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Effects of Fining Agents, Reverse Osmosis and Wine Age on Brown Marmorated Stink Bug (*Halyomorpha halys*) Taint in Wine

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Abstract: *Trans*-2-decenal and tridecane are compounds found in wine made from brown marmorated stink bug (BMSB)-contaminated grapes. The effectiveness of post-fermentation processes on reducing their concentration in finished wine and their longevity during wine aging was evaluated. Red wines containing *trans*-2-decenal were treated with fining agents and put through reverse osmosis filtration. The efficacy of these treatments was determined using chemical analysis (MDGC-MS) and sensory descriptive analysis. Tridecane and *trans*-2-decenal concentrations in red and white wine were determined at bottle aging durations of 0, 6, 12 and 24 months using MDGC-MS. Reverse osmosis was found to be partially successful in removing *trans*-2-decenal concentration from finished wine. While tridecane and *trans*-2-decenal concentrations decreased during bottle aging, post-fermentative fining treatments were not effective at removing these compounds. Although French oak did not alter the concentration of tridecane and *trans*-2-decenal in red wine, it did mask the expression of BMSB-related sensory characters. Because of the ineffectiveness of removing BMSB taint post-fermentation, BMSB densities in the grape clusters should be minimized so that the taint does not occur in the wine.

Keywords: *trans*-2-decenal; tridecane; MDGC-MS; red wine; Pinot noir

1. Introduction

Brown marmorated stink bug (BMSB) contamination in grape clusters can have a negative effect on wine quality [1,2]. BMSB is an invasive pest that is believed to have arrived into the United States from East Asia and is currently detected in 43 states. Globally, it is also found in Canada, Italy, Hungary and other European countries where wine has economic importance [3,4]. When present in the vineyard, the pest can lower crop yield and effect quality. When present in grape cluster, it may enter wine processing where it can harm wine quality through the release of "BMSB taint" compounds. The chance of BMSB entering wine processing is increasing, as greater densities of BMSB are being observed in the vineyard [3,5,6]. In order to maintain wine quality, techniques are needed to minimize BMSB taint concentration in finished wine.

BMSB primarily secretes tridecane and *trans*-2-decenal when stressed [7–9]. Tridecane is an odorless compound and its effect on wine quality is currently unknown. *Trans*-2-decenal is considered to be the main component of BMSB taint due to its strong "green", "cilantro"-like aroma [2,10]. It has been shown to have a negative effect on red wine quality, significantly decreasing consumer preference at a concentration as low as 4.8 µg/L, the determined consumer rejection threshold (CRT) [1]. Above this concentration *trans*-2-decenal can add green, musty, herbal characteristics to wine which are not desirable [11]. Additionally, a reduction in favorable attributes such as dark fruit, red fruit and floral characteristics has also been observed [11]. Due to this negative impact of BMSB taint on wine quality and consumer preference, efforts are needed to minimize the concentration of these taint compounds in finished wine.

It has been shown that as low as three BMSB per cluster in the vineyard can result in finished wine with 2.02 μg/L of *trans*-2-decenal [12]. The same bug density may also result in *trans*-2-decenal concentration at or above the CRT when winemaking causes stress to BMSB, resulting in higher secretion of taint compounds. Additionally, previous work suggests that the presence of dead BMSB can also result in wine containing tridecane but not *trans*-2-decenal [12].

Modifications in wine making protocol may reduce BMSB taint concentrations in final wine, as winemaking processes are known to alter aroma composition. Alterations in harvesting, pressing and fermentation have shown potential in reducing BMSB taint in finished wine [12]. However, modification of wine processing may not be always appropriate as it can restrict the style of wine made from BMSB contaminated grapes. Additionally, process modification may not be sufficient against high BMSB densities and finished wine may still contain *trans*-2-decenal or tridecane. Therefore, post-fermentative measures are required to be able to produce a desired wine style while minimizing taint levels.

