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Comparison of aroma character impact volatiles of Thummong leaves (*Litsea petiolata* Hook. f.), Mangdana water beetle (*Lethocerus indicus*) and a commercial product as flavoring agents in Thai traditional cooking

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TITLE RUNNING HEAD : Character impact volatiles in Thummong and Mangdana

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ABSTRACT

Thummong (*Litsea petiolata* Hook. f.) is a tree native to southern Thailand. The leaves of this tree are highly aromatic and used to flavor Thai dishes in place of the traditional water beetle Mangdana (*Lethocerus indicus*) for religious and cultural reasons. Total and aroma active volatiles from both flavoring materials were compared using GC-O and GC-MS. The volatiles from Thummong leaves and the Mangdana water beetle were collected and concentrated using headspace SPME. Twenty-three and twenty-five aroma active volatiles, were identified in Thummong leaves and Mangdana respectively. The major aroma active volatiles in Thummong leaves consisted of seven aldehydes, five ketones, and three esters. In contrast, the aroma active volatiles in the water beetle consisted of 11 aldehydes and three esters, and two ketones. Both had (*E*)-2-nonenal as the most intense aroma active volatile. The water beetle character impact volatile (*E*)-2-hexenyl acetate was absent in the leaves, but its aroma character was mimicked by 11-dodecen-2-one in the leaves which was absent in the beetle. In addition, a commercial Mangdana flavoring was examined using GC-O and GC-MS and found to contain only a single aroma active volatile, hexyl acetate. All three flavoring sources exhibited similar aroma characteristics but produced from profoundly different aroma active volatiles.

KEYWORDS: Thai food flavoring, plant volatiles, insect volatiles

INTRODUCTION

One of the natural flavoring sources in traditional Thai foods comes from the volatile compounds of a water beetle Mangdana (*Lethocerus indicus*). This edible insect is used for both flavoring and a source of protein. In Thailand, villagers in the rural northeast and north, use a local water beetle as both a food and a flavoring[1]. Only the male water beetle produces the desired aroma. The aroma from this beetle is an important flavoring material in Thai cuisine, especially in "nam prig" or chili sauce dishes. Previously (*E*)-2-hexenyl acetate was reported as the character impact compound in Mangdana water beetle[2]. This was subsequently confirmed by omission studies using an aroma reconstitution model[3].

Thummong (*Litsea petiolata* Hook. f.) is a tree native to southern Thailand. The leaves of this tree are highly aromatic and have been used in Thai cooking in place of the water beetle Mangdana as they have a similar sensory character. There is also a cultural/religious reason for this substitution as the population in southern Thailand is predominantly Muslim and insects are considered unclean. Both the leaf and the insect are popular, natural flavoring agents in Thai traditional dishes but they have limited availability in some areas and the beetle is only available during certain times of the year. A commercial artificial Mangdana flavoring has been produced to replace the natural sources. Since all three sources of flavoring are used for the same purpose in Thai traditional dishes there was a question if the similar sensory characteristics were the result of the same character impact aroma active volatiles. The purpose of this study was to compare and contrast the aroma active volatiles in these flavoring substances. The resulting GC-olfactory profiles (based on the grouping of aroma active volatiles with similar character) of both flavoring materials will be compared with each other as well as with the results from classical sensory profiling (quantitative descriptive analysis).

MATERIALS AND METHODS

Chemicals

Hexanal, octanal, 1-octen-3-one, nonanal, methional, (*E*)-2-nonenal, (*Z*)-2-nonenal, octanol, (*E*,*E*)-2,4-decadienal, decanal, (*E,Z*)-2,6-nonadienal, (*E,E*)-2,4-nonadienal, (*E,Z*)-2,4-decadienal, β-ionone, ethyl 2-methylbutanoate, (*Z*)-3-hexenal, ethyl pentanoate, (*E*)-2-hexenal, ethyl hexanoate, (*E*)-2-heptenal, 2-methyl-3-furanthiol, cis-rose oxide, (*E*)-2-heptenyl acetate, 1-hexanol, dimethyl trisulfide, (*E*)-2-hexenol, (*Z*)-3-hexen-1-ol, cis-linalool oxide, (*E,E*)-2,4-octadienal, butanoic acid, (*E*)-2-decenal, hexanoic acid, 2-hexenoic acid, and guaiacol were obtained from Sigma-Aldrich Co. LLC. R-(-)-carvone, β-damascenone, and acetaldehyde were gifts from Huangshan Kehong Bio-flavors Co. Ltd. China. Acetic acid was purchased from Fisher. 2-Acetyl-2-thiazoline, (*E*)-2-hexenyl acetate, (*E*)-2-hexenyl butyrate, and 2-undecanone were gifts from Givaudan (Thailand) Ltd. 11-Dodecen-2-one was not commercially available and was synthesized in the lab.

