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Solid Phase Micro Extraction of Flavor Compounds in Beer

Application note - Food/Flavor

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Abstract

Solid Phase Micro Extraction (SPME) is a widely used sampling technique in the food analysis industry. SPME fibers are coated with a stationary phase that will extract analytes from the sample and onto the fiber for desorbtion and separation by Gas Chromatography (GC). The beauty of SPME is its simplicity. The SPME fiber is exposed to the sample headspace or solution and the stationary phase acts to soak up the analytes in the sample. Through use of different stationary phases, the SPME fiber can extract analytes of different polarities and volatilities by simply employing a stationary phase that best suits the needs of the application. Coupling SPME sampling with GC for separation and Mass Spectrometry (MS) for analysis provides an ideal method to discern flavor compounds. This paper will employ SPME in order to investigate the flavor composition of different beers providing an examination of what flavors beers have in common and what sets them apart.

Introduction:

The food industry is a very competitive business. A lot of work goes into the development of a new product. This statement is especially true for the beer industry. Since most of us have a favorite beer there is a different beer to satisfy each individual preference. There are light, citrus, regular and dark beers, and each type and/or brand has its own special combination in order to win over our taste buds.

Discussion:

Beer is basically composed of malt, hops, yeast and water. In order to distinguish one beer from another, different malts, hops and yeast can be used. A further way to differentiate one beer from another would be the addition of assorted essences such as fruity, ethereal, spicy, and the like. Brewing techniques can also help characterize the flavor of assorted beers. All of these practices and more are used in order to create the beer of choice.

Several different brands and flavors of beer were chosen for this investigation. Beers were evaluated for their various commonalities and differences. Solid Phase Micro Extraction (SPME) is an effective tool in capturing volatile organic compounds (VOCs) that contribute to the flavor of beer. In order to automate the process of sampling these compounds by SPME, the EST Analytical FLEX Autosampler was used.

Experimental:

The sampling system used for this study was the FLEX Autosampler. A Polydimethylsiloxane/Divinylbenzene (PDMS/DVB) SPME fiber was used for headspace sampling of the beer samples. The FLEX was coupled to an Agilent 7890 GC and 5975 MS analytical system while the GC was configured with a Restek Rxi-624 Sil MS 30m x 0.250mm x 1.4µm column. Tables 1 and 2 list the experimental parameters for the FLEX and the GC/MS respectively.

Autosampler	FLEX
General	
Method Type	SPME
Sample Incubate Agitate	
Incubation Temp.	40ºC
Incubation Time	1.0min
Agitation Speed	75%
Agitation Delay	0.0min
Agitation Duration	1.0min
Extraction	
Fiber Depth	70%
Sample Vial Depth	95%
Fiber Extraction Time	5.0min
Fiber Extraction Agitation	No
Desorbtion	
Fiber Guide Speed	10%
Fiber Guide Depth	50%
Fiber Insertion Speed	60%
Fiber Insertion Depth	90%
Fiber Desorbtion Time	5.0min

Table 1: FLEX Autosampler Experimental Parameters

GC/MS	Agilent 7890/5975
Inlet	Split/Splitless
Inlet Temp.	250ºC
Inlet Head Pressure	19.041 psi
Mode	Split
Injection Pulse Pressure	20psi for 5.0min.
Septum Purge Flow	Pulsed Splitless
Desorption	5min at 250ºC
Column	Rxi-624 Sil MS 30m x 0.25mm I.D. 1.4µm film thickness
Oven Temp. Program	35ºC hold for 5.0 min., ramp 5ºC/min. to 100ºC hold for 0min., ramp 3ºC/min. to 220 hold for 1min., total run time 59 min
Column Flow Rate	2.0ml/min.
Gas	Helium
Total Flow	32.536ml/min.
Source Temp.	230ºC
Quad Temp.	150ºC
MS Transfer Line Temp.	180ºC
Scan Range	m/z 25-500
Scans	5.76 scans/sec
Solvent Delay	0.7 min.

