented according to small-scale winemaking protocols (Holt et al. 2006).

An additional parcel of Mataro grapes (80 bunches) was hand-harvested for use in a separate trial, during which the different AC fabric bags (in duplicate) were repeatedly exposed to smoke (10 × 15 min smoke applications, as above). A fresh bunch of grapes was placed in each bag before each smoke application; two uncovered bunches were also exposed to smoke during each application. Strips of each AC fabric (~2 × 12 cm) were also subjected to repeated smoke exposure. Following each smoke application, grapes (30 berries per bunch per treatment, chosen randomly) were again sampled for GC/MS analysis.

Chemical analysis of grapes, wine and AC fabrics

Volatile phenols were quantified in grapes and wine using an Agilent 6890N GC coupled to a 5973N mass selective detector (Agilent Technologies, Palo Alto, CA, USA), and with established stable isotope dilution analysis methods (Pollnitz et al. 2004, Hayasaka et al. 2013). Basic wine chemistry parameters (pH, TA, volatile acidity, alcohol) were measured by Fourier transform infrared (FTIR) spectroscopy using a Foss Wine Scan (Mulgrave, Vic., Australia). Wine colour and total phenolic compounds were measured using the modified Somers method (Mercurio et al. 2007).

Control and smoke-exposed strips of each AC fabric were analysed using different physicochemical techniques. Morphology and elemental composition were examined by scanning electron microscopy (SEM) coupled with energy dispersive X-ray (EDX) analysis (FEI Quanta 450 FEG, USA; Ultim Max, Oxford Instruments, Abingdon, England). Chemical structures were determined by FTIR (Nicolet 6700, Thermo Fisher Scientific, Melbourne, Vic., Australia), with measurements of crystalline forms collected in the range of $2\theta = 10\text{--}80^\circ$ (scanning at $10^\circ/\text{min}$). Raman spectra (LabRAM HR Evolution, Horiba Jvon Yvon Technology, Kyoto, Japan) were acquired between 1200 and 1500 cm⁻¹, using a 532 nm laser, to analyse vibrational characteristics.

Headspace solid-phase microextraction (SPME) GC/MS analysis was performed on an Agilent 6890N GC coupled to a 5973N mass selective detector (Agilent Technologies, Forest

Hill, Vic., Australia). The SPME fibre [2 cm Supelco DVB/CAR/PDMS (Merck, Bayswater, Vic., Australia)] was exposed into a vial containing the pre-cut fabric (~1 cm²) for 30 min at 50°C, with agitation (500 rpm), using a Gerstel MPS2 autosampler (Lasersan Australasia, Tanunda, SA, Australia). The fibre was then desorbed in the GC inlet at 240°C for 15 min in pulsed splitless mode with a pulsed pressure of 310 kPa for 0.5 min. The GC oven had a 60 m J&W DB-Wax UI column (0.25 mm ID, 0.25 µm DF, Agilent Technologies) and was started at 40°C for 5 min, then increased to 240°C at 4°C/min and maintained at this temperature for 10 min, with a column flow of 1.5 mL/min; ultra-high purity helium was used as the carrier gas (BOC, Mile End, SA, Australia). The auxiliary temperature was held at 240°C throughout the run and the MS scan range was *m/z* 35–350 for the duration of the run. A series of alkanes (C7–C40) were injected at the end of the run to determine linear retention indices of each of the compounds detected.

Sensory analysis of wine

The wine replicates from each treatment were assessed by three sensory experts from The University of Adelaide to ensure there were no faults or obvious differences among replicates. Replicates were then blended and their sensory profiles determined using the Rate-All-That-Apply method (Danner et al. 2018) and a panel comprising staff and students from The University of Adelaide and The Australian Wine Research Institute and regular wine consumers (*n* = 51, 16 male and 35 female, aged between 23 and 67 years). Panellists rated the intensity of 19 aroma, flavour, taste and mouthfeel attributes adapted from previous smoke taint studies (Ristic et al. 2016, Szeto et al. 2020) using a 7-point scale (where 0 = 'not perceived', 1 = 'extremely low' and 7 = 'extremely high'). Sensory evaluation was undertaken under controlled conditions in a purpose-built sensory laboratory, with wine samples (30 mL) presented monadically in covered, three-digit-coded 215 mL stemmed glasses, using a randomised order across panellists. Panellists rested for at least 1 min between samples, with water and plain crackers provided as palate cleansers. Data were acquired with RedJade software (Redwood Shores, CA, USA). Sensory panellists gave informed consent before participating in the study, which was approved by the Human

Research Ethics Committee of The University of Adelaide (ethics approval no. H-2019-095).

Statistical analysis

Chemical and sensory data were analysed by ANOVA using GenStat (19th edition, VSN International, Hemel Hempsted, England) and XLSTAT (version 2018.1.1, Addinsoft, New York, NY, USA), respectively. Mean comparisons were performed by Fisher's least significant difference multiple comparison test at *P* = 0.05.