The wine industry relies on fining agents to correct wine faults and to improve wine quality [13–15]. Wine sensory characteristics such as flavor, color and mouthfeel can be adjusted by fining agents [14,16,17]. Fining agents are chosen for their affinity to unwanted compounds in wine through mechanisms such as hydrophobic interaction, hydrogen bonds, Vander Waals interaction and electrostatic interactions [13,18]. The fining agent, along with unwanted or taint compounds, are then removed by racking, centrifugation or filtering. Commonly used fining agents such as bentonite, gelatin, casein and activated charcoal have previously been used on taint compounds from lady bug and smoke exposure [13,15]. In these studies, oak was able to mask green aroma characteristics of lady beetle taint in red and white wine whereas activated charcoal and synthetic mineral were effective against smoke taint compounds.

In addition to fining agents, reverse osmosis filtration has also been explored as a viable option for taint removal [19]. In this process, selective taint removal can be achieved by carrying out filtration under pressure. Reverse osmosis is often combined with an adsorption or ion-exchange column to remove taint compounds more efficiently. This technique has been found to be successful in removing 4-ethylguaiacol and 4-ethylphenol from *Brettanomyces*-affected wine [20] using Amberlite XAD-16 HP resin and smoke taint compounds (guaiacol, 4-methylguaiacol, 4-ethylguaiacol and 4-ethylphenol) using a polystyrene-based adsorbent resin [19].

Another possible technique to reduce BMSB taint in wine is through aging. During aging, complex reactions occur that are known to change wine composition and sensory characteristics [21,22]. Since most wines are aged for at least a year, it is important to understand how BMSB taint is modified during aging. This information is important in order to assess the quality of an aged wine. Currently, there is no technique known to be effective against BMSB taint in finished wine and reduction techniques are needed to help deal with this spoilage issue when control of BMSB in the vineyard is ineffective.

2. Materials and Methods

Wines—Three different Pinot noir wines (PN1, PN2 and PN3) were used in this study. All wines were produced on a small scale at the Oregon State University research winery (Corvallis, OR, USA). PN1 and PN2 were produced from the same grapes sourced from a vineyard located in Oregon and under the same winemaking protocol. PN1 was made from grapes that did not contain any BMSB. PN2 on the other hand, was made from grapes to which BMSB were added prior to destemming at a density of three per cluster. PN2 therefore contained both taint compounds, tridecane and *trans*-2-decenal whereas PN1 was taint free. Both wines, PN1 and PN2 were made using a winemaking protocol as described in [12]. These wines were used to study the effect of aging and reverse osmosis filtration. PN3 was also produced without any addition of BMSB but the grapes were sourced from a different vineyard in Oregon. Winemaking procedure used to make PN3 was similar to the protocol given in [17]. *Trans*-2-decenal was added to this wine to study the effect of fining treatment, as not enough BMSB were available that year to make naturally tainted wines at the concentrations needed.

Fining agents—Fining agents and their dose levels were selected based on preliminary studies and/or manufacturer recommendations. In the end, five fining agents: gelatin (BBL, Div Becton Dickinson & Co., Sparks, MD, USA), egg albumin, potassium caseinate (Laffort USA, Petaluma, CA, USA), bentonite, yeast lees (Oenolees®, Laffort USA, Petaluma, CA, USA) and French oak bean, medium plus toast (StaVin Inc., Sausalito, CA, USA) were tested. Egg albumin solution was prepared in 1% NaCl using eggs from the local grocery store. Other fining agents were prepared in hot or cold water per manufacturer's instruction. The following dose levels were used for each fining agent: gelatin at 30 mg/L, egg albumin at 67 mg/L, potassium caseinate at 150 mg/L, bentonite at 75 mg/L, yeast lees at 150 mg/L and French Oak bean at 1.5 g/L The addition rate for French oak was based on the manufacturer's instruction for 50% new oak.

