NYWGF RESEARCH - FINAL REPORT

Funding for fiscal year: 2022-23

SECTION 1:

Project title: Validating accelerated testing for predicting maximum SO₂ concentrations in canned wines

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New Research \boxtimes Continued Research \square

Amount Funded \$ 53,672

SECTION 2: (This section should be in depth and akin to an academic report)

Project Summary Impact Statement: Canned wines represent one of the fastest growing sectors of wine packaging in the US, including in New York State, but concerns about off-aromas from hydrogen sulfide (H₂S, "rotten egg") and other defects have limited their adoption. Using newly developed assays, we validated that molecular SO₂ is an excellent predictor of H₂S formation. We also observed that conventional BPA Epoxy and newer BPA-NI Epoxy can liner materials provide best performance across can manufacturers. These results will be delivered to winemakers through peer-reviewed and trade publications in coming months and will limit the unnecessary avoidance of cans due to concerns by producers.

Objectives: Our objective was to use both a recently developed accelerated aging test and long term room temperature studies to validate and extend recent findings regarding canned wines: to predict failure of canned wines during long term storage (H₂S formation, corrosion) for canned wines; to confirm the importance of molecular SO₂ as a predictor of H₂S formation during storage; and to evaluate the performance of different can liners across different manufacturers.

Materials & Methods:

Experiment 1, Methods: Validation of accelerated aging assay across multiple wines with varying composition

- 1) <u>Wines</u>: Five wines (two white, two red, one rose) were provided by an industry partner. Wines were adjusted through SO₂ and base additions in the following manner to achieve a range of molecular and free SO₂ values
 - a. Control (no adjustment)
 - b. Increased free SO₂, decreased molecular SO₂ (add free SO₂ and increase pH)
 - c. Increased free SO₂, increased molecular SO₂ (add free SO₂; pH constant)
- 2) <u>Accelerated aging trials:</u> A recently validated method (See Figure 1, left) will be used for accelerated aging tests. Briefly, wines were stored in 20 mL amber vials in the presence of 5 cm x 1 cm coupons with BPA-NI coating Uncoated coupon edges were protected with EVA hot-melt glue. A negative control coupon was prepared by coating the entire coupon in epoxy. Vials were be purged with inert gas to keep total package oxygen < 3 mg/L, and vials stored at elevated temperature (50 °C) for 3 and 14 days prior to measurements. Treatments were performed in triplicate.</p>
- 3) <u>Storage experiments</u>: Wines were sourced and prepared as described for accelerated aging trials. Can seaming was performed manually using previous validated protocols at Cornell. Cans were sampled at 16 and 32 weeks. All experimental treatment timepoints were performed in triplicate.
- 4) <u>Corrosion characterization</u>: Corrosion was measured in both the accelerated and longterm storage studies
 - a. Dissolved AI was measured as a proxy for overall aluminum corrosion using ICP-AES
 - b. Hydrogen sulfide (H₂S) was measured by a gas detection tube method (Allison et al. 2021).

Experiment 2, Methods: Validation of accelerated aging to predict liner quality

- 1) <u>Wine:</u> A single high molecular SO₂ white wine (pH 3, free SO₂ = 50 mg/L, molecular SO₂ = 2.1 mg/L) was utilized for the trial.
- 2) <u>Cans, coupons, and storage experiments:</u> Multiple commercial sources of BPA Epoxy (n=3 manufacturers), BPA-NI (n=2), and acrylic (n=3) were obtained. For long-term storage trials, canned wines were prepared as described for Experiment 1. Cans were sampled at 16 and 32 weeks. For accelerated aging trials, 5 cm x 1 cm coupons were cut from cans using tin snips, after which the Experiment 1 protocol was followed.
- 3) <u>Corrosion characterization</u>: H₂S formation and dissolved AI were measured as described for Experiment 1.

Results/Outcomes/Next Steps:

Experiment 1, Results and Outcomes

- Commercial wines were adjusted to varying pH and free SO₂ concentrations, for a total of 15 wines.
- The wines were stored under accelerated and long-term can conditions
- The importance of molecular SO₂ in predicting H₂S formation during long-term storage was validated. For example, after 16 weeks room temperature storage, the five wines with high molecular SO₂ (1.5-2.5 mg/L) produced the highest concentrations of H₂S (25-55 µg/L). Wines with lower molecular SO₂ produced H₂S below sensory threshold (<10 µg/L).
- The mediocre correlation of free SO₂ and eventual H₂S formation was also validated. No significant correlation was observed, and high free SO₂ only led to high H₂S production for wines with low pH (and, thus, high molecular SO₂).
- For three of the five base wines, good correlation in H₂S formation was observed between accelerated-aging and long-term storage conditions. However poor correlation was observed two of the five base wines, which produced far less H₂S than expected under accelerated aging conditions. Follow-up work determined that those two wines had been contaminated with copper, which suppressed H₂S accumulation.
- <u>Next Steps</u>: H₂S measurements will be performed in canned wines at the 32-week (8 month) time point. Dissolved aluminum will be measured in all samples, 16-week and 32-week.