Results and discussion

Smoke and AC fabric treatments influence wine composition and sensory profiles

The composition and sensory profile of wines made from Control and smoke-exposed Mataro grapes were compared to evaluate to what extent enclosing grape bunches in AC fabric bags mitigated the effects of smoke exposure, with volatile phenols measured as chemical markers of smoke taint (Table 1). Guaiacol, *o*-cresol and syringol were detected in the Control wine at 2, 1 and 6 µg/L, respectively, in agreement with previous studies that have reported volatile phenols (and their glycosides) as natural constituents of grapes from some cultivars (Hayasaka et al. 2013, Ristic et al. 2013, 2016, Noestheden et al. 2018b, Szeto et al. 2020). In contrast, the wine made from smoke-affected grapes had an increased concentration of each of the volatile phenols measured, but guaiacol, *o*- and *m*-cresol and syringol in particular, which were detected at a concentration of 16, 11, 9 and 21 µg/L, respectively. The concentration of volatile phenols in wines corresponding to the AC fabric treatments was similar to that of the Control wine, and only an increase of 1 µg/L in guaiacol, *o*- and *m*-cresol and/or syringol was observed (Table 1). These results indicate the AC fabrics successfully protected the grape bunches from exposure to smoke.

Whereas the sensory profile of the Control wine was largely characterised by fruit aromas and flavours, exposing Mataro grapes to smoke resulted in wine with diminished fruit intensity and prominent smoke, cold ash, medicinal and burnt rubber aromas, smoky flavour and an ashy after-taste (Figure 1). The wines corresponding to the AC fabric treatments, however, exhibited a sensory profile that was comparable to that of the Control wine (Figure 1, Table S1),

Table 1. Composition of wines made from Control and smoke-affected Mataro grapes, with and without bunches being enclosed in activated carbon fabric bags during smoke exposure.

	Control	Smoke	AC fabric (felt)	AC fabric (light)	AC fabric (heavy)	<i>P</i> -value
Guaiacol (µg/L)	2 b	16 a	3 b	3 b	2 b	0.001
4-Methylguaiacol (µg/L)	n.d.	4	n.d.	n.d.	n.d.	—
<i>o</i> -Cresol (µg/L)	1 b	11 a	2 b	1 b	2 b	0.001
<i>m</i> -Cresol (µg/L)	n.d.	9 a	1 b	1 b	1 b	0.001
<i>p</i> -Cresol (µg/L)	n.d.	2	n.d.	n.d.	n.d.	—
Syringol (µg/L)	6 b	21 a	7 b	7 b	6 b	0.001
4-Methylsyringol (µg/L)	n.d.	4	n.d.	n.d.	n.d.	—
pH	3.3	3.3	3.3	3.3	3.3	n.s.
TA (as g/L of tartaric acid)	8.3	8.9	8.9	8.7	9.0	n.s.
Alcohol (% v/v)	14.8 a	14.8 a	14.1 b	14.4 b	14.2 b	0.005
Wine colour density (a.u.)	9.0 a	9.1 a	7.2 b	7.7 b	7.6 b	0.003
Wine hue	0.58	0.58	0.58	0.59	0.59	n.s.
Anthocyanins (mg/L)	350 a	356 a	295 b	311 b	316 b	<0.001
Total pigments (a.u.)	20.0 a	20.2 a	16.7 b	17.6 b	17.8 b	<0.001
Total phenolics (a.u.)	36.7 a	37.3 a	30.7 c	32.0 bc	33.3 b	<0.001

Values are means of three replicates (*n* = 3); n.d., not detected. Different letters (within rows) indicate statistical significance (*P* = 0.05); n.s., not significant; AC, activated carbon.

irrespective of the fabric type (i.e. felt vs light or heavy cloth). These results showed good agreement with compositional data (Table 1) and further validated the potential for the AC fabrics to be used as protective coverings to mitigate smoke exposure, and thus, the risk of smoke taint. Previous studies evaluated partial defoliation of grapevines before or after smoke exposure (Ristic et al. 2013), the application of protective sprays to grapevine foliage (Favell et al. 2019, 2021, van der Hulst et al. 2019), in-canopy misting during smoke exposure (Szeto et al. 2020), washing grapes after smoke exposure (Noestheden et al. 2018b) and postharvest ozonation of smoke-affected grapes (Modesti et al. 2021a,b) as potential strategies for mitigating smoke taint in grapes, with limited to moderate success. None of the mitigation strategies employed to date have shown the efficacy afforded by the AC fabric bags in the current study.