To run fining trials, Pinot noir with *trans*-2-decenal concentration of 30 μg/L was prepared. This was done by adding *trans*-2-decenal standard (50 mg/L made in 14% ethanol) into the base wine, PN3. The concentration of 30 μg/L was selected for fining treatment because it is significantly above *trans*-2-decenal CRT (4.8 μg/L) and has been shown to add green sensory characteristics associated with *trans*-2-decenal. Additionally, a significant proportion of consumers (78%) were seen to reject Pinot noir containing 30μg/L of *trans*-2-decenal [1].

Twenty-four hours after *trans*-2-decenal addition into PN3, fining agents were added. PN3 containing *trans*-2-decenal and fining agent were then stored at 4 °C for three days. At the end of three days, fining agents were removed by racking, wines were rebottled and stored for analysis at 4 °C. Twenty hours after racking, wines were analyzed using sensory descriptive analysis. At the same time, 40 mL sample of these racked wines was collected for *trans*-2-decenal quantification using MDGC-MS. Samples were stored in amber vials with PTFE lined caps (Sigma Aldrich, Darmstadt, Germany) at −18 °C until their analysis. All fining trials were conducted in triplicate.

Reverse osmosis—Two wines, PN1 and PN2 went through reverse osmosis filtration conducted by WineSecrets Corp. (Sebastopol, CA, USA). PN1 was treated by reverse osmosis as well as the BMSB tainted wine to determine the effect of reverse osmosis on aroma compounds not typically associated with BMSB. Therefore, both wines were treated in exactly the same manner. Reverse osmosis was performed on a lab scale Memstar unit with a CBC-5 carbon block filter cartridge (Pentair Pentek, Milwaukee, WI, USA) attachment. Feeding pump pressure was maintained between 1700–1800 kPa and total sample flow rate at 50 mL per minute. All wines that went through reverse osmosis were done in triplicate.

Aging—PN1 and PN2 wines were aged in 750 mL screw-cap closed (Stelvin, Amcor, CA, USA) bottles in a dark wine cellar located at Oregon State University at 13 °C for 1 year. At 0, 6 and 12 months, bottles were removed from the cellar and 40 mL samples were stored in amber vials with PTFE lined caps (Sigma Aldrich) at −18 °C for later analysis. All wines were aged in triplicate.

Chemical Analysis—Taint compounds were measured using a previously developed HS-SPME-MDGC-MS method [12].

Sensory Analysis—Descriptive analysis was used to determine the effect of fining treatment and reverse osmosis on wine sensory characteristics. Sixteen wine professionals (12 M, 4 F) from the Oregon wine industry participated in this study. Each panelist had more than 10 years of experience tasting wines. Consent was obtained from all panelists and the study was approved by Oregon State University's Internal Review Board (IRB). Descriptive analysis data was collected over three tasting sessions, each lasting two hours. Each of the three sessions was conducted in the morning. The third session was conducted in a different room but under similar light and temperature (21 ± 2 °C) conditions. Wines were served in INAO black glasses (International Organization for Standardization 1977) to remove any influence of color [23]. All samples were coded with a three digit random numbers and served in a random order.

At the start of each session, panelists were given a set of three wine samples; control, Pinot noir with *trans*-2-decenal at its CRT (4.8 μg/L), and Pinot noir with *trans*-2-decenal above its CRT (30 μg/L). This was done to familiarize panelists with the taint compound and its effect on Pinot noir aroma and flavor. These wines were prepared an hour before the tasting session by adding a *trans*-2-decenal standard (50 μg/L prepared in 14% ethanol). Wines were served in three sets containing five samples each. To avoid the effect of fatigue, panelists were given a one-minute break after each wine and five minutes after each set. Panelists were requested to rinse their palate and eat a cracker during each break to minimize any carryover effect.

Samples were evaluated for ten aromas (dark fruit, earthy, herbal, musty, red fruit, floral, fresh green, spice) and three flavors (fruit density, green, and spice). These attributes have been used previously to evaluate the effect of *trans*-2-decenal on Pinot noir quality [11]. Each attribute was rated on a 100 mm visual analog scale with indented word anchors, none and extreme. Panelists were allowed to rate any other attribute they thought was relevant to describe these samples, to avoid any dumping effect [24].

