PROGRESS REPORT

Native aroma carryover in *V. vinifera* wines – Part 2: A comprehensive survey of affected wines in Pennsylvania and possible solutions to prevent the problem.

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Objective, Project Overview, and Goals
We proposed a short project that would survey the extent to which native aromas (e.g., 2-aminoacetophenone (2AAP) and methyl anthranilate (MA)) are present in strictly *V. vinifera* wines in the Commonwealth. Our second objective is to conduct a simple, pilot study to investigate potential cleaning methods for PA winemakers that would be effective in desorbing native aroma compounds from polymeric materials in the winery.

Current Results and Status of Project
As of 1 November 2015, we have completed a survey of 35 PA wines, all of which were made solely from *V. vinifera* grapes, wherein we attempted to confirm the presence of MA and/or 2AAP. MA and 2AAP were measured by gas chromatography-mass spectrometry (GC-MS) with sample pre-concentration, as described below. We have also investigated the efficacy of the cleaning/remediation plan described in the original propose and summarized below.

Objective 1: Survey of Pennsylvania Wines Made from *V. vinifera* for Detectable Concentrations of MA and 2AAP

Background & Rationale: In recent years, there have been anecdotal reports of the perception of *V. labrusca*-associated aromas in wines prepared solely from *V. vinifera* grapes. The two main compounds thought to be responsible for these characteristic *V. labrusca* associated aromas are MA (grapey aroma) and 2AAP (foxy, moth ball, acacia blossom, musky aroma). It should be noted that MA and 2AAP are not expected to be present or perceived at all in *V. vinifera* wines, as their presence in these varietals is considered to be atypical and is also considered a defect. Therefore, we have analyzed 35 white *V. vinifera* wines made throughout the Commonwealth with a sensitive GC-MS method to determine the prevalence of these *V. labrusca* associated aromas in those wines. GC-MS is a highly sensitive analytical technique that can separate volatile compounds in a complex mixture (e.g. aroma compounds in wine) and allowed us to detect MA and 2AAP at extremely low concentrations that are comparable to their respective sensory threshold values.

Results. MA was detected 5 *V. vinifera* wines (14.3% of wines surveyed) at concentrations ranging from 30 ng/L (ppt) to 76.1 μg/L (ppb), as shown in Table 1. 2APP was detected (2.28 μg/L) in only 1 wine (2.9% of wines surveyed).

Table 1: PA Wine Survey Results (MA=Methyl Anthranilate and 2AAP= 2Aminoacetophenone). Values for both odorants are displayed in µg/L. *ND indicates that none of the odor was detected.*

Wine	MA	2AAP
1	76.12	2.28
2	28.57	ND
3	0.03	ND
4	5.25	ND
5	ND	ND
6	ND	ND
7	ND	ND
8	ND	ND
9	ND	ND
10	ND	ND
11	ND	ND
12	ND	ND
13	ND	ND
14	ND	ND
15	ND	ND
16	ND	ND
17	ND	ND
18	ND	ND
19	ND	ND
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22	ND	ND
23	ND	ND
24	ND	ND
25	ND	ND
26	ND	ND
27	ND	ND
28	ND	ND
29	ND	ND
30	ND	ND
31	ND	ND
32	ND	ND
33	ND	ND
34	17.5	ND
35	ND	ND

Objective 2: Pilot Study to Investigate Potential Cleaning and Remediation Approaches to Removing MA and 2AAP from Winery Equipment

Background & Rationale. One means to minimize the inadvertent transfer of MA and 2AAP to *V. vinifera* wines is through the use of cleaning solutions. To test the efficacy of various cleaning solutions, PVC was exposed to MA and 2AAP from model juice. PVC was chosen as it was shown to scalp the highest concentration of MA and 2AAP, as reported in our previous work. After scalping, PVC was subjected to acidic (3%HCl), alkaline (3% NaOH) and ethanolic (20%, 40% and 80%) solutions. The acidic and alkaline solutions were chosen in order to mimic current sanitation protocols carried out in food processing environments, and ethanol solutions were chosen based on previous work showing that it is an effective tool in removing odors from polymers. The solutions were allowed to remain in contact with the polymers over three time periods (15, 30, 60 min) and two temperatures (25°C, 75°C) in order to assess the effects of these parameters.

Results. The increase in temperature, time and ethanol content led to increased levels of desorption (Figures 1-4). Increasing temperature causes plasticization of polymers, leading to a higher free volume and faster migration of MA and 2AAP from the polymers into the cleaning solutions. Increasing temperature also increases the volatility of the odorants, which enhances the rate at which MA and 2AAP are desorbed from the polymers. Previous work has shown that increasing ethanol content leads to higher desorption rates. This occurs due to an increase in the nonpolar nature of the solution, making it a more compatible solution for the MA and 2AAP.