Following the mitigation trial, fibres from the AC fabrics were observed adhering to grape bunches and were therefore retained during fermentation. Subsequent analysis of basic wine chemical parameters [pH, TA, alcohol, colour and phenolic compounds (Table 1)] identified decolourisation as a potential shortcoming of the AC fabrics. The propensity for AC to adsorb anthocyanins from red wine is well established (Singleton and Draper 1962), and along with stripping of aroma volatiles (Waterhouse et al. 2016), is a key consideration when AC is used as a fining agent during wine production, including the use of AC for the amelioration of smoke-tainted wine (Fudge et al. 2012, Culbert et al. 2021). In the current study, a small but statistically significant loss of colour (measured as wine colour density, anthocyanins and total pigments) was observed in wines corresponding to AC fabric treatments, relative to the Control (Table 1), and attributed to adsorption of anthocyanins and pigmented polymers by AC fibre residues. As a consequence, the concentration of total phenolics of these wines was also significantly lower than that of the Control and smoke-affected wines. The AC fabric treatments apparently resulted in

wines with a significantly lower alcohol concentration (Table 1), but this was attributed to a difference in the TSS of the fruit parcels, which varied by up to 0.8°Brix (Table S2). All wines were fermented to dryness, that is residual sugar concentration was <1 g/L. Importantly, the observed difference in alcohol concentration ($\leq 0.7\%$ v/v) was not expected to impact wine sensory evaluation. No significant difference in wine pH or TA was observed (Table 1).

Activated carbon fabrics protect grapes during repeated smoke treatments

In a subsequent experiment, the AC fabric bags were subjected to ten successive smoke treatments to evaluate how many times they could be re-used before they became saturated with smoke. The profile of the volatile phenols of Mataro grapes was compared following alternate smoke treatments (Figure 2, Table S3), again with and without bunches being enclosed in the different AC fabric bags during smoke exposure. None of the volatile phenols measured were detected in Control grapes (Table S3), but an elevated concentration of volatile phenols (up to 53 $\mu\text{g}/\text{kg}$) was observed in smoke-affected grapes without protective coverings; guaiacol, *o*- and *m*-cresol and syringol were again the most abundant volatile phenols, in agreement with wine compositional data (Table 1). Some variation in the concentration of grape volatile phenols was observed between smoke treatments, for example the concentration of guaiacol and syringol ranged more than twofold, that is from 20 to 53 and 21 to 46 $\mu\text{g}/\text{kg}$, respectively (Figure 2). Statistical analysis confirmed that smoke-affected grapes from the eighth successive smoke treatment had a significantly higher concentration of volatile phenols ($P < 0.0001$; Figure 2, Table S3), indicative of increased smoke exposure. Given the duration of each smoke treatment was standardised, at 15 min per treatment, the observed variation in grape volatile phenols likely reflected differences in

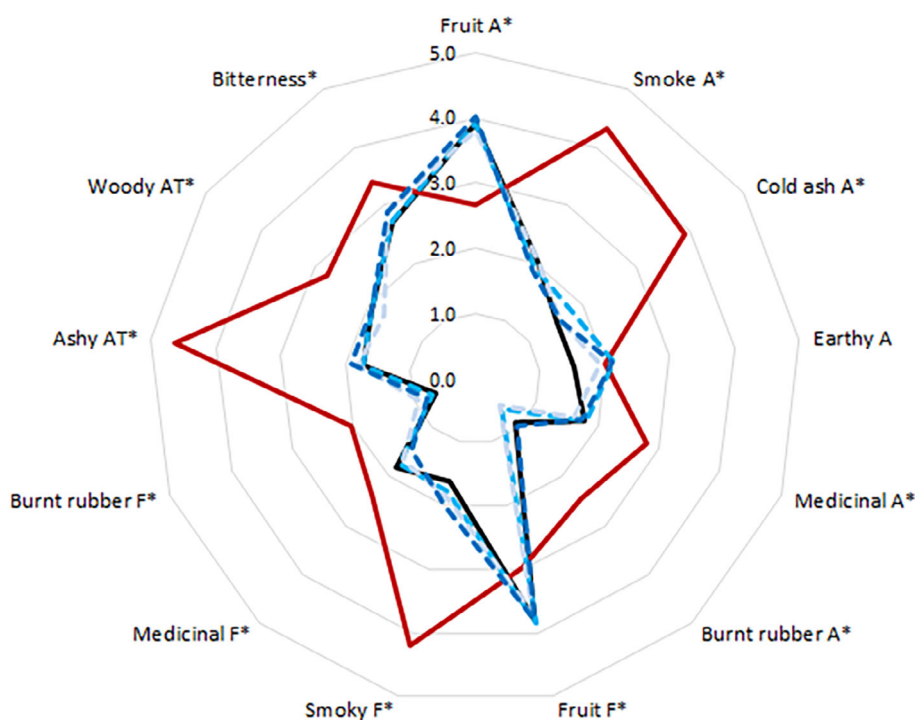


Figure 1. Sensory profiles of wines made from Control and smoke-affected Mataro grapes, with and without bunches being enclosed in activated carbon (AC) fabric bags during smoke exposure. Control (—); smoke (—); AC fabric (felt) (---); AC fabric (light) (···); AC fabric heavy (-·-·); A, aroma; F, flavour; AT, aftertaste; *Statistical significance ($P = 0.05$).