Statistical Analysis—Any differences in *trans*-2-decenal concentration between PN3, with and without fining agents was analyzed using one-way ANOVA and Dunnett's test. Descriptive analysis data was analyzed using mixed model ANOVA to determine consensus among the assessors for each attribute and wine. The fixed effect was wine and the random effects were panelists and replication. Canonical variate analysis (CVA) was used to explore the separation between wine treatments [25,26]. Significant differences during aging were analyzed using one way Analysis of Variance (ANOVA) and Tukey's HSD. All analyses were conducted using XLSTAT-Pro 2015 (Addinsoft, New York, NY, USA).

3. Results

3.1. Chemical Analysis

Trans-2-decenal was added to PN3 and was present at a concentration of 23.88 μg/L prior to the addition of any fining agents. After fining, no significant differences were found between *trans*-2-decenal concentrations in base wine and fined or oaked wines ($p < 0.05$, Dunnett's). Treatment of PN2 wines by reverse osmosis reduced the *trans*-2-decenal concentration in wine from 2.02 μg/L to 1.82 μg/L (*t*-test, p-value < 0.05), a 10% reduction of the compound. Chemical analysis of the aged PN2 wines showed a decrease in tridecane concentration in PN2 wine during bottle aging (Figure 1). *Trans*-2-decenal was 2.02 μg/L at 0 months and decreased to undetectable by 6 months.

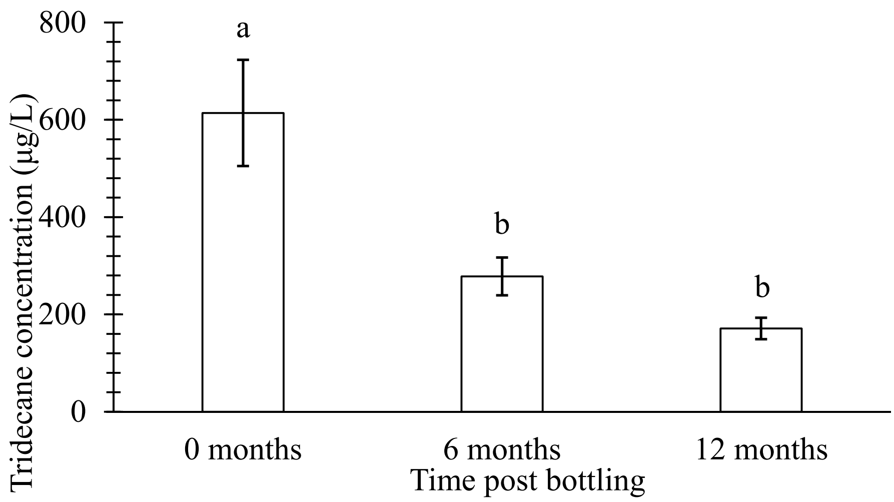

Figure 1. Tridecane concentration in Pinot noir made from BMSB containing grapes, PN2 at 0, 6 and 12 months of bottle aging ($n = 3$). Means with the same letter are not significantly different from each other (Tukey's HSD, α: 0.05).

3.2. Sensory Analysis

The effect of fining agent on reducing the sensory impact of *trans*-2-decenal was evaluated using eight wines. Six wines relating to the different fining treatments were evaluated. The other two wines were PN3, with and without *trans*-2-decenal. A significant interaction between panel and wine treatment was observed for spice flavor. This indicates inconsistency in the use of intensity scale or interpretation of this attribute by wine professional [24]. The effect of such an interaction can result from inherent anatomical differences in panelists. There is also the possibility of the term "spice" being too generic and therefore being interpreted differently by each panelist. Given that the interaction exists, spice flavor was excluded from CVA analysis. ANOVA of the sensory descriptors showed a significant difference in red fruit aroma between wine treatments (F value = 3.07, df = 7, p-value = 0.03). However, no difference in *trans*-2-decenal-related attributes, such as green, musty and earthy, were found. The only observed difference (a change in red fruit aroma) is typically considered as a side effect of using fining agents [27].

