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Profiling the shelf-life and shelf-stability of amber ale and IPA using GC-MS/O and trained sensory panel evaluation

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Abstract

Evolutions of flavor and aroma compounds present in two major beer brands. An amber ale and an IPA were tracked over a three-month period to develop a detailed aroma fingerprint of each beer and to determine the flavor stability and shelf life of the beers. The products were evaluated on a weekly basis by a trained sensory panel using a triangle test to identify aroma, flavor, and taste differences between samples stored at room temperature (20°C) and cold temperature (2°C). The triangle test determined that 41.6% of panelists could correctly identify differences in the amber ale and that beer stored at room temperature was most preferable. In comparison, 57.0 % of panelists could correctly identify differences in the IPA and that beer stored at cold temperature was most preferable. In addition the beer was analyzed via gas chromatography/mass spectrometry to identify the change in chemical composition of the beer throughout aging. From each beer 26 chemical compounds are being monitored throughout the aging process to identify changing chemical composition. It has thus far been determined that a synergistic effect among aromatic and non-aromatic constituents most likely contribute to sensory profile changes. An olfactory detection port connected to the GC will be used in future research to identify the prominent aroma compounds found in each beer during the aging.

Introduction

It is well understood that over time storage conditions and oxidation have a significant impact on flavor stability. For example, carbonyl compounds, such as Acetaldehyde, increase during the aging process and can produce the aroma of bruised apple, paint or raw pumpkin. Acetaldehyde has a flavor threshold of 25 mg/L in fresh beer. Acetaldehyde is formed from the oxidation of ethyl alcohol and the synthesis is most rapid at 40^{0}C in the presence of air[1]. Another carbonyl compound, trans-2-nonenal, is responsible for producing the cardboard off flavor in aged beer. Trans-2-nonenal is a result of lipid oxidation during the brewing process and has a flavor threshold of 0.11(µg/L) [1].

This change in beer over time is often associated with the degradation of quality. The organoleptic attributes of beverages is essential to know in order to increase quality and to understand production processes that could contribute to excess deterioration or flaws. This knowledge is relevant to all beverage industries whether it be wine, beer, or coffee. This analysis of beer aging is complex due to the fact that there are many chemical processes that occur to contribute to the overall aging of beer and the reactions involved are different for specific styles of beer[2]. A chemical fingerprint of beer could be used to represent beer quality and an efficient analytical method could be used to acquire this data.

The analysis of flavor and aroma compounds of beverage products has been widely studied. Most often this analysis is performed by gas chromatography coupled with mass spectrometry (GC/MS)[1]. The methods used to analyze the chemical composition of beer and other beverages often involve tedious sample preparation, which can be time consuming. Typically, solid phase microextraction (SPME), Headspace analysis, or a combination thereof, is used to isolate analytes of interest[3]. These procedures can be intensive and; thus, there is a need to streamline sample preparation methods in order reduce time and costs for research and industrial purposes.

The environment in which the beer is stored dictates much about how quality will evolve throughout the beer's life[2]. Thus, determining the relationship between how beer is stored and the chemical change that occurs during this time is crucial for producers to know in order to improve quality. The purpose of this study will to be to develop a stir bar sorptive extraction thermal desorption-gas chromatography-mass spectrometry coupled with olfactometry (SBSE-TD-GC-MS/O) method which will provide a simple and straightforward method for the brewing industry to use to identify a chemical fingerprint for beer. An IPA and an Amber Ale will be analyzed to determine the impact of storage temperature and duration on beer character.

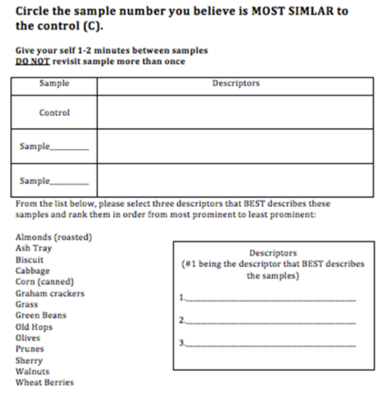
Methods

Sensory:

To develop an aroma fingerprint of hop- and a malt-driven beer, a descriptive panel developed a list of descriptors that best represent aroma attributes present in each beer. Panelists tasted an array of known products that best define the descriptor in question. For example if the panel says the beer is "corny" they must then compare different types of corn products (corn chips, canned corn, pop corn) until they find one that best matches the aroma. It is important that the list be comprehensive but not overlapping. The descriptive panel will meet weekly to reevaluate the samples and make corrections to the list.

Sensory evaluations will take place weekly. Panelists will be presented with a duo-trio test, for both a malt and hop driven beer, stored at either room temperature (25°C) or cold temperature (2°C).The duo-trio test presents the panelists with two coded samples, and one control sample, denoted by the letter "C". One of the coded samples is identical to the control sample and the panelists are asked to identify the sample most similar to the control. The purpose is to see if panelists can determine a significant difference between the two types of storage conditions. In addition panelists will be asked to rank the three most prevalent descriptors (set forth by descriptive panel) from highest to lowest as a method of tracking attributes over time.

Sample Sensory Evaluation Form



Circle the sample number you believe is MOST SIMLAR to the control (C).