the density of replicate smoke treatments, because of incomplete combustion of fuel and/or loss of smoke to the surrounding environment. Nevertheless, grapes enclosed in AC fabric bags during the eighth smoke treatment did not absorb a significant concentration of smoke-derived volatile phenols, despite the apparent increased level of smoke exposure for that treatment. Guaiacol was detected at the same concentration (2 µg/kg) as early smoke treatments, while 1 µg/kg of syringol was detected in one grape sample (corresponding to the lighter AC fabric bag treatment). An increased concentration of guaiacol, *o*- and *m*-cresol and syringol was detected only in grapes enclosed in protective coverings during the sixth and tenth smoke treatments. In the case of the sixth

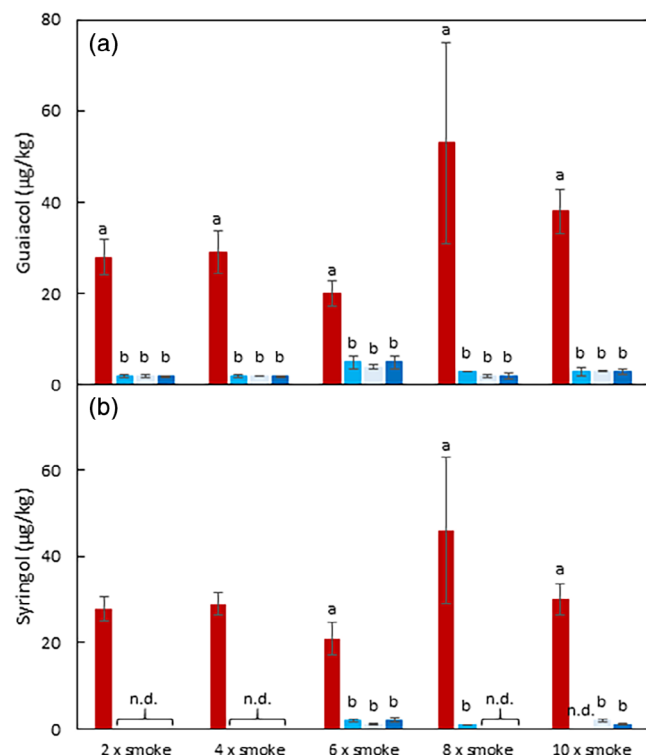


Figure 2. Concentration of (a) guaiacol and (b) syringol in smoke-affected Mataro grapes, with and without bunches being enclosed in activated carbon (AC) fabric bags, during repeated smoke applications. Values are means of two replicates ($n = 2$); n.d., not detected. Different letters indicate statistical significance within alternate smoke treatments ($P = 0.05$). Neither guaiacol nor syringol were detected in Control Mataro grapes. Smoke (■); AC fabric (felt) (■); AC fabric (light) (■); AC fabric heavy (■).

smoke treatment, this was attributed to some small tears in the AC fabrics, which may have compromised the protection afforded by the bags. The tears were patched with adhesive tape prior to further smoke treatments, after which they effectively mitigated the uptake of volatile phenols by grapes once more. In the case of the tenth smoke treatment, the presence of smoke-derived volatile phenols in grapes suggested either permeation of smoke because of further tearing of the AC fabrics or reduced efficacy of protective coverings, possibly because of saturation with smoke. Even during these treatments, however, the AC fabric bags still mitigated the uptake of volatile phenols by grapes by 80–90% (Figure 2, Table S3). These results suggest the AC fabrics adsorbed smoke particles, thereby preventing contamination of grapes. Adsorption of smoke might account for the 5.6, 1.9 and 3.7% increase in mass observed for the strips of each AC fabric (the 1 mm felt and two 0.4 mm cloths of light and heavy weave, respectively), following exposure to ten successive smoke treatments.

The AC fabric strips were subjected to a range of physicochemical analyses to evaluate compositional changes because of repeated smoke exposure. Surface characterisation, however, by SEM, EDX analysis, X-ray diffraction (XRD) analysis, FTIR and Raman spectroscopy did not provide conclusive evidence of smoke adsorption by the AC fabrics. Scanning electron microscopy confirmed the surface of fabric fibres was coated with AC (Figure S2), while EDX analysis confirmed carbon (86.42–93.90%) and oxygen (5.8–11.06%) as the main chemical elements of the AC fabrics (Table S4), in agreement with previous research (Medellín-Castillo et al. 2021). Small changes in elemental composition occurred with repeated smoke exposure, but an increase in oxygen content for the heavier AC fabric (from 5.80 to 8.74%) was the only statistically significant compositional change detected. Spectral analyses (Figure S3) indicated the surface chemistry of samples was preserved during smoke exposure, that is there was no evidence of new functional groups or chemical binding. Irrespective of smoke exposure, the XRD patterns of AC fabric fibres each had two broad diffraction peaks at $2\theta = 24^\circ$ and 43° (Figure S3b), consistent with graphitic carbon (Xu et al. 2014), and corresponding to the (002) reflection and the superposition of the (100) and (101) reflections of graphitic-type lattice, respectively (Xu et al. 2014). The Raman spectra of the AC fabrics (Figure S3c) were similar, with the G band indicative of phonon excitation of sp^2 -hybridised carbon atoms (Xu et al. 2014) located at 1580 cm^{-1} and the D band associated with disordered