CVA analysis (Figure 2) showed a clear separation between the wines. Three distinct groups were visible:

Group 1—PN3 without *trans*-2-decenal
Group 2—French oak treatment
Group 3—remaining six wines (PN3 with *trans*-2-decenal and the other five fining treatments).

PN3 without *trans*-2-decenal was mainly characterized by fruity aroma and flavor. Wine with French oak treatment showed a strong spice aroma but no *trans*-2-decenal-related attributes. Since the chemical analysis of oak treated wines did not show any change in *trans*-2-decenal concentration the effect of oak is most likely a masking effect.

The two wines that underwent reverse osmosis (RO) were assessed by sensory analysis. PN3 with and without *trans*-2-decenal were also included in the analysis for comparison. Thus, in the second CVA analysis, a total of four wines were analyzed: (1) PN1 (made from BMSB free grapes) after RO; (2) PN2 (wine made from BMSB added grapes) after RO; (3) PN3 with *trans*-2-decenal and (4) PN3 without *trans*-2-decenal. The last two wines were included in the analysis in order to provide a means to compare the effect of reverse osmosis filtration and high levels of *trans*-2-decenal on wine aromatics.

A significant interaction was observed between panel and wine for green flavor. Using the same justification as before, green flavor was removed from CVA analysis. The results of CVA analysis are shown in Figure 3. Three groups were seen on a CVA plot:

Group 1—PN3 with *trans*-2-decenal
Group 2—PN3 without *trans*-2-decenal
Group 3—PN1 and PN2 after RO.

These three groups show clear differentiation between wine without *trans*-2-decenal, with *trans*-2-decenal and those wines that went through RO.

As with the previous sensory analysis, PN3 without *trans*-2-decenal was perceived as fruity and floral. The addition of *trans*-2-decenal brought out green, musty herbal characteristics in the same wine. None of these negative characteristics were found in PN1 and PN2 treated with RO. After reverse osmosis, PN2 was mainly characterized by earthy notes but none of the more pronounced attributes associated with *trans*-2-decenal such as green, musty and herbal were observed.