While the 20% ethanol treatment led to significantly ($p<0.05$) more desorption than the 3% NaOH treatment, the difference was relatively minor. This is in agreement with previous findings from our previous scalping and desorption studies that found that the behavior of the odorants was similar in both wine (12% ethanol) and juice. Others support the findings in this cleaning study by showing that odorant sorption behavior into polymers is similar for both 20% ethanol solutions and completely aqueous ones. Other reports also found that as ethanol concentrations increased to above 20% there was a sharp increase in the partitioning of the odors from the polymers into the ethanolic solutions, which was documented in this study. This is likely due to the increasing effects of ethanol on reducing the polarity of the cleaning solutions, making it a more thermodynamically favorable environment into which the odorants can partition. Unexpectedly, the alkaline solution treatment allowed for more desorption of MA and 2AAP from PVC in comparison to the acidic solution. Other studies have found similar findings where basic solutions allowed for the desorption of odorants from polymeric materials.

In conclusion, we observed that increasing time, temperature and ethanol content allowed for more extensive desorption of MA and 2AAP from PVC. The efficacy of these cleaning solutions on other polymers with different structures may vary and should be further investigated. Altering other parameters of the cleaning protocols, such as increasing time and temperature, performing multiple washings, and using other solvents (i.e., more nonpolar solvents) may alter the reported results and should be further evaluated.

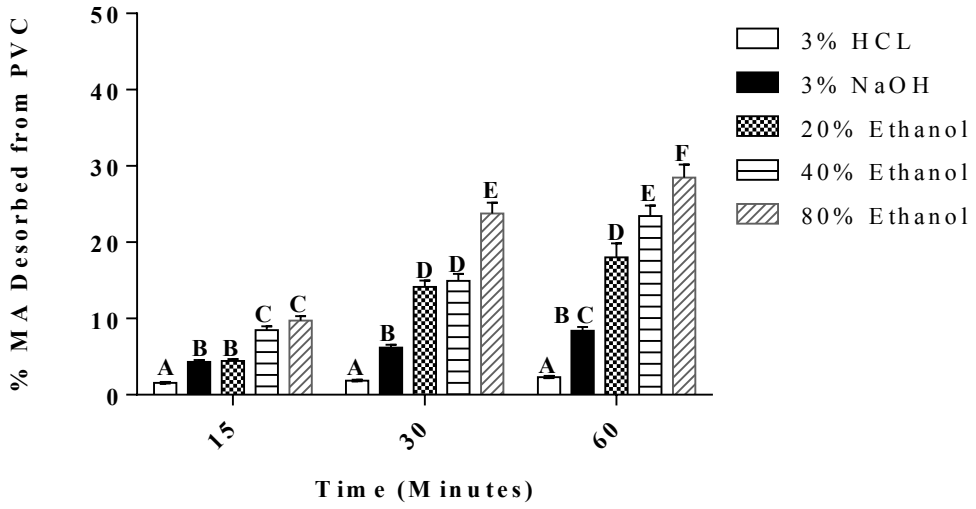

Figure 1: Desorption of Methyl Anthranilate from PVC at 25°C
Values with different capital letters are considered significantly ($p<0.05$) different from one another. Error bars represent +/- 1 SD from mean.

