Comparative Analysis of VOCs and Flavor/Odor Contribution in Double-matured Whiskey from Dented Brick Distillery

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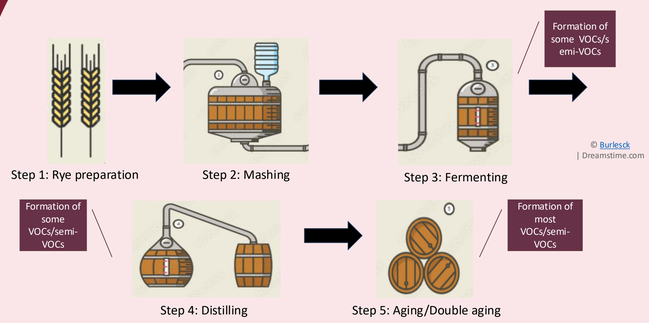
Poster # 558

Introduction



Dented Brick Distillery is a local Utah distillery, specializing in the production of a variety of spirits. The topic of research involves their rye American whiskey, aged for two years in American Oak barrels. The manufacture of said whiskey goes through multiple stages, as seen above, with the aging process resulting in the most volatile and semi-volatile organic compounds. These VOCs/semi-VOCs, produced during maturation when the spirits chemically react with the wood, enhance the overall flavor, aroma, and color. Double maturation, in which Dented Brick Distillery has lengthened the aging process in ex-Cabernet and Chardonnay barrels, can further enhance these components. To learn what VOCs/semi-VOCs are created, and what flavor/odor they contribute, analysis through GC-MS was implemented. Dilution through LLE was performed to compare with pure sample runs in the GC-MS. Multiple literature reviews were referenced regarding flavor/odor contribution.

References

(1) Wanikawa, A., Sugimoto, T. A Narrative review of Sulfur compounds in Whisk(e)y. *Molecules.* **2022**, 27, 1672.

(2) Caldeira, M., Rodriques, F., Perestrelo, R., Marques, J.C., Camara, J.S. Comparison of two extraction methods for evaluation of volatile constituents patterns in commercial whiskeys: Elucidation of the main odour-active compounds. *Talanta.* **2007**, 74, 78-90.

(3) Ling, M., Bai, X., Cui, D., Shi, Y., Duan, C., Lan, Y. An efficient methodology for modeling to predict wine aroma expression based on quantitative data of volatile compounds: A case study of oak barrel-aged red wines. *Food Research International*. **2023**, 164, 112440.

(4) Katsuki, Y. Analysis and Origin of characteristic aroma of whisky matured in ex-red wine casks. *Doctorate Thesis, Kyushu University.* **2021**

Acknowledgements

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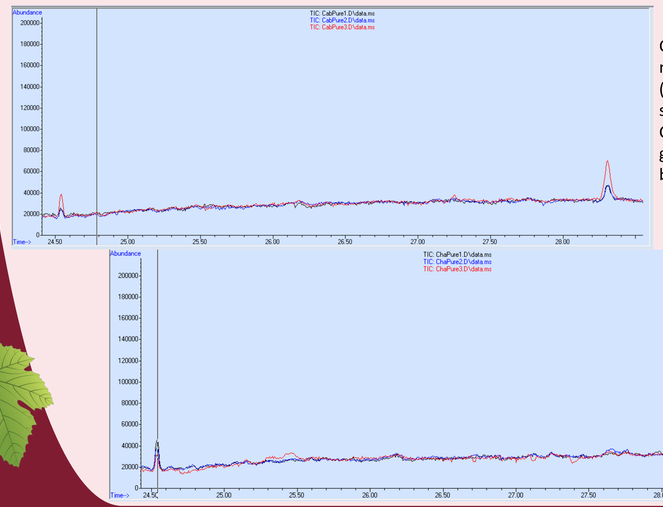
Results