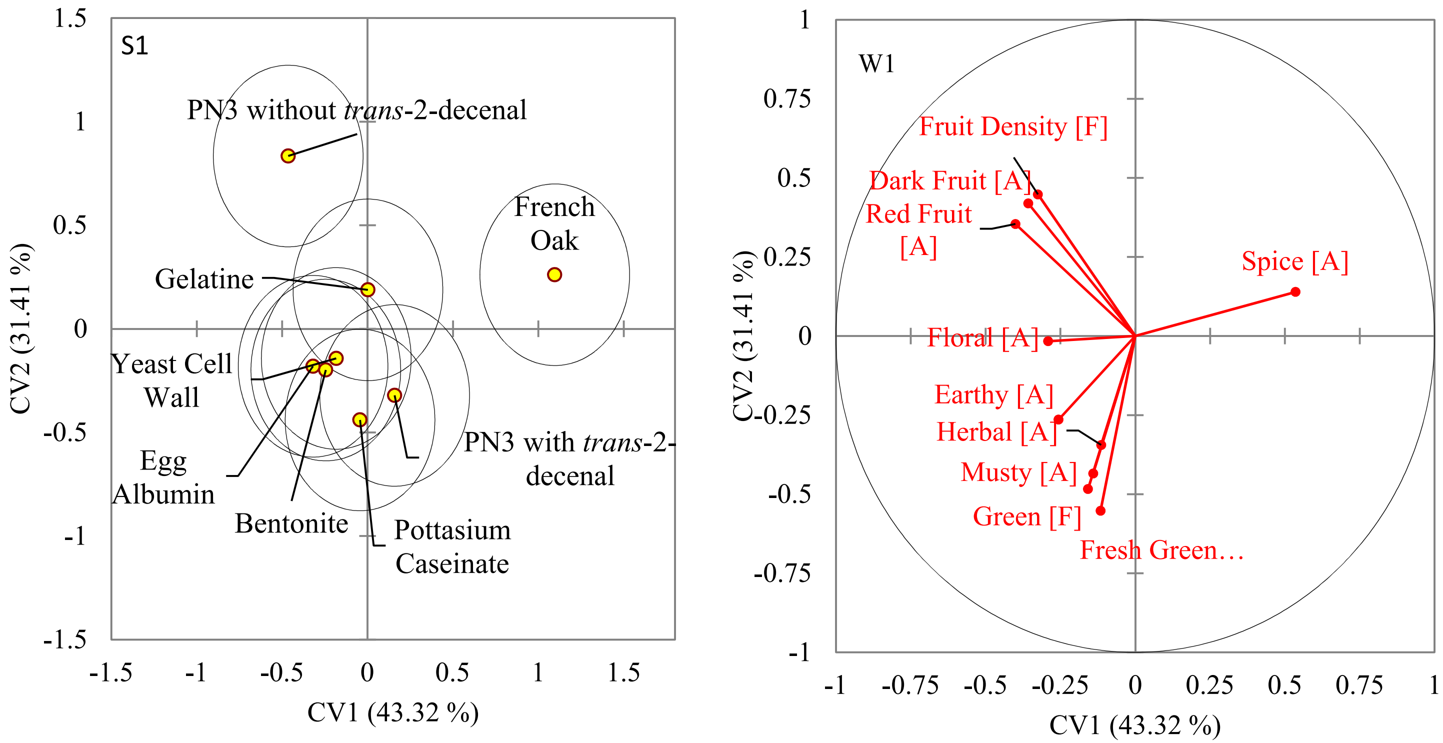

Figure 2. Separation between PN3 without *trans*-2-decenal, PN3 with *trans*-2-decenal (23.88 µg/L) and PN3 containing *trans*-2-decenal (23.88 µg/L) when treated with fining agents (Gelatin, Bentonite, Yeast cell wall, Potassium caseinate, Egg albumin, French oak). Wines are positioned using the centroids. Circles represent 95% confidence intervals surrounding the wine means. Vectors for sensory terms (A = aroma, F = in mouth flavor) are in W1 and scores for wines are in S1. Significant differences for wines are for circles that do not touch in S1.

