Solid Phase Micro Extraction of Tea Flavor Components

Application Note

Food and Flavor

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Abstract

Tea flavors can vary from spicy to flowery to fruity and any combination thereof. Moreover, the flavor profile of tea can depend on where the tea leaves are grown, the brewing time and temperature, the processing of the tea leaves, and the type of leaf used. Using Head Space Solid Phase Micro Extraction (HS-SPME) sampling in conjunction with Gas Chromatography/Mass Spectrometry (GC/MS) for separation and analysis, assorted teas will be examined for their varied flavor components.

Introduction:

Green, Black and Jasmine Teas are all made from the same plant. They derive their flavor distinctions from how they are processed. Green tea leaves experience very little treatment while black tea leaves are oxidized before they are dried. This oxidation process causes the leaves to darken and produces a much stronger flavor. Jasmine tea, on the other hand, is green tea leaves scented with jasmine flowers.

In order to determine the differences in the three teas, the aroma compounds were extracted from the headspace of the brewed tea using Solid Phase Micro Extraction (SPME). The SPME fiber was then desorbed onto a column in a Gas Chromatograph (GC) for separation and analyzed using a Mass Spectrometer (MS). The resulting chromatograms were then analyzed in order to determine the flavor compounds of each respective tea and how they differed from one another.

Experimental:

The EST Analytical FLEX autosampler was used in order to automate the sampling process. A Divinylbenzene Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) SPME fiber with a 50/30μm film thickness was found to be the most efficient SPME fiber for this examination. The Shimadzu QP2010 SE GCMS was fitted with a SPME liner and a Restek Rxi-5 Sil MS column was used for separation and analysis.

Headspace SPME is a non-exhaustive sampling technique so the experimental conditions had to be optimized in order to make the extraction technique both efficient and reproducible. The FLEX suite software simplified the sample method development process with the ease of its drag and drop method builder. The autosampler and GCMS experimental conditions developed for this analysis are listed in Tables 1 and 2.

Autosampler	FLEX
General	
Method Type	SPME
GC Ready	Continue
GC Cycle Time	30min
Constant Heat Mode	Yes
Sample Incubate Agitate	
Incubation Temp.	80°C
Incubation Time	0.2min
Agitation Duration	0.0min
Extraction	
Fiber Guide Depth	45%
Sample Vial Fiber Depth	1cm
Extraction Time	10.1min
Fiber Extraction Agitate	Yes
Agitate Type	Oscillate
Agitate Duration	10.0min
Wait	
Wait on Input	Yes
Wait Input	GC Ready
Desorbtion	
Injection Port	A
Fiber Guide Speed	40%
Fiber Guide Depth	50%
Fiber Insertion Speed	75%
Fiber Insertion Depth	1cm
Fiber Desorbtion Time	2.0min
Injection Start Output	Start
Condition Fiber	
Fiber Temp	250°C
Condition Time	5.0min
Fiber Guide Speed	60%
Fiber Guide Depth	60%
Fiber Insertion Speed	20%
Fiber Insertion Depth	1cm

Table 1: FLEX Autosampler Experimental Parameters

GC/MS	Shimadzu QP 2010 SE
Inlet	Split/Splitless
Inlet Temp.	250°C
Inlet Head Pressure	51.6kPa
Mode	Splitless
Injection Pulse Pressure	100kPa for 2.0 min
Carrier Gas Split Ratio	10:1
Desorption	2.0min at 250°C
Column	Rxi-5 Sil MS 30.0m X 0.25mm X 0.25µm
Oven Temp. Program	45°C hold for 1.0 min., ramp 20°C/min to 275°C, hold for 1.5min, 14min run time
Column Flow Rate	1.0ml/min
Gas	Helium
Linear Velocity	36.2ml/min
Source Temp.	220°C
MS Transfer Line Temp.	220°C
Scan Range	m/z 35-500
Event Time	0.20sec
Solvent Delay	2.1min

Table 2: GC/MS Experimental Parameters

The tea leaves were acquired in China and each tea was prepared in the same manner. Two grams of the tea leaves were placed in 250 milliliters of de-ionized water at 80°C. The tea was allowed to steep in the heated water for five minutes. Ten milliliters of the prepared tea was then placed into a prepared 20 milliliter headspace vial. The headspace vials each had one half gram of sodium chloride in them in order to aid in analyte extraction. Five replicates of each tea were sampled and analyzed so as to verify reproducibility.