Table 2: GC/MS Experimental Parameters

Assorted beer samples were procured from the local market. Five milliliters of each beer sample was measured and poured into headspace vials and sealed. The sealed headspace vials were then placed in the FLEX Autosampler sample tray, and the FLEX ran the samples as described above. Each of the beer samples were run in triplicate in order to ensure reproducibility. F nally, samples were analyzed for flavor compounds. The predominant flavor compounds in the beer samples analyzed are listed in Table 3. The flavor comparison of light, regular and dark beer of brand A are displayed in Figure 1. Figure 2 contrasts the flavor components of a regular beer versus a citrus beer. Finally, Figure 3 compares three different brands of regular beer and their respective flavor components. hromatograms of the citrus beer and the beer brands A, B, and C are displayed in Figure 4.

Flavor Compound	Flavor
Ethanol	alcohol
Ethyl Acetate	ethereal, solvent like
isobutyl alcohol	alcohol
Isoamyl alcohol (fusel oil)	alcohol
amyl alcohol (fusel oil)	alcohol
isoamyl acetate	banana
amyl acetate	banana
ethyl caproate	fruity
Limonene	citrus
linalool	sweet/rosewood
phenylethyl alcohol	rose/rose oil
ethyl caprylate	fruity
phenethyl acetate	fruity
ethyl caprate	fruity, winy
lauric acid	floral