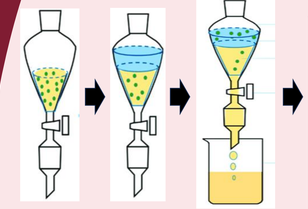
Compound Name for both Cabernet and Chardonnay samples	NIST Match	Sample Derivation
Water	Moderate-High	Both
Isoamyl Alcohol	High	Both
Acetic Acid	High	Both
Methanol	High	Both
Ethyl Acetate	High	Both
1-Propanol	Moderate-High	Both
Isobutyl Alcohol	High	Both
Furfural	Low-Moderate	Both
1-Butanol	High	Pure
Z-Oaklactone	Low-Moderate	Pure
Vanillin/Vanillin Lactoside	Low-Moderate	Pure
Syringaldehyde	Low-Moderate	Pure
Isobutyl Ethyl Ether	High	Diluted
1-Butyl Ethyl Ether	Moderate	Diluted
Methyl Isobutyrate	High	Diluted

Compound Name for both Cabernet and Chardonnay samples	Flavor/Odor Contribution	Abundance	Production Derivation
Water	Enhances and dampens certain notes	High	All stages
Isoamyl Alcohol	Banana	Moderate-High	Fermentation
Acetic Acid	Vinegar	Moderate-High	Fermentation and maturation
Methanol	Tasteless	Moderate-High	Fermentation
Ethyl Acetate	Fruity (low), nail polish (high)	Moderate-High	Fermentation
1-Propanol	Pungent, alcohol	Low-Moderate	Fermentation
Isobutyl Alcohol	Sweet, musty	Moderate-High	Fermentation
Furfural	Almond, caramel, toast	Low	Fermentation and maturation
1-Butanol	Pungent, harsh	Low-Moderate	Fermentation
Z-Oaklactone	Coconut, woody	Low	Maturation
Vanillin/Vanillin Lactoside	Vanilla	Low-Moderate	Maturation
Syringaldehyde	Spicy, smoky, and woody	Moderate	Maturation
Isobutyl Ethyl Ether	Most likely fruity, floral	Moderate-High	Most likely fermentation, distillation, and maturation
1-Butyl Ethyl Ether	Most likely fruity, floral	Low-Moderate	Most likely fermentation, distillation, and maturation
Methyl Isobutyrate	Fruity, sweet, apricot	Low-Moderate	Fermentation



Comparison of vanillin (~24.50 min) and syringaldehyde (~28.40 min) in Cabernet sample (top graph) and Chardonnay sample (bottom graph) with gradual column bleed.

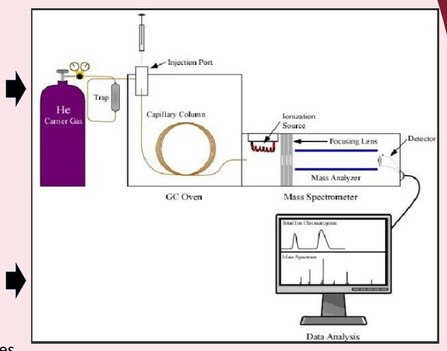
Methodology



Diluted samples through LLE (ResearchGate, 2021)

OR

Pure samples



GC-MS auto-injection (ResearchGate, 2011)

GC-MS configuration

Settings	Diluted values	Pure values
Inlet mode	Split, 25:1	Split, 25:1
Inlet temp (°C)	200	240
Pressure (psi)	16.08	16.08
Gas	He	He
Initial oven temp (°C); hold (min)	40; 5.00	40; 4.00
Ramp 1 (°C); next (°C); hold (min)	10.00; 240; 5.00	10.00; 210; 5.00

LLE method

LLE component	Chemical compound
Organic solvent	Ethyl Acetate
Aqueous solvent	Water
Aqueous density modifier	Saturated Sodium Chloride
Drying material	Anhydrous Sodium Sulfate

Conclusion

Based on current results, varying methodologies, from extraction techniques to GC-MS configurations, have resulted in multiple outcomes regarding VOC/semi-VOC identification. Different methodologies should be further explored to reveal which combination of set-up would divulge the most VOCs/semi-VOCs. Column configuration should be further refined, so as to reduce instrumental error, which has already affected results, such as the vanillin and syringaldehyde identification (see results section). Comparative analysis of single-matured whiskey might also be implemented to reveal varying abundances of double-matured compounds as suggested in literature review. Next steps include future experimentation and methodology refinement with SPME fibers and manual injection. As Dented Brick Distillery uses its own water source for distillation purposes, water qualities might also be considered and its effects on VOC/semi-VOC production.