Experiment 2, Results and Outcomes

- A highly corrosive wine (i.e. high molecular SO₂) was stored in the presence of 8 different commercial liner materials (3 acrylic, 3 BPA Epoxy, 2 BPA-NI) under both accelerated and room-temperature storage conditions.
- Within a liner material category, no significant differences were observed among manufacturers. However, considerable variation was observed among liner material categories. H₂S production for acrylic liners was highest (from 25 to >200 µg/L) but was generally <10 µg/L for BPA Epoxy and BPA-NI liners.
- Certain liner manufacturers appeared to have greater can-to-can variability, although the ability to assess this was limited by the low number of replicates per liner (n=3).
- <u>Next Steps</u>: Accelerated aging trials will be performed on a larger number of samples per liner source to evaluate manufacturer consistency.

Technology Transfer Plan: Following completion of data collection in March, we expect to prepare a manuscript for submission to the American Journal of Enology and Viticulture. We also plan to submit an article summarizing our learnings on canned wine to Wine Business Monthly or a related trade publication.

Attachments:

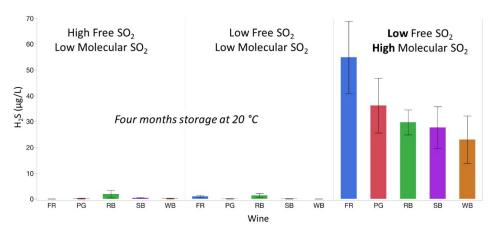


Figure 1 – H_2S production in canned wines as a function of initial free and molecular SO_2 . Five commercial wines (FR, PF, RB, SB, WB) were sourced, and pH and SO_2 adjusted to yield 15 wines for evaluation. Wines were stored at room temperature for four months in BPA-NI epoxy lined cans.

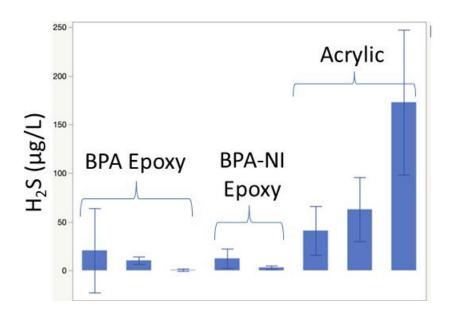


Figure 2 – H_2S production during four month, room temperature storage of a high molecular SO_2 white wine in one of eight commercial cans: BPA epoxy (n=3), BPA-NI epoxy (n=2), and acrylic (n=3).

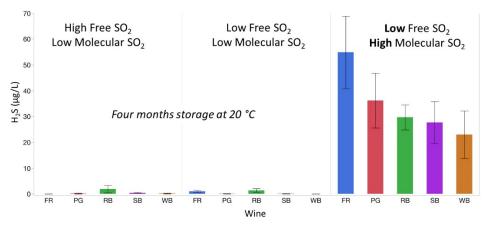
SECTION 3:

Project summary and objectives: Sales of canned wines and many other alcoholic beverages have grown markedly in the last decade. However, recent work by our group has demonstrated that canned wines may suffer from liner degradation, corrosion, and off-aromas (H₂S, "rotten egg") after several months' storage. Preliminary work suggested that storage problems are best predicted by the concentration of wine molecular SO₂. Our

objective was to validate the importance of molecular SO₂ under both accelerated and longterm storage conditions, and to evaluate the variability of can liner performance among can manufacturers.

Importance of research to the NY wine industry: Canned wines represent one of the fastest growing sectors of wine packaging in the US, including in New York State. However, despite demand for canned wines by consumers, concerns about off-aromas and can failure have limited that adoption of canned wines by some winemakers. This research validates high molecular SO₂ as a major predictor of corrosion problems during canned wine storage, and also determines that liner type (and not can manufacturer) is the most important criteria for selecting a wine can. These insights can help NY State winemakers avoid undesirable changes to their wines during can storage.

Project Results/next steps: Using both accelerated and ordinary storage conditions out to 32 weeks, we validated that molecular SO₂ (and not free SO₂) is the best predictor of formation of H₂S ("rotten egg" aroma). We also observed that BPA Epoxy and BPA-NI Epoxy deliver similarly good performance across can manufacturers, but acrylic yields unacceptably high H₂S formation over normal storage times. Canned wine producers should aim to keep molecular SO₂ low – preferably <0.5 mg/L – and use epoxy (BPA or BPA-NI) lined cans. Ongoing work is evaluating can consistency across manufacturers, and full results will be shared in trade and peer-reviewed publications.



Supporting attachments:

Supporting figure: H₂S production in canned wines is best predicted by initial molecular SO₂, not free SO₂. Five commercial wines (FR, PF, RB, SB, WB) were sourced, and pH and SO₂ adjusted to yield 15 wines for evaluation. Wines were stored at room temperature for four months in BPA-NI epoxy lined cans.