Examination of Pesticide Residues in Wine, Beer and Their Constituent Products Using High-Throughput Techniques to Maximize Extraction & Efficiency

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Abstract

Commercial red wine and beer samples were examined for their pesticide concentrations. In addition to the examination of the finished alcoholic beverage, the constituent agricultural products of wine and beer production; grains, malts, hops, and wine grapes, were also examined to determine the levels of pesticides found in those products. The sample preparation was extraction process efficiency and recovery were examined by processing samples using manual versus high-throughput techniques. The QuEChERS method was used to process a greater number of samples in a shorter period of time than the other extraction methods.

Methods & Materials

Samples:

The wine & component samples included:

- Malbec and Syrah grapes obtained from a commercial urban vintner
- Six red wines of the same varietals from South America, US, Europe, and Australia

The beer and component samples included:

- Two Briess grain samples (organic and non-organic)
- A dark malt sample
- Four hop samples (Cascade, Magnum, Centennial, and CitraHops)
- Six US craft beer samples representing the same hop varieties examined

Solid Sample Preparation:

- 2.5 g of solid samples (grain and hop samples) were ground using SPEX SamplePrep 6970 EFM Freezer/Mill®
- Grape samples were used whole and ground in the centrifuge tubes using the SPEX SamplePrep ShaQer® 1500 with ceramic mixing media

Liquid Sample Preparation:

- The alcohol content of each liquid material was noted in each sample for later calculations
- No additional sample preparation was conducted on liquid samples

Sample Extraction:

The AOAC 2007.1 methods for the extraction and clean-up of agricultural products using QuEChERS were followed.

Analytical Conditions:

- Instrument: Agilent GC/MS in scan mode with EIC (35-450 m/z)

Results & Conclusions

Wine and Wine Grapes (Figure 1):

Grapes: Imidacloprid was found in only the mechanical samples at a concentration of 0.9 ppm above spiked levels.

Wine: Carbaryl and Imidacloprid were found in levels above 2.8 ppm. Residues increased by 5-15% using mechanical over manual shaking.

Results & Conclusions (cont'd)

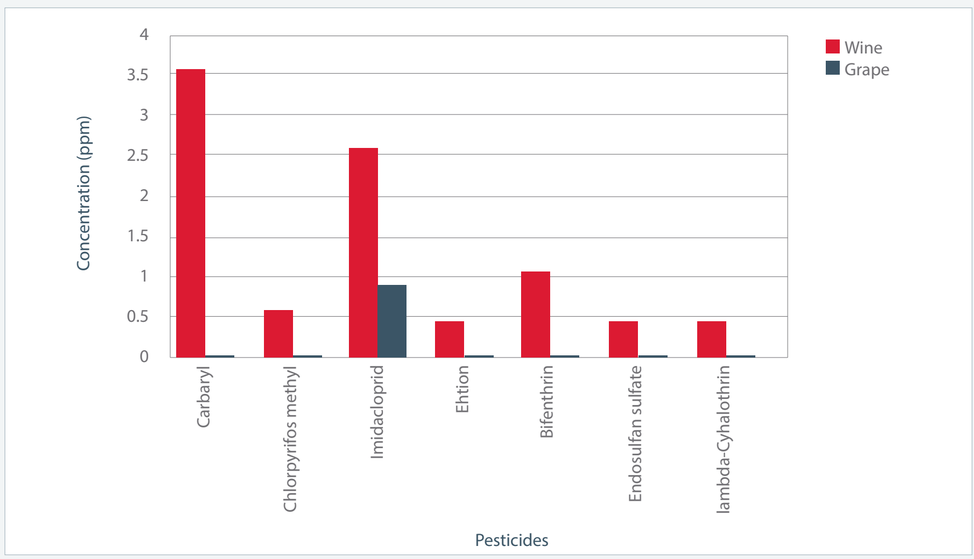

Figure 1. Pesticide Concentration (ppm) in Wine and Grape Samples.

Beer, Grain, Malt, and Hops (Figures 2, 4 and 5):

Grain: Pesticide residues were overall lower in organic vs. non-organic samples. However, Bifenthrin and Ethion were detected in both samples.

The use of mechanical shaking increased recovery in the grains by 10-25% over manual shaking methods.

Malt: Imidacloprid, Bifenthrin and Endosulfan Sulfate were all detectable above spiked concentrations.