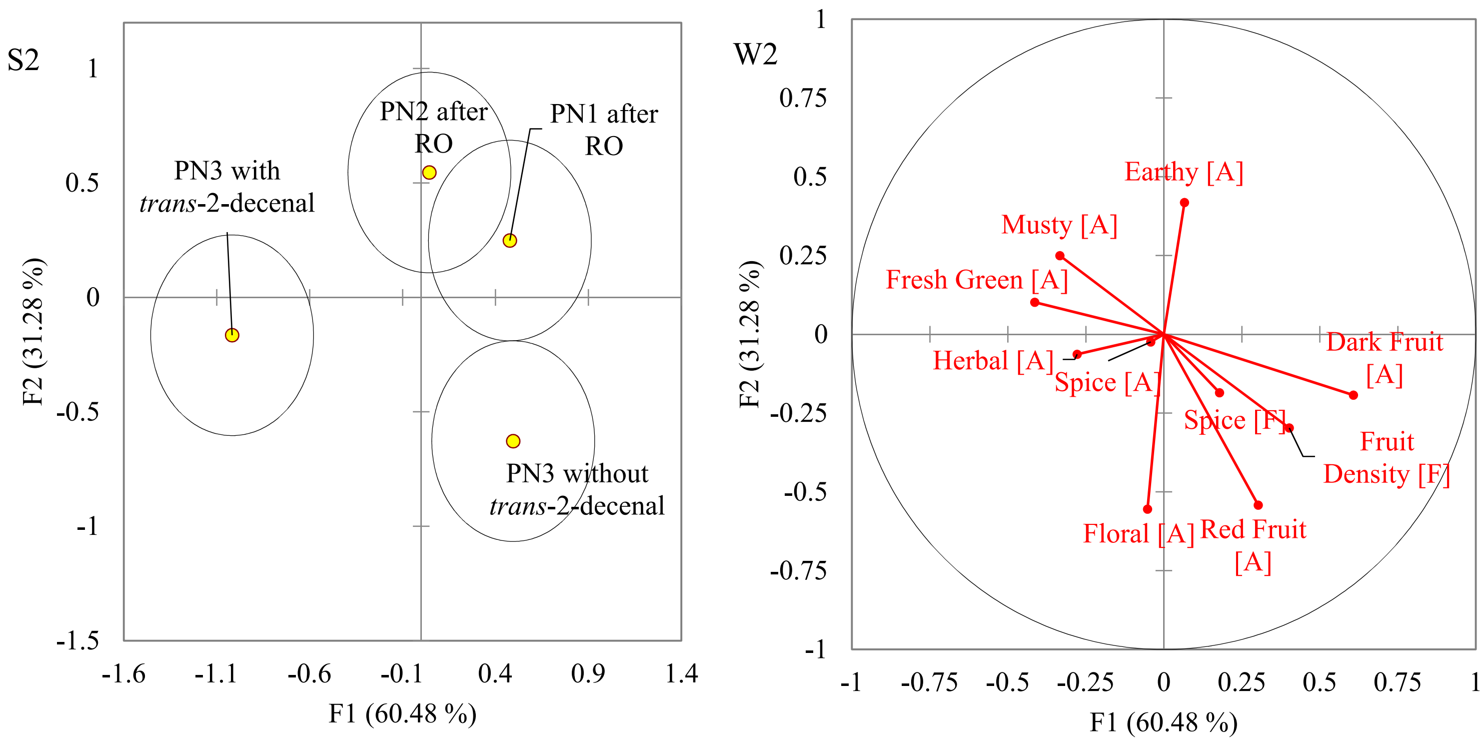

Figure 3. Separation among wines that went through reverse osmosis (RO) filtration: PN1 (wine made from *BMSB free* grapes), PN2 (wine made from BMSB containing grapes and contains 1.82 µg/L of *trans*-2-decenal) and wines that did not go through reverse osmosis (RO) filtration: PN3 without added *trans*-2-decenal, PN3 with *trans*-2-decenal (23.88 µg/L) by CVA. Wines are positioned using the centroids. Circles represent 95% confidence intervals surrounding the wine means. Vectors for sensory terms (A = aroma, F = in mouth flavor) are in W2 and scores for wines are in S2. Significant differences for wines are for circles that do not touch in S2.

4. Discussion

BMSB taint in wine can be detrimental to wine quality but little is known regarding ways to remove this taint from wine. While fining agents are often used during winemaking to remove wine taints, the fining agents evaluated in the present study were incapable of removing *trans*-2-decenal in BMSB tainted wines. Additional work is needed to determine the underlying factors for this result. One potential reason may be that the fining agents have only weak binding ability with *trans*-2-decenal. Alternatively, the fining agents may have higher affinity for phenolic compounds or other aroma compounds compared to *trans*-2-decenal. This explanation seems likely given that most fining agents are known to bind with non-volatile components in wine such as proteins and phenolic compounds [14,16,18,28]. Their interaction with volatile compounds is considered to be a secondary binding action and an undesirable effect that can be exploited for taint reduction [13,14,28,29].

The addition of French oak chips was the only treatment to have an impact on BMSB taint in the wines with a masking effect being observed during sensory analysis. Previous work on lady bug taint has also reported a similar masking effect of oak addition [15]. Wines treated with fining agents were all described with similar characteristics as the *trans*-2-decenal wines. Overall, the results of sensory evaluation agree with the conclusion of the chemical analysis data, namely, that fining agents failed to remove *trans*-2-decenal from wine.