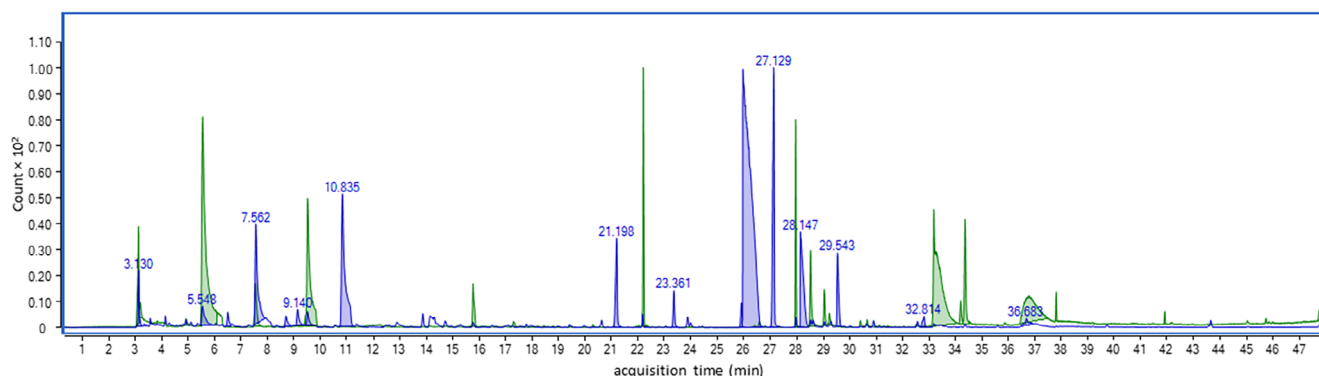


Figure 3. Chromatograms from HS-SPME-GC/MS analysis of Control (—) and smoke-exposed (—) activated carbon felt; data are autoscaled.

carbon or defects in the graphite structure (Kim et al. 2011, Xu et al. 2014) located at 1320 cm^{-1} . The absence of a compositional differences between Control and smoke-exposed AC fabrics by Raman spectroscopy was consistent with FTIR and XRD results, and likely reflects the inadequacy of the surface characterisation methods to detect adsorption of smoke particles within pores of the AC coatings of the respective fabrics.

The Control and smoke-exposed AC fabrics were subsequently analysed by head space SPME-GC/MS and a difference in the composition was observed between samples as a consequence of smoke exposure (Figures 3,S4). Volatile compounds were identified by comparison with a mass spectral library and literature retention indices (Table S5). With the exception of 4-methyl-2-pentyl acetate, all of the compounds detected in the headspace of smoke-exposed AC fabrics were volatiles previously reported as constituents of wood smoke (Maga 1988) and/or emitted from combustion of different biomass (Hatch et al. 2014), including several markers of smoke taint, guaiacol, *o*-cresol and phenol (Table S5). In contrast, the headspace of Control AC fabrics largely comprised column- or septa-derived siloxanes (data not shown) or trace concentration of volatiles that were detected in the smoke-exposed AC fabrics. These results confirm the adsorption of smoke volatiles by the AC fabrics, which prevented grape bunches from being tainted by smoke.

Conclusion

This study validates an innovative approach to mitigating smoke taint in grapes and wine by demonstrating that AC fabric can be used as a protective covering to prevent grapes from exposure to smoke. These results are highly promising and provide ‘proof-of-concept’, nevertheless, several shortcomings need to be overcome before this strategy can be implemented by industry for use in commercial vineyards. First, the AC fabrics studied were prone to tearing and had to be handled carefully to avoid damage that would likely have compromised their efficacy. Second, the labour cost associated with applying AC fabric bags to individual grape bunches on a commercial scale is prohibitively expensive and likely viable only for ultra-premium grapes. Incorporation of AC into more durable and functional materials that can be readily deployed in vineyards should be explored. One plausible option might be AC-based netting that could be applied to the grapevine fruit zone. This approach would be more cost-effective and would offer dual protection from birds, but any impact on photosynthesis, fruit composition and disease pressure because of shading and/or diminished air-flow would need to be evaluated. The potential for AC fibre residues to remain in either the vineyard or in finished wine, and cause health and/or environmental issues, also needs to be evaluated. Nevertheless, of all the vineyard-based mitigation strategies evaluated to date, the use of AC fabric described herein seemingly offers the most promising opportunity for overcoming the issue of smoke taint, thereby addressing an issue of major concern to grape and wine producers around the world.