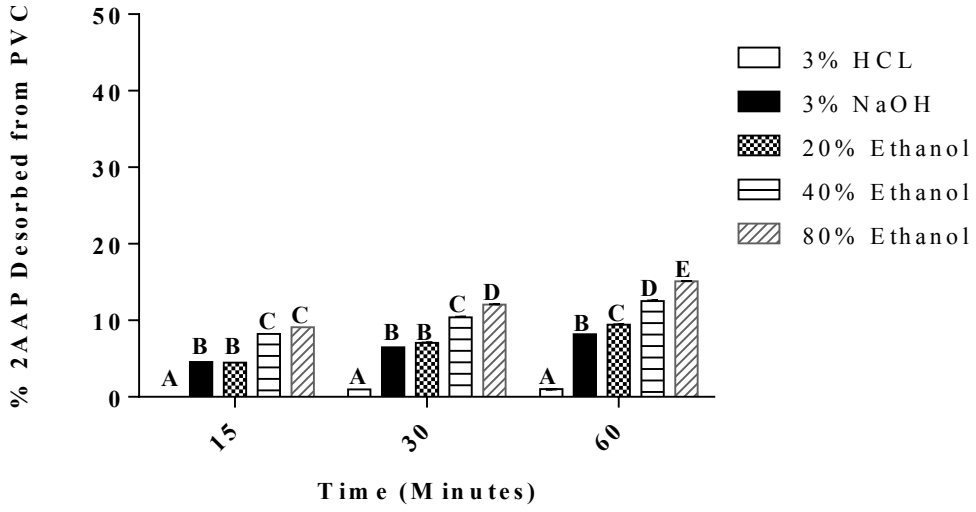

Figure 2: Desorption of 2-Aminoacetophenone from PVC at 25°C
Values with different capital letters are considered significantly ($p<0.05$) different from one another. Error bars represent +/- 1 SD from mean.

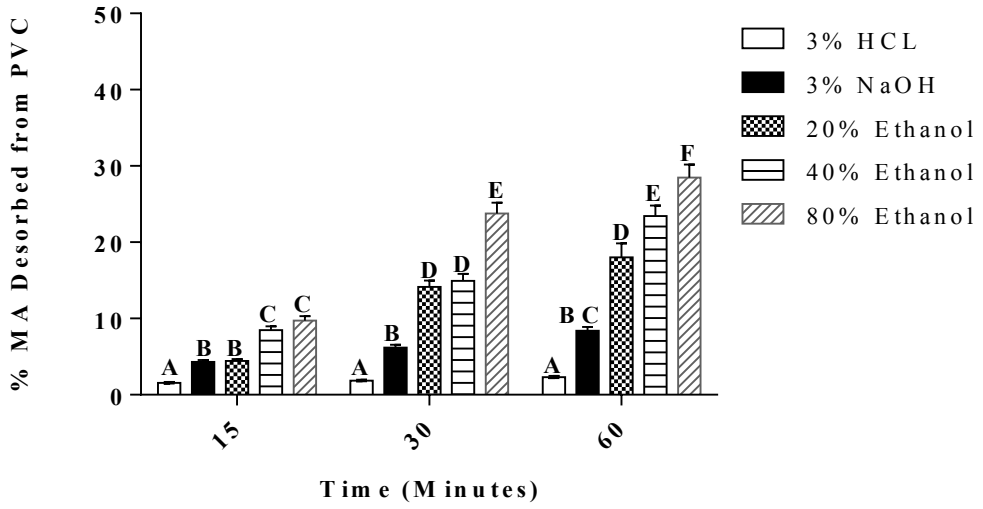

Figure 3: Desorption of Methyl Anthranilate from PVC at 75°C
Values with different capital letters are considered significantly ($p<0.05$) different from one another. Error bars represent +/- 1 SD from mean.

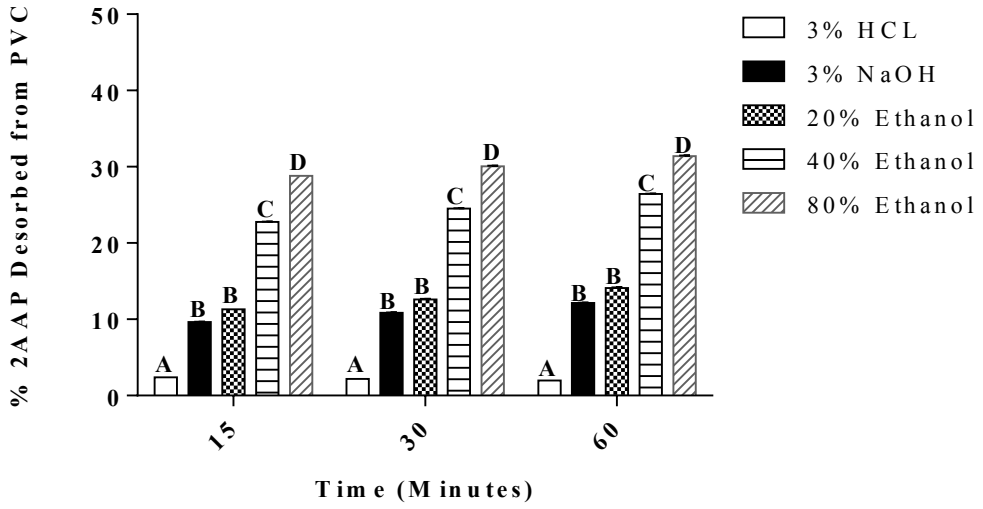

Figure 4: Desorption of 2-Aminoacetophenone from PVC at 75°C
Values with different capital letters are considered significantly ($p<0.05$) different from one another. Error bars represent +/- 1 SD from mean.