Reverse osmosis resulted in a slight reduction of *trans*-2-decenal of 0.2 µg/L. This is minimal and would likely only be effective in changing sensory perception if the final concentration after fining was near *trans*-2-decenal CRT. However, this result indicates the ability of reverse osmosis to reduce *trans*-2-decenal, which was not found with other treatments. Therefore, with additional improvements reverse osmosis may prove to be a viable option for BMSB taint management. The use of other semipermeable membranes, adsorption/ion exchange column, pressure and flow rate should be investigated to remove greater amounts of *trans*-2-decenal.

The impact of RO on the sensory characteristics of *trans*-2-decenal are unclear. BMSB associated aromas including green, musty and herbal where not found in RO wines, but an earthy aroma was described in these wines. Previous sensory analysis conducted on wines with 5 µg/L *trans*-2-decenal also were found to be "earthy" [11]. However, wine after RO was described as different from the control (PN3 without *trans*-2-decenal) so it is unclear if this would be problematic to wine quality. The usage of RO to reduce BMSB warrants further research.

Aging appeared to be the most effective treatment to reduce *trans*-2-decenal and tridecane in BMSB tainted wines. A longer aging period may be preferable in wines containing BMSB taint since the aging process appears to naturally decrease their levels. The decrease in BMSB taint post bottling is likely to be a result of aging-related reactions occurring in wine such as hydrolysis, component degradation, condensation and reduction reactions [21,22,30]. These reactions can modify existing compounds or generate new ones.

Aldehydes, such as *trans*-2-decenal, are highly reactive and can bind with a number of different compounds such as SO_2 or phenolic compounds [31,32]. Prior work has shown that reductive conditions during bottle aging can cause aldehydes to decrease in wines as they change to their corresponding alcohol [14,33]. 1-Decanol, the corresponding alcohol for *trans*-2-decanal, has a detection threshold (5 mg/L) that is much greater than *trans*-2-decenal [34]. Therefore, as the wine ages and *trans*-2-decenal decreases the effects of 1-decanol may be minimal. However, if wines are matured under oxidative condition, acetals can form as result of reaction between aldehydes and alcohol [14,35]. Additional research is needed to better understand the transformation of *trans*-2-decenal during aging. This taint may only be problematic for young wines, as *trans*-2-decenal was undetectable after 6 months. However it is unknown if the transformation products of *trans*-2-decenal may also be problematic to wine quality.

The corresponding sensory impact of BMSB taint during aging also needs to be evaluated. This will estimate the effect after compounds released by BMSB have undergone aging-related changes. We did not have enough wine to conduct sensory tests on the aged wines but this is needed as preliminary sensory tests showed that BMSB taint wines were reduced in 1 year aged wines, but wines were very different from the control wines.

5. Conclusions

Corrective measures are of significant importance for wine containing BMSB taint. They provide the last option for winemakers to correct wine faults. Taking this into consideration, this study evaluated the effectiveness of reverse osmosis, commonly used fining agents, and bottle aging on BMSB taint compound. While fining agents proved ineffective at removing BMSB taint, reverse osmosis showed promise as *trans*-2-decenal concentrations in wine were reduced by 10% and resulted in improvements in the wines sensory characteristics. In addition to reverse osmosis, oak addition can also be considered while dealing with BMSB taint as it successfully masked the green characteristics associated with *trans*-2-decenal in wine. Finally, aging wines for extended periods of time may offer the best strategy for reducing BMSB taint in red wines and is also a process that most wines will undergo as part of the winemaking process. Taken together, wine containing BMSB can be treated by one of three options, addition of French oak, reverse osmosis or aging. Overall, the outcome of this study provides means of dealing with BMSB taint in finished wine. This information is important to minimize the impact of BMSB taint and maintain wine quality.

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Conflicts of Interest: "The authors declare no conflict of interest."

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