Acknowledgements

The authors thank Sir Peter Michael and Robert Fiore for their informed discussion and valuable feedback and the AWRI's Commercial Services Laboratory for wine analysis. This research was conducted by the Australian Research Council Training Centre for Innovative Wine Production (www.arcwinecentre.org.au), which is funded as part of the

ARC's Industrial Transformation Research Program (project number ICI70100008). Renata Ristic was funded by the Australian Government via a Cooperative Research Centre Project grant (project number CRCPIX000220). Dusan Losic and Le Yu acknowledge support from the ARC's Research Hub for Graphene Enabled Industry Transformation (project number IH150100003). Open access publishing facilitated by The University of Adelaide, as part of the Wiley - The University of Adelaide agreement via the Council of Australian University Librarians.

References

- Chen, J.Y. (2017) Introduction. Chen, J.Y., ed. Activated carbon fiber and textiles (Woodhead Publishing: Oxford, England) pp. 3–20.
- Collins, C., Gao, H. and Wilkinson, K.L. (2014) An observational study in the recovery of grapevines (*Vitis vinifera* L.) following a bushfire. *American Journal of Enology and Viticulture* **65**, 285–292.
- Coombe, B.G. (1987) Influence of temperature on composition and quality of grapes. *Acta Horticulturae* **206**, 25–35.
- Culbert, J.A., Jiang, W., Bilogrevic, E., Likos, D., Francis, I.L., Krstic, M.P. and Herderich, M.J. (2021) Compositional changes in smoke-affected grape juice as a consequence of activated carbon treatment and the impact on phenolic compounds and smoke flavor in wine. *Journal of Agricultural and Food Chemistry* **69**, 10246–10259.
- Danner, L., Crump, A.M., Croker, A., Gambetta, J.M., Johnson, T.E. and Bastian, S.E.P. (2018) Comparison of rate-all-that-apply and descriptive analysis for the sensory profiling of wine. *American Journal of Enology and Viticulture* **69**, 12–21.
- Dabrowski, A., Podkościelny, P., Hubicki, Z. and Barczak, M. (2005) Adsorption of phenolic compounds by activated carbon—a critical review. *Chemosphere* **58**, 1049–1070.
- Dungey, K.A., Hayasaka, Y. and Wilkinson, K.L. (2011) Quantitative analysis of glycoconjugate precursors of guaiacol in smoke-affected grapes using liquid chromatography–tandem mass spectrometry based stable isotope dilution analysis. *Food Chemistry* **126**, 801–806.
- Favell, J.W., Noestheden, M., Lyons, S.M. and Zandberg, W.F. (2019) Development and evaluation of a vineyard-based strategy to mitigate smoke-taint in wine grapes. *Journal of Agricultural and Food Chemistry* **67**, 14137–14142.
- Favell, J.W., Fordwour, O.B., Morgan, S.C., Zigg, I. and Zandberg, W. (2021) Large-scale reassessment of in-vineyard smoke-taint grapevine protection strategies and the development of predictive off-vine models. *Molecules* **26**, 4311.
- Foo, K.Y. and Hameed, B.H. (2009) A short review of activated carbon assisted electrosorption process: an overview, current stage and future prospects. *Journal of Hazardous Materials* **170**, 552–559.
- Fudge, A.L., Ristic, R., Wollan, D. and Wilkinson, K.L. (2011) Amelioration of smoke taint in wine by reverse osmosis and solid phase adsorption. *Australian Journal of Grape and Wine Research* **17**, S41–S48.
- Fudge, A.L., Schietecatte, M., Ristic, R., Hayasaka, Y. and Wilkinson, K.L. (2012) Amelioration of smoke taint in wine by treatment with commercial fining agents. *Australian Journal of Grape and Wine Research* **18**, 302–307.
- Gambetta, G. (2016) Water stress and grape physiology in the context of global climate change. *Journal of Wine Economics* **11**, 168–180.
- Hatch, L.E., Luo, W., Pankow, J.F., Yokelson, R.J., Stockwell, C.E. and Barsanti, K.C. (2014) Identification and quantification of gaseous organic compounds emitted from biomass burning using two-dimensional gas chromatograph/time-of-flight mass spectrometry. *Atmospheric Chemistry and Physics Discussions* **14**, 23237–23307.
- Hayasaka, Y., Baldock, G.A., Parker, M., Pardon, K.H., Black, C.A., Herderich, M.J. and Jeffery, D.W. (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, G.A., Pardon, K.H., Black, C.A., Jeffery, D.W. and Herderich, M.J. (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.
- Holt, H.E., Iland, P.G. and Ristic, R. (2006) A method for mini-lot fermentation for use in research and commercial viticultural and winemaking trial. *The Australian & New Zealand Grapegrower & Winemaker* (**509a**), 74–81.
- Hsiao, T. (1973) Plant responses to water stress. *Annual Review of Plant Physiology* **24**, 519–570.
- Intergovernmental Panel on Climate Change (2014) Impacts, adaptation, and vulnerability. <http://ipcc.ch/report/ar5/wg2/> [accessed 22/10/2021].
- Kennison, K.R., Gibberd, M.R., Pollnitz, A.P. and Wilkinson, K.L. (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, K.R., Wilkinson, K.L., Pollnitz, A.P., Williams, H.G. and Gibberd, M.R. (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, K.R., Wilkinson, K.L., Pollnitz, A.P., Williams, H.G. and Gibberd, M.R. (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.
- Kim, H., Cho, J., Jang, S.-Y. and Song, Y.W. (2011) Deformation-immunized optical deposition of graphene for ultrafast pulsed lasers. *Applied Physics Letters* **98**, 021104.
- Kliwer, M. and Torres, R. (1972) Effect of controlled day and night temperatures on grape coloration. *American Journal of Enology and Viticulture* **23**, 71–77.
- Krstic, M.P., Johnson, D.L. and Herderich, M.J. (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.
- Larsen, J. (2009) Wildfires by region: observations and future prospects. http://www.earth-policy.org/images/uploads/graphs_tables/fire.htm [accessed 22/10/2021].