The results were analyzed in order to compare the relative response of the flavor compounds that the individual teas had in common. Table 3 and Figures 1 and 2 display the results of this analysis. Figures 3 through 5 presents labeled chromatograms of each tea in order to demonstrate the differences in the overall analyte response of each tea.

Compound	Odor	Black Tea	Green Tea	Jasmine Tea
hexanal	grass	2861326	5433817	
2-amino-6-methyl-benzoic acid	fruity, musty	4713330	2439315	3121718
benzaldehyde	fruity, almond cherry	1914082	98979	2726334
beta-myrcene	spicy	7401212		6131413
d-limonene	citrus	3261019		1666963
geraniol	floral	3049259	1537017	3952291
methyl salicylate	minty	6103515		41879957
methyl anthranilate	fruity	2389211		31610862
2,6-di-tert-butyl p-benzoquinone	new mown hay	6386643	4740502	4122720
trans-beta ionone	floral, woody	2983205		2937920
isopropyl myristate	faint oil	1115581	2251202	1248786
isopropyl palmitate	faint oil	1527085	2247817	2386125
linalool	citrus, floral	72921687	3045770	166582273

Table 3: Flavor Compound Responses

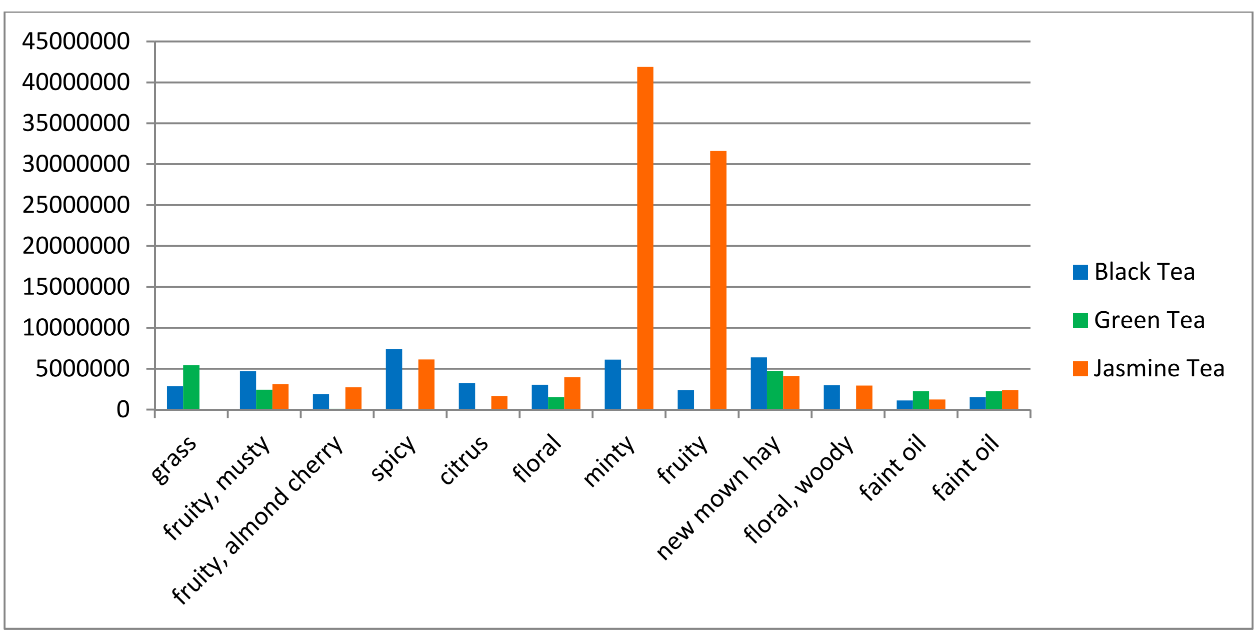

Figure 1: Diagram of Flavor Compound Comparison

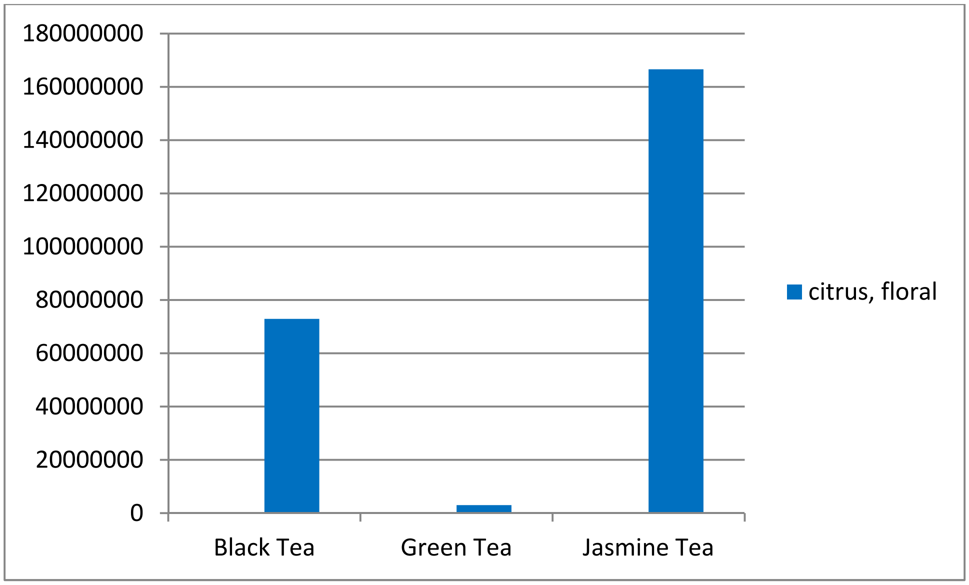

Figure 2: Difference in Linalool Response for Each Tea

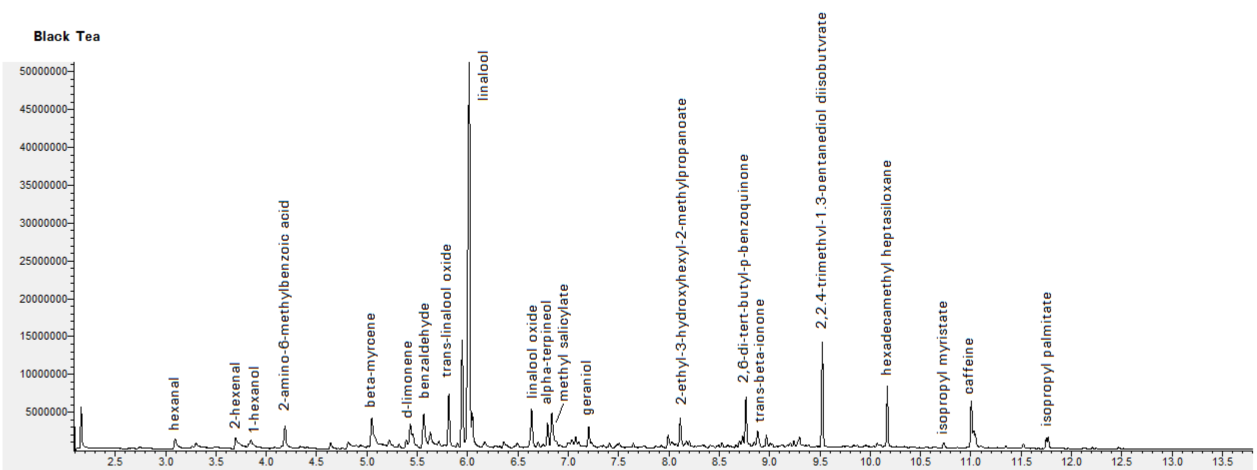

Figure 3: Chromatogram of Black Tea

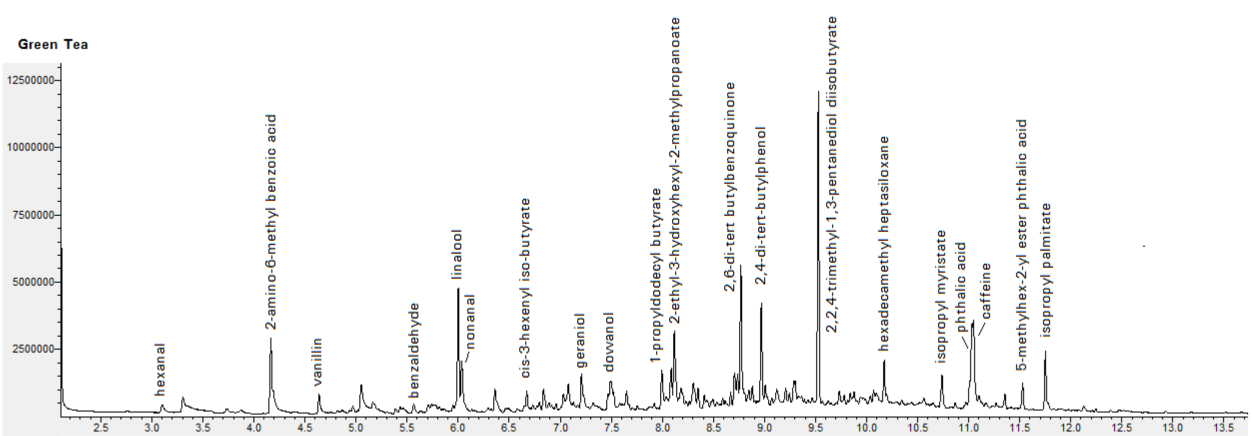

Figure 4: Chromatogram of Green Tea

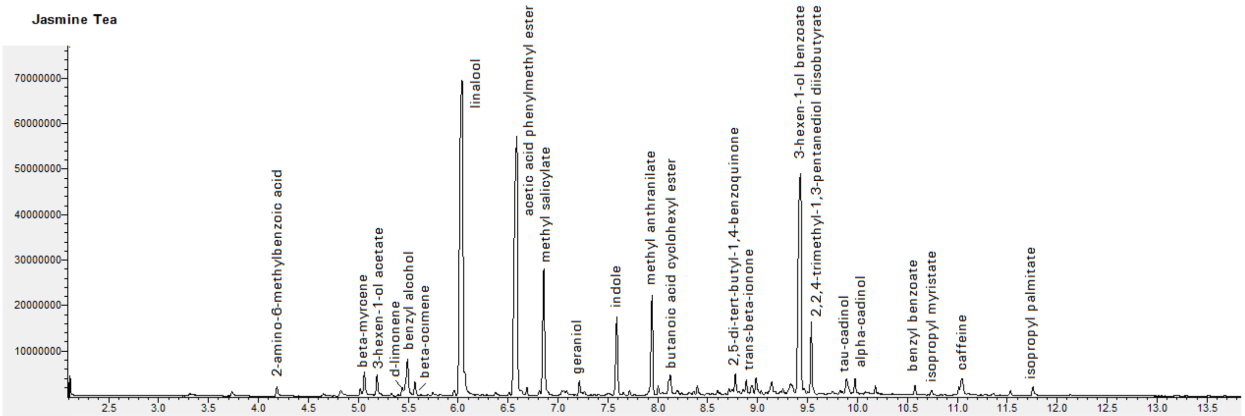

Figure 5: Chromatogram of Jasmine Tea

Conclusions:

Overall, the black and the jasmine teas had the most flavor compounds in common. Linalool, which gives tea a floral flavor, was common to all of the teas, however the jasmine tea had over twice the amount of linalool than the black tea and 50 times the amount in the green tea. The chromatography of each tea was quite unique from the others with green tea having the most complex chromatogram. The FLEX provided an excellent platform for the SPME sampling of the brewed teas. The FLEX method builder software enabled efficient method development and using the Shimadzu LabSolutions software the analysis of the flavor compounds in the tea was a straightforward process.

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