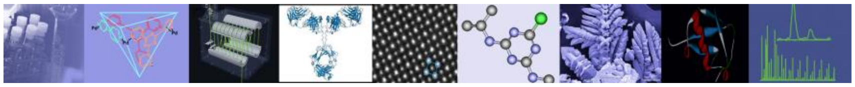


Analysis of Fermentation Gas from Home-Brewed Beer by Solid-Phase Microextraction (SPME) and GC/MS

Product: JMS-Q1500GC GC/MS System

Introduction

Fermenters used for home beer brewing are fitted with an airlock consisting of a liquid barrier that permits the fermentation gases to escape while preventing contamination from atmospheric microbes. Large volumes of carbon dioxide are produced during the most vigorous stages of fermentation, which can occur as early as the first 24 hour period after an ale yeast is added (pitched) to the sweet liquid (wort) produced from barley during the initial mashing step. The gases emitted from the airlock can have a pleasant aroma that can be quite distinctive during the initial stages of fermentation. Solid-phase microextraction (SPME) combined with gas chromatography/mass spectrometry (GC/MS) was used to determine the volatile components contained in the fermentation gas.

Experimental

Five gallons of beer were brewed using 11.5 pounds of 2-row malt and 1.5 pounds of debittered black malt. Homegrown Columbus, Chinook, and Centennial hops were added at different stages during the boil for bittering and aroma. Wyeast 1098 British Ale yeast was added to the cooled wort to initiate fermentation at a temperature of approximately 21.1°C (70°F).

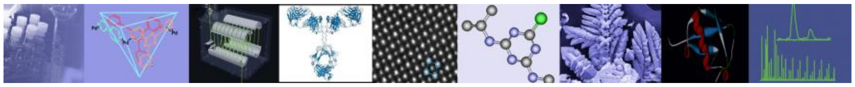
The fermentation gases were sampled by suspending a SPME Arrow above the airlock for 10 minutes during the vigorous fermentation that occurred within 24 hours after pitching the yeast. After sample collection, the SPME Arrow was manually injected into the GC inlet. Gas chromatography was carried out using an Agilent 7890 equipped with a Restek SPME Liner and a 30 m Restek Rxi®-5Sil MS column. Detailed GC conditions are shown in Table 1.

The JEOL Q1500 single quadrupole mass spectrometer was operated with the electron ionization (EI) source and an ionizing electron energy of 70 eV. Mass spectra were acquired over the range *m/z* 35 to *m/z* 500 and searched against the NIST17 Mass Spectral Database by using the JEOL data processing software. Database matches and retention times were used to confirm compound assignments.

Table 1. Measurement Conditions.

SPME	
SPME fiber	Restek PAL SPME Arrow (120µm DVB/Carbon-WR/PDMS, Cat # 27875)
Injector accessory	Manual Injection Kit (Restek, Cat#: 27490) SPME Arrow Conversion Kit (Restek, Cat#: 27493)
GC	
GC column	Rxi-5Sil MS (Restek, Cat#: 13623), 30m x 0.25mm, 0.25µm
GC inlet temp.	280˚C
GC inlet septum, liner	Thermolite Plus (Restek, Cat#: 23865) SPME Topaz liner (Restek, Cat#: 23280)
Inlet mode	Split 50:1
Carrier gas	He, 1 mL/min (Constant flow)
Oven temp.	40˚C (1min) → 10˚C/min → 300˚C (0 min)

MS	
Ion source temp.	250˚C
Interface temp.	250˚C
Ionization mode	EI+: 70 eV, 50µA
Scan step	+525V
Measurement mode	SCAN, *m/z* 35 – 500, 0.1 step
Acquisition rate	2.176 spectra/second
Mass Spectral Database	NIST17, version 2.3



Results

Figure 1 shows the aroma components detected in the fermenter gases. The major components are hop volatiles, while the fermentation byproducts ethyl acetate, isoamyl alcohol, and isoamyl acetate ("banana oil") are detected as smaller peaks are early elution times. β-myrcene is known to be the major contributor to hop aroma[1], comprising between 25% to 55% in the hop varieties used here[2]. β-myrcene is also the most abundant volatile component detected in the fermentation gases escaping through the airlock, with lesser contributions from the hop compounds β-pinene, γ-terpinene, limonene, sylvestrene, and humulene. Although ethanol is being formed it is not detected in the airlock aroma at this stage of fermentation, possibly being trapped in the airlock liquid barrier.

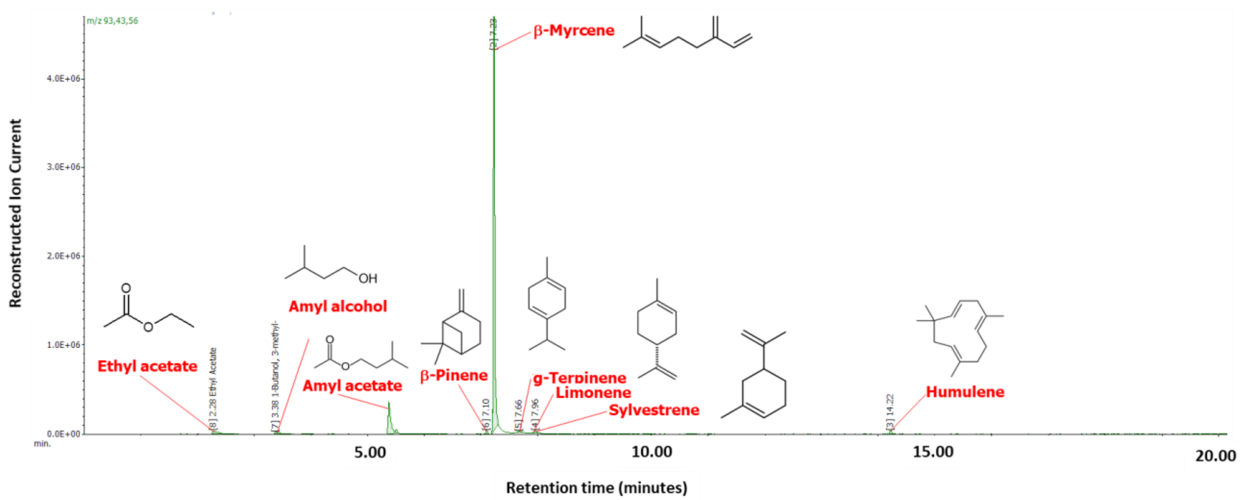


Figure 1. TICC.

Conclusion

The major contribution to the airlock aroma in the first 24 hours of ale fermentation is coming from hop volatiles, with fermentation byproducts present at lower levels. This supports a loss of hop volatiles during fermentation due to sparging by evolved carbon dioxide[3], which is the reason that post-fermentation addition of hops ("dry hopping") is commonly used to increase the aroma of hoppy beers such as India Pale Ales (IPAs).

References

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