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## Amelioration of Smoke Derived Taint in Wine by Membrane Filtration and Solid Phase Adsorption: Final Report to the Grape and Wine Research & Development Corporation January 30, 2009

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## **Background and Methodology:**

Worldwide, vineyards have been increasingly exposed to smoke from wildfires as climate-induced weather changes intensify temperatures, drought, wind and natural ignition sources. The impact of this exposure has hit the Australian winegrape and wine industries especially hard following several years of bush- and wild-fires in wine regions. Previous research by this author and others has shown a definite correlation between fruit exposure to smoke and a smoke taint character in the resultant wine.

While previous articles on smoke taint from Australia dealt mostly with the cause and effect of smoke exposure in the vineyard, this Australian report is the first to study the effectiveness of an amelioration technique in smoke-affected wine. With the assistance of a filtration services supplier, this researcher investigated the use of membrane filtration followed by solid phase adsorption to decrease the concentrations of the compounds that are believed to contribute to smoke taint in affected wines. The specific compounds chiefly targeted by this report include guaiacol (Gu) and 4-methylguaiacol (4Mg), called creosol, which are believed to be marker compounds for this particular taint character. The paper also reports on levels of 4-ethyl guaiacol, 4-ethyl phenol and eugenol, other compounds suspected of contributing to smoke taint.

Smoke taint has been described as "smoky", "dirty", "earthy", "burnt" with a lingering retro-nasal "ash" character on the palate. The literature reports that in other food products, such as liquid smoke, smoke character is known to be derived from guaiacol and 4-methyl guaiacol, as they are products of the combustion of lignin. As such, these compounds are routinely found in wines aged in toasted-oak barrels. They are also found in wines made from smoke-affected grapes, but are not found in wines made from unaffected grapes. These compounds are so volatile that their detection levels in wine are measured in parts per billion. Even when they are diluted to sub-threshold concentrations in wine, they continue to contribute to smoke taint. Since Gu and 4Mg can be readily quantified by GC-MS, they are considered useful marker compounds.

The filtration/adsorption process studied in this project works, simply, by separating compounds in wine by their respective molecular weights, then removing the targeted compounds by adsorption. Lower molecular weight compounds, such as water, ethanol, acetic acid, Gu, 4Mg and other volatiles pass through the filter and are captured in the permeate stream. The size of the compounds is determined by the respective filter membrane pore size. Larger compounds don't pass through the membrane, so are retained in the portion called the retentate stream. The permeate, containing the smaller, more volatile compounds, can then be treated by solid phase adsorption to remove specific constituents, according to the type of adsorbent used. The treated permeate and the retentate are then recombined. After recombination, if the levels of the targeted compounds are still too high, the treatment can be repeated until the wine is deemed acceptable.

Two commercial wines were used in this research project: a 2007 Sauvignon Blanc and a 2007 Pinot Noir wine, both from an area in north-east Victoria that was especially impacted by bushfire smoke on 2 different occasions during the growing season. The Sauvignon Blanc wine received 5 complete passes through the filtration/adsorption process, with the adsorbent used being activated carbon (AC). Samples were taken in the untreated wine, the untreated permeate, the treated permeate and the recombined wine. Each sample was analyzed for pH, TA, glucose, ethanol, phenols and browning activity as well as guaiacol and 4-methyl guaiacol. There were only trace amounts of 4-ethyl guaiacol, 4-ethyl phenol and eugenol detected, so they were not discussed.

The adsorbent used in the Pinot Noir treatment was a polystyrene-based resin. Polystyrene was chosen because the AC substrate used in the Sauvignon Blanc treatments was determined to be saturated after only 2-3 passes, so it was not considered suitable for further treatments.

This paper reports the GC-MS analyses (volatile phenols) on samples taken before and after treatment, and at 12 months post-bottling for the Pinot Noir wine. No permeate samples were taken on the red wine. Untreated and treated samples were also checked for pH, TA, glucose, ethanol, total phenolics, color density and hue.

## Results:

The Sauvignon Blanc wine showed a moderate increase in pH (from 3.38 to 3.62) and a corresponding decrease in TA (6.3 gm/L to 5.9 gm/L) after the amelioration treatment. The researchers observed non-significant changes in glucose, ethanol, total phenolics and brown pigments. The decrease noted in total phenolics from 6.36au to 5.34au may be due, in part, to the removal of the smoke-derived phenols by the process. The concentration of guaiacol was decreased from  $8\mu g/L$  (ppb) to  $5\mu g/L$ ; however both concentrations are below the published thresholds for guaiacol in wine. The concentration of 4-methyl guaiacol was 2  $\mu g/L$  to start (also below the reported threshold) and only traces were detected following the treatment. Importantly, NO discernible smoke taint was detected in the untreated Sauvignon Blanc during bench-top sensory evaluations (which was substantiated by the analytical data), so no formal sensory trials were reported for this wine.

The Pinot Noir wine showed a similar decrease in TA, from 9.0 gm/L to 7.8 gm/L, following treatment, but there was no change in the pH. Glucose decreased from 900mg/L to 515 mg/L, alcohol decreased from 14.1% to 13.7%, and total phenolics decreased slightly from 39.13au to 34.79au. These variations were not considered to have a major impact on wine quality. Wine color and density remained constant; an important observation.

In the Pinot Noir samples, the GC-MS analyses of volatile phenols was carried out on samples taken of the untreated wine, the treated wine, and both wines after 12 months bottle-aging. The untreated wine showed 12  $\mu$ g/L guaiacol at bottling, and 15  $\mu$ g/L guaiacol at 12 months post-bottling. The treated wine showed only 5  $\mu$ g/L guaiacol post-treatment (at bottling) and 6  $\mu$ g/L at 12 months. The 4-methyl guaiacol concentration in the Pinot Noir, 3  $\mu$ g/L in the untreated wine at bottling, increased to 4  $\mu$ g/L in 12 months. In the treated wine, the 4Mg was 2  $\mu$ g/L after treatment (at bottling), and increased to 3  $\mu$ g/L at 12 months. The other 3 volatile phenols tested were not detected.

Using a triangle difference test, the author was able to establish that the treatment did have a significant impact on wine aroma. To establish whether the impact was positive or negative, an acceptability test was subsequently performed. Using a 9-point scale, the 48 trained judges gave the untreated wine a 3.64 rating and the treated wine a 4.51 rating; corresponding to a statistically significant preference for the treated wine. Interestingly, the panel didn't associate any smoke-related descriptors with the untreated wine, but instead, found the treated wine to have a cleaner, more fruit-driven character. The author concluded that "the treatment process is, therefore, considered to have improved the overall sensory properties and consumer acceptability of the tainted wine".

## Recommendations for future research:

Since it is possible that low molecular weight wine compounds, such as the volatile compounds responsible for positive aroma attributes in wine, could also permeate the membrane and then be adsorbed by the carbon or polystyrene, the author notes that research has begun to investigate the impact of filtration/adsorption on a broader range of volatile wine constituents.

The author also noted that the activated carbon used in this work was shown to become saturated, and therefore much less effective, over time. Future work might involve the development of a rapid analytical method to test the viability of the substrate, so wine is not unnecessarily subjected to a treatment that has no positive outcome. The adsorption efficiency and selectivity of alternative solid-phase substrates should also be investigated to allow the user to remove only more specific compounds without negatively impacting the concentrations of others.

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