- Maga, J.A. (1988) Smoke in food processing (CRC Press: Boca Raton, FL, USA) pp. 61–68.
- Mendez-Costabel, M.P., Wilkinson, K.L., Bastian, S.E.P., Jordans, C., McCarthy, M., Ford, C.M. and Dokoozlian, N. (2014) Effect of winter rainfall on yield components and fruit green aromas of *Vitis vinifera* L. cv. Merlot in California. *Australian Journal of Grape and Wine Research* **20**, 100–110.
- Medellín-Castillo, N.A., Ocampo-Pérez, R., Forgioony, A., Labrada-Delgado, G.J., Zárate-Guzmán, A.L., Cruz-Briano, S.A. and Flores-Ramírez, R. (2021) Insight into equilibrium and adsorption rate of phenol on activated carbon pellets derived from cigarette butts. *Processes* **9**, 934.
- Mercurio, M.D., Damberg, R.G., Herderich, M.J. and Smith, P.A. (2007) High throughput analysis of red wine and grape phenolics—adaptation and validation of methyl cellulose precipitable tannin assay and modified Somers color assay to a rapid 96 well plate format. *Journal of Agricultural and Food Chemistry* **55**, 4651–4657.
- Mirabelli-Montan, Y.A., Marangon, M., Graça, A., Mayr Marangon, C.M. and Wilkinson, K.L. (2021) Techniques for mitigating the effects of smoke taint while maintaining quality in wine production: a review. *Molecules* **26**, 1672.
- Modesti, M., Szeto, C., Ristic, R., Jiang, W., Culbert, J., Bindon, K., Catelli, C., Mencarelli, F., Tonutti, P. and Wilkinson, K. (2021a) Potential mitigation of smoke taint in wines by post-harvest ozone treatment of grapes. *Molecules* **26**, 1798.
- Modesti, M., Szeto, C.S., Ristic, R., Jiang, W., Culbert, J., Catelli, C., Mencarelli, F., Tonutti, P. and Wilkinson, K. (2021b) Amelioration of smoke taint in Cabernet Sauvignon wine via post-harvest ozonation of grapes. *Beverages* **7**, 44.
- Noestheden, M., Dennis, E.G. and Zandberg, W. (2018a) Quantitating volatile phenols in Cabernet Franc berries and wine after on-vine exposure to smoke from a simulated forest fire. *Journal of Agricultural and Food Chemistry* **66**, 695–703.
- Noestheden, M., Dennis, E.G., Romero-Montalvo, E., DiLabio, G.A. and Zandberg, W.F. (2018b) Detailed characterization of glycosylated sensory-active volatile phenols in smoke-exposed grapes and wine. *Food Chemistry* **259**, 147–156.
- Parker, M., Osidacz, P., Baldock, G.A., Hayasaka, Y., Black, C.A., Pardon, K.H., Jeffery, D.W., Geue, J.P., Herderich, M.J. and Francis, I.L. (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, A.P., Pardon, K.H., Sykes, M. and Sefton, M.A. (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., Pinchbeck, K.A., Fudge, A.L., Hayasaka, Y. and Wilkinson, K.L. (2013) Effect of leaf removal and grapevine smoke exposure on colour, chemical composition and sensory properties of Chardonnay wines. *Australian Journal of Grape and Wine Research* **19**, 230–237.
- Ristic, R., Osidacz, P., Pinchbeck, K.A., Hayasaka, Y., Fudge, A.L. and Wilkinson, K.L. (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, A.L., Pinchbeck, K.A., De Bei, R., Fuentes, S., Hayasaka, Y., Tyerman, S.D. and Wilkinson, K.L. (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.
- Romano, A. (2021) The impact of 2020's wildfires. *Wine Spectator*. <https://www.winespectator.com/articles/the-impact-of-2020-s-wildfires-063021> [accessed 22/10/2021].
- Shimoda, M. and Shibamoto, T. (1990) Isolation and identification of headspace volatiles from brewed coffee with an on-column GC/MS method. *Journal of Agricultural and Food Chemistry* **38**, 802–804.
- Singleton, V.L. and Draper, D.E. (1962) Adsorbents and wines. I. Selection of activated charcoals for treatment of wine. *American Journal of Enology and Viticulture* **13**, 114–125.
- Song, C., Härtl, K., McGraphery, K., Hoffman, T. and Schwab, W. (2018) Attractive but toxic: emerging roles of glycosidically bound volatiles and glycosyltransferases involved in their formation. *Molecular Plant* **11**, 1225–1236.
- Szeto, C., Ristic, R., Capone, D., Puglisi, C., Pagay, V., Culbert, J., Jiang, W., Herderich, M., Tuke, J. and Wilkinson, K.L. (2020) Uptake and glycosylation of smoke-derived volatile phenols by Cabernet Sauvignon grapes and their subsequent fate during winemaking. *Molecules* **25**, 3720.
- van der Hulst, L., Munguia, P., Culbert, J.A., Ford, C.M., Burton, R. A. and Wilkinson, K.L. (2019) Accumulation of volatile phenol glycoconjugates in grapes following grapevine exposure to smoke and potential mitigation of smoke taint by foliar application of kaolin. *Planta* **249**, 941–952.
- van Leeuwen, C. and Darriet, P. (2016) The impact of climate change on viticulture and wine quality. *Journal of Wine Economics* **11**, 150–167.
- Walpole, M. (2020) The financial impact of 2020 summer bushfire smoke on the wine regions of north east Victoria. <https://www.tafo.com.au/farmsmart/r-d/143-bushfire-2020.html> [accessed 22/10/2021].
- Waterhouse, A.L., Sacks, G.L. and Jeffery, D.W. (2016) *Understanding wine chemistry* (Wiley: Chichester, England) p. 342.
- Whiting, J. (2012) Recovery of grapevines from fire damage. *Australian & New Zealand Grapegrower & Winemaker* (**580**), 25, 27–31.
- Wittkowski, R., Ruther, J., Drinda, H. and Rafiei-Taghanaki, F. (1992) Formation of smoke flavor compounds by thermal lignin degradation. Teranishi, R., Takeoka, G.R. and Güntert, M., eds. *Flavor precursors: thermal and enzymatic conversions* (American Chemical Society: Washington, DC, USA) pp. 232–243.
- Xu, J., Zhang, Q.G., Tan, Y., Tan, W., Zhu, L. and Jiang, L. (2014) Preparing two-dimensional microporous carbon from pistachio nutshell with high areal capacitance as supercapacitor materials. *Scientific Reports* **4**, 5545.