Table 3: Flavor Compounds in Beer

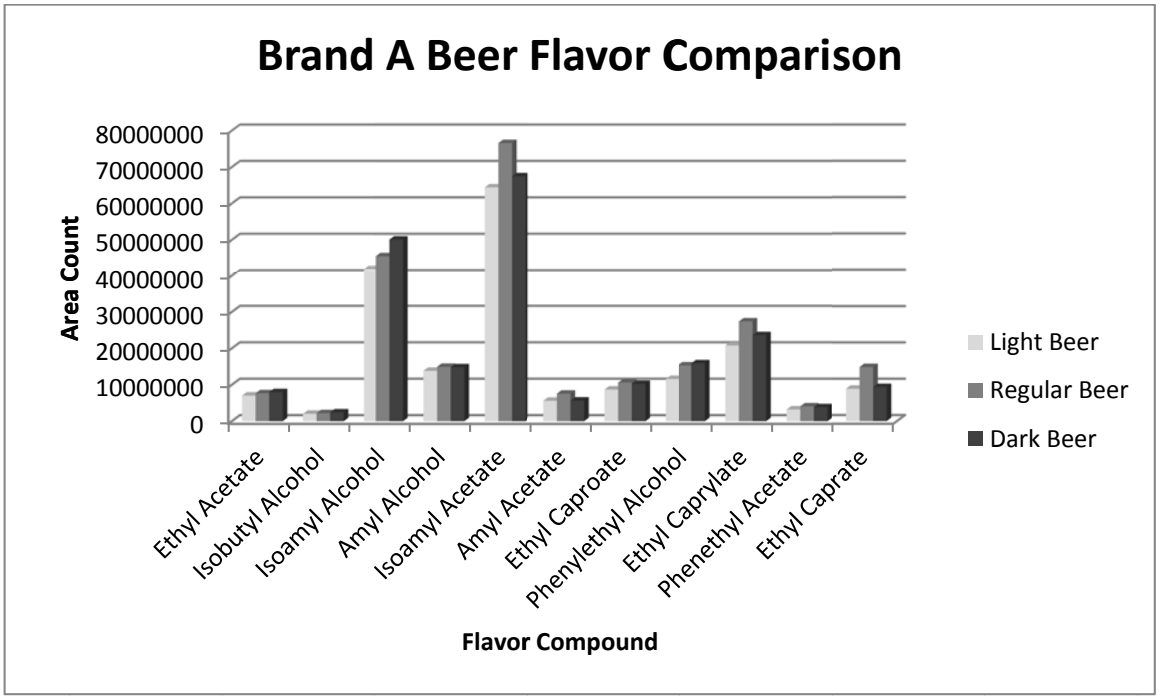

Figure 1: Flavor Compound Comparison of Light, Regular and Dark Beer

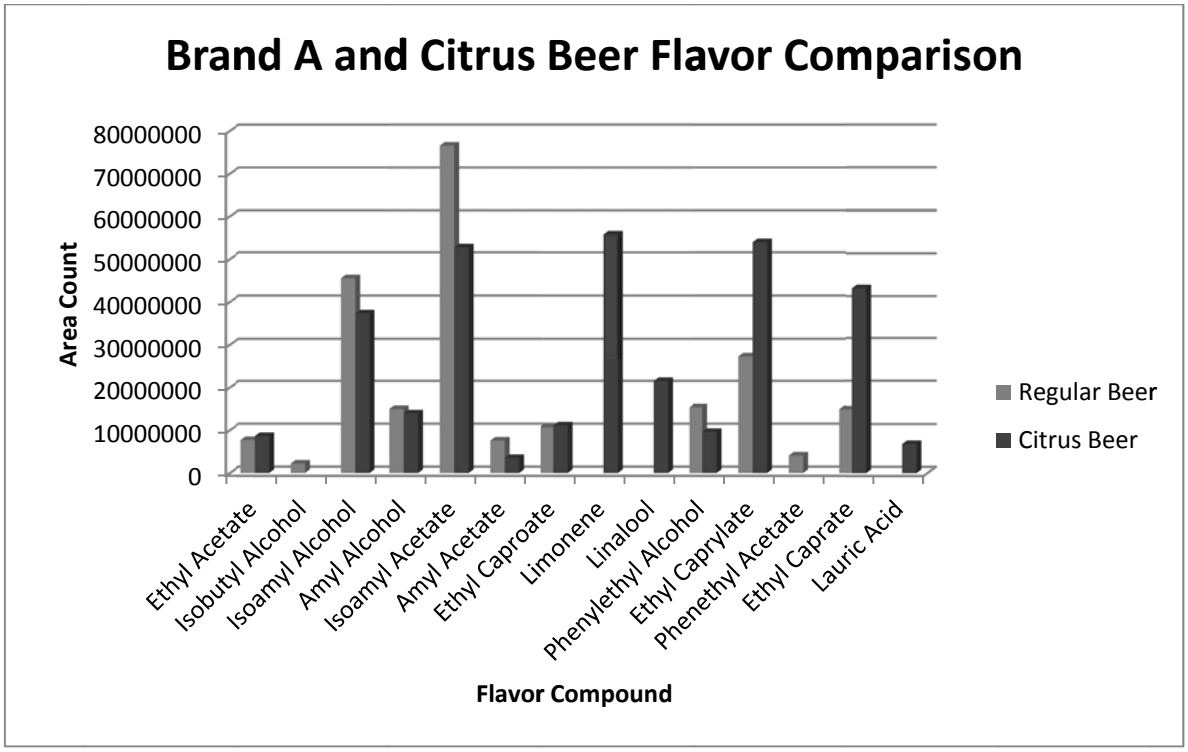

Figure 2: Flavor Compound Comparison of Regular Beer vs. Citrus Beer

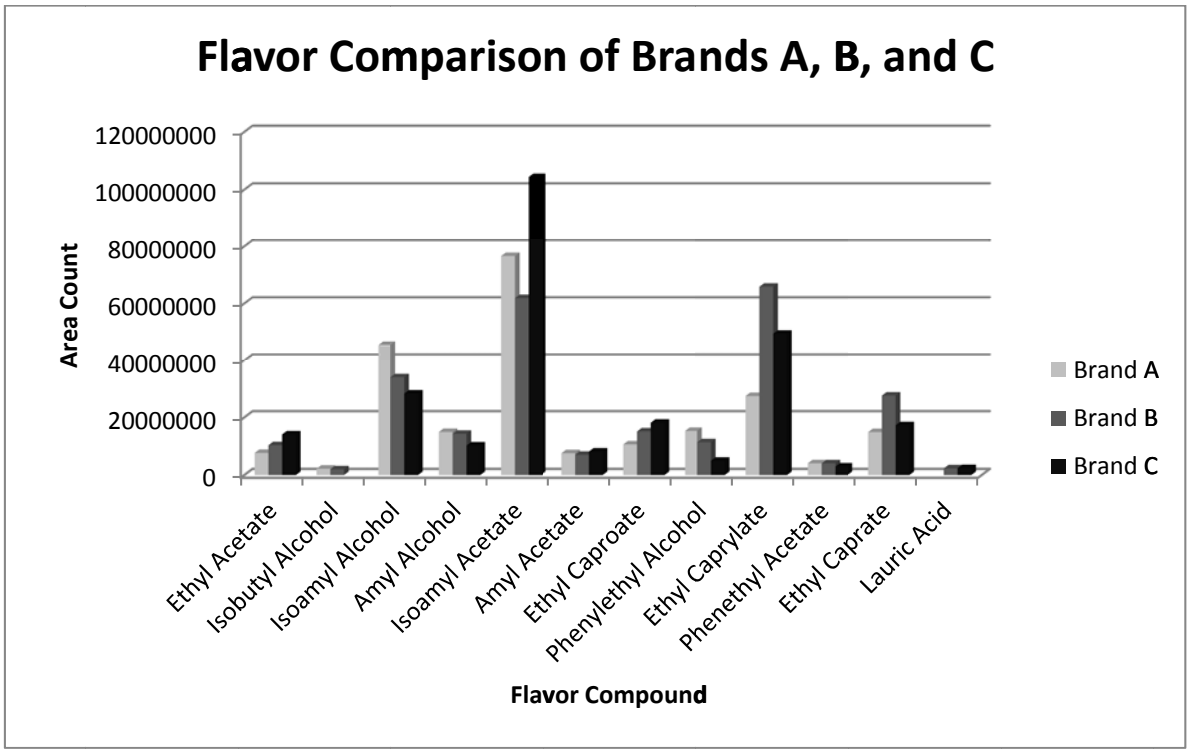

Figure 3: Flavor Comparison of Three Different Beer Brands

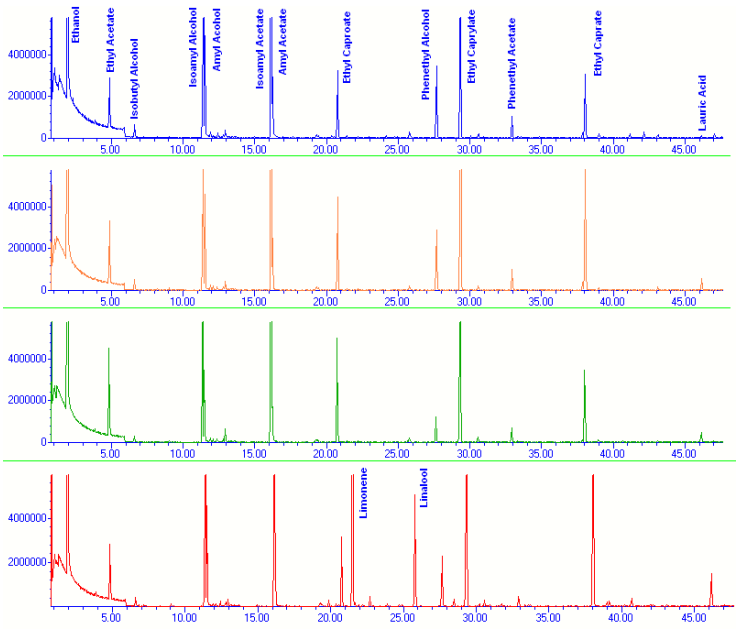

Figure 4: Chromatograms of Beer A (Blue), Beer B (Orange), Beer C (Green) and Citrus Beer (Red)

Conclusions:

In comparing all of the beer flavors and brands, it was clear that all of the brands and flavors had many flavor components in common. A single brand of beer was chosen for the regular, light and dark beer analysis, and the flavor profiles of these beers were very similar. However, when comparing regular beer to citrus beer, there was a distinct difference in that the citrus beer contained limonene and linalool where the regular beer did not. Furthermore, there were clear differences when comparing different brands of regular beer. Although the different brands contained many of the same flavor components, the amount of these components varied. The use of an autosampler enabled multiple samples of beer to be run and aided in sampling and result reproducibility.

References:

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