Give your self 1-2 minutes between samples
DO NOT revisit sample more than once

Sample	Descriptors
Control	
Sample________	
Sample________	

From the list below, please select three descriptors that BEST describes these samples and rank them in order from most prominent to least prominent:

Almonds (roasted)
Ash Tray
Biscuit
Cabbage
Corn (canned)
Graham crackers
Grass
Green Beans
Old Hops
Olives
Prunes
Sherry
Walnuts
Wheat Berries

Descriptors
(#1 being the descriptor that BEST describes the samples)

1.________________
2.________________
3.________________

Instrumentation:

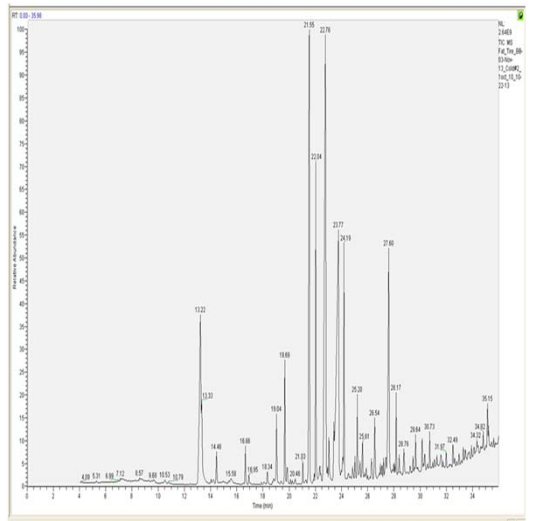
The samples were analyzed using a Thermo Scientific Trace GC Ultra gas chromatograph attached to a Thermo Scientific TSQ Quantum XLA mass analyzer. The carrier gas was helium and the column used was a RESTEK RTX-1, 60m, 100% dimethyl polysiloxane. The stir bars used were GERSTEL Twister EG silicone. The stir bars were desorbed with a GERSTEL Thermal Desorption Unit (TDU). The TDU had an initial temperature of 35°C and a final temperature of 190°C with a 2.00 minute hold time. The auto sampler used was a GERSTEL Multipurpose Sampler (MPS).The GC runtime was 40 minutes with an initial temperature of 40 minutes. The oven temperature was ramped up to 80°C and held for 1.00 minute and then ramped to 260°C in 7.00 minutes and held for 2.00 minutes before finally being ramped up to 280°C and held for 6:00 minutes. The mass spectrometer was set to full scan (Q1MS) with a first mass of 50.0 (m/z) and a final mass of 650.0 (m/z).

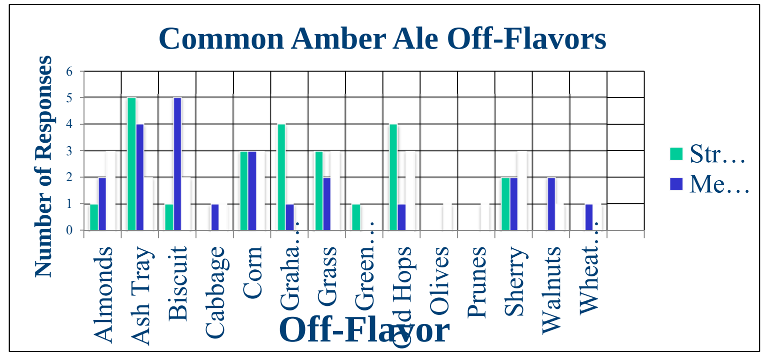
Experimental

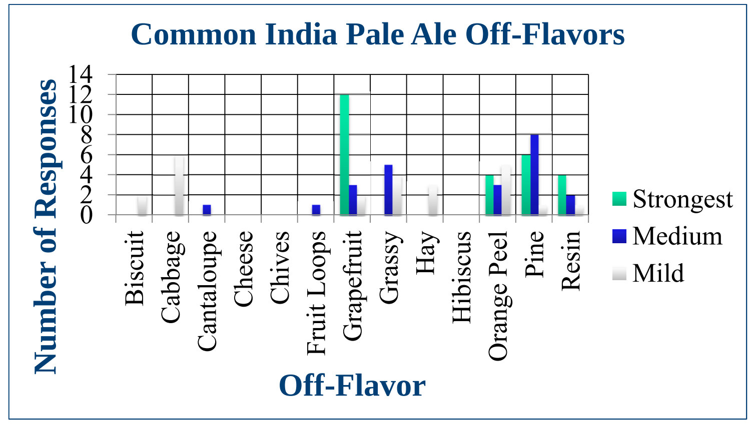
Three bottles of Amber ale and IPA beer samples were analyzed per sample to account for bottle variability. Each sample was degassed with a sonicator to remove carbonation. Aliquots of each bottle, 5 mL, were pipetted via micropipette, 100-1000 µL (± 1.6), (Eppendorf) into GC headspace vials. 10 µL of the internal standard, 1-octanol, was added to each sample. One stir bar was placed into the GC vial of beer and each vial was covered with parafilm to reduce the loss of volatile compounds. Each sample was stirred for one hour. The stir bar was removed from the sample, dabbed dry, rinsed with a few drops of DI water, and dabbed dry for a final time before being placed into the stir bar vials to be analyzed. The GERSTEL autosampler placed each stir bar vial into the TDU for analysis via stir bar sorptive extraction thermal desorption-gas chromatography-mass spectrometry coupled with olfactometry (SBSE-TD-GC-MS/O).

Results

Table 1; GC Chromatogram Results of beer analysis







Discussion and Conclusions

The panelist identified the two alike amber ale samples 41.6% of the time and 57.0% of the time for the IPA. The values are based on the sensory panel as a whole and do not take into account individual sensory limitations. Additionally, the strongest sensory attributes recognized by the panel for the amber ale were ashtray, graham crackers, and old hops. The strongest sensory attributes found in the IPA were grapefruit, pine, resin, and orange peel. Future work will be done to determine what chemical compounds are associated with these characteristics and at what concentration they are present in the samples.

From the GC/MS data 26 compounds were identified which may contribute to the changing sensory attributes of the beer. Preliminary results indicate that a synergistic effect between several compounds may contribute more to overall change during aging than the effect of individual compounds.

Acknowledgements

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