Manuscript received: 21 October 2021

Revised manuscript received: 19 December 2021

Accepted: 25 December 2021

Supporting information

Additional supporting information may be found in the online version of this article at the publisher's website: <http://onlinelibrary.wiley.com/doi/10.1111/ajgw.12548/abstract>.

Table S1. Mean intensity ratings for sensory attributes of wines made from Control and smoke-affected Mataro grapes, with and without bunches being enclosed in activated carbon fabric bags during smoke exposure.

Table S2. Total soluble solids of juice from Control and smoke-affected Mataro grapes, with and without bunches being enclosed in activated carbon fabric bags during smoke exposure.

Table S3. Effect of repeated smoke applications on the concentration of volatile phenols in Control and smoke-affected Mataro grapes, with and without bunches being enclosed in activated carbon fabric bags.

Table S4. Elemental analysis of activated carbon fabrics before and after repeated smoke applications.

Table S5. Volatile compounds detected by HS-SPME-GC/MS analysis in smoke-exposed activated carbon fabrics, and in Control samples in trace quantities.

Figure S1. (a) Schematic diagram of smoke chamber and fire box smoker, and photographs taken (b) before and (c) during smoke exposure of Mataro grapes, with and without bunches being enclosed in activated carbon (AC) fabric bags.

Figure S2. Micrographs obtained by scanning electron microscopy (SEM) for (a,b) Control activated carbon (AC) felt, (c,d) smoke-affected AC felt, (e,f) Control light AC cloth, (g,h) smoke-affected light AC cloth, (i,j) Control heavy AC cloth and (k,l) smoke-affected heavy AC cloth at low resolution (a,c,e,g,i,k) and at (b,d,f,h,j,l) high resolution for the area highlighted.

Figure S3. (a–c) FTIR, (d–f) XRD and (g–i) Raman spectra of Control and smoke-exposed (a,d,g) activated carbon (AC) fabric (felt), (b,e,h) AC fabric (light) and (c,f,i) AC fabric (heavy).

Figure S4. Chromatograms from HS-SPME-GC/MS analysis of (a) Control and (b) smoke-exposed activated carbon (AC) fabrics; AC fabric (light) (—); AC fabric (heavy) (—); AC fabric (felt) (—).