Hops: Showed highest concentrations of all samples. Hops also contained numerous organic compounds (mainly terpenes) which interfered with the detection and quantitation of some pesticides. Additional clean-up procedures are needed to accurately ID and quantitate these pesticides.

The hops showed high levels of Ethion, Imidacloprid and Carbaryl.

Beer: Beer samples contained Carbaryl, Ethion and Imidacloprid. Mechanical shaking increased recoveries by up to 35% over manual shaking (Figure 3).

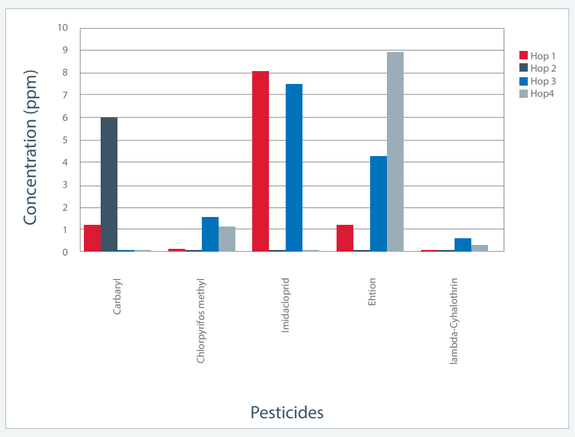

Figure 2. Pesticide Residue (ppm) in Hops.

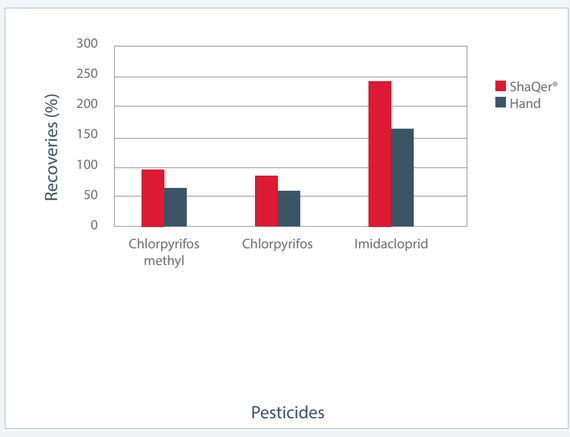

Figure 3. Comparison of Select Pesticide Residue Recovery (%) in Beer Mechanical Versus Hand-Shaken Techniques.

The beer samples contained detectable pesticide residue levels for Carbaryl (3 ppm), Ethion (2.5 ppm) and Imidacloprid (5.25 ppm). The use of the mechanical shaking techniques increased the pesticide recoveries in beer up to 35% over the manual shaking.

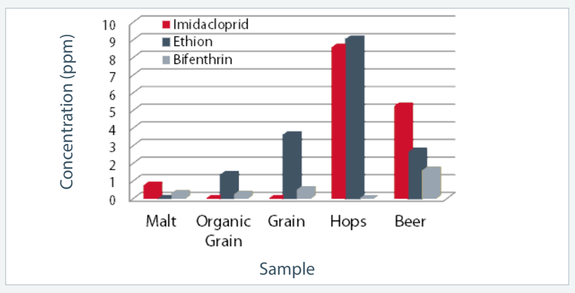

Figure 4. Pesticide Residue (ppm) in Beer & Components.

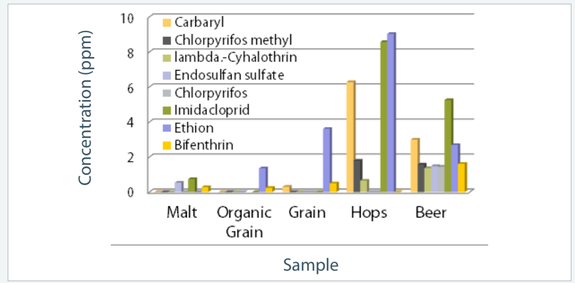

Figure 5. Pesticide Residue (ppm) in Beer and Components.

The highest pesticide levels detected were found in the beer hops with Imidacloprid detected at 8.5 ppm. The pesticides found in the beer components were also found in the finished beer products at up to 5 ppm (Imidacloprid) with most pesticides in the finished beer products being detected below 1-2 ppm. The use of mechanized shaking increased the recovery of the pesticides in all of the sample types from 5-35%.

The wine and wine grapes showed one consistent pesticide residue (Imidacloprid) up to 3 ppm in the finished wine. The wine grapes only showed a detectable pesticide residue level in samples that were mechanically shaken versus hand-shaken.

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