Synthesis of 11-dodecen-2-one

To a solution of undec-10-enal (3.0 mL, 15 mmol) in THF (15 mL) at 0˚C under inert nitrogen atmosphere, was added dropwise a solution of 3M methylmagnesium bromide in THF (5.5 mL, 16.5 mmol, 1.1 equiv.). The reaction was allowed to warm to room temperature. After 12 h, methanol (2 mL) was slowly added dropwise. Saturated aqueous NH_4Cl (20 mL) was added. The reaction was diluted with EtOAc (20 mL) and poured into a separatory funnel. The layers were separated and the aqueous layer was extracted with EtOAc (3 × 20 mL). The combined organic extracts were washed with brine (60 mL), dried over Na_2SO_4, and concentrated. The crude dodec-11-en-2-ol was used without further purification. To a solution of chromium (VI) oxide, **toxic!** (1.5 g, 15 mmol, 1 equiv.) in 4 M sulfuric acid (95 mL) at 0˚C, was added a solution of crude dodec-11-en-2-ol in acetone (15 mL) dropwise over 15 min. The reaction mixture was allowed to warm to room temperature. After 6 h, the reaction mixture was diluted with EtOAc (100 mL) and poured into a separatory funnel. The layers were separated and the aqueous layer was extracted with EtOAc (3 × 80 mL). The combined organic extracts were washed with brine (200 mL), dried over Na_2SO_4, and concentrated. Flash column chromatography (5% EtOAc in hexane) gave the desired product as colorless oil (1.85 g, 68% over two steps). The observed NMR spectra of the synthesized dodec-11-en-2-one consisted of: ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.80 (dd, 1H, J = 10, 17 Hz); 4.90−5.00 (m, 2H); 2.41 (t, 2H, J = 7.5 Hz); 2.13 (s, 3H); 1.90−2.10 (m, 2H); 1.54−1.59 (m, 2H); 1.25−1.38 (m, 10H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.3, 139.2, 114.1, 43.8, 33.8, 29.8, 29.3, 29.2, 29.1, 29.0, 28.9, 23.8. The observed spectral data matched that of the published NMR spectra of dodec-11-en-2-one[4]. ^{1}H NMR ($CDCl_3$): d = 1.19–1.56 (m, 10 H), 1.95–2.42 (m, 7 H), 2.38 (t, 2 H, J = 7.3 Hz), 4.87–5.00 (m, 2 H), 5.67–5.87 (m, 1 H). ^{13}C NMR ($CDCl_3$): d = 23.8, 28.8, 29.0, 29.1, 29.2, 29.3, 29.8, 33.7, 43.7, 114.0, 139.0, 209.2.

Sample and Headspace Sampling

Thummong leaves were cut into 0.5 cm^2 small pieces within three days of being harvested. One gram of cut leaves was placed into a 40 mL glass vial (La-Pha-Pack, Germany). The vial headspace was purged with nitrogen before sealing with a Teflon-coated septum. The sample was equilibrated at 40 °C for 15 min in a water bath and a 2 cm 50/30 µm divinylbenzene/Carboxen/-polydimethylsiloxane (DVB/Carboxen/PDMS) Stable Flex fiber (Supelco, Bellefonte, PA) was exposed to the leaf headspace volatiles for 30 min. The sorbed leaf volatiles were introduced into the GC injector set at 200 °C. Fresh frozen water beetle Mangdana was purchased from a local market. The sample preparation of the beetle was the same as the leaf except using only five grams of the cut up water bug. A commercial liquid Mangdana flavoring was purchased from a local supermarket in Bangkok, Thailand and a100 µL aliquot analysed in the same manner as the leaf and bug.

Gas Chromatography-FID/Olfactometry

Volatiles were separated and evaluated using an Agilent GC 7890 equipped with a sniffing port. Samples were run separately on both a polar (DB-wax, J&W Scientific, Folsom, CA; 30 m. × 0.32 mm. i.d. × 0.5 μm film thickness) and a 5% phenyl, 95% dimethyl-polysiloxane nonpolar column (Zebron ZB-5, Phenomenex, Torrance, CA; 30 m. × 0.32 mm. i.d. × 0.5 μm film thickness,). Oven temperature was programmed from 40 to 220 °C at 7 °C/min for both columns. Helium was used as carrier gas at a flow rate of 1.5 mL/min. Injector and detector temperature were 200 °C and 250 °C, respectively. A 0.75 mm. injector liner was employed to improve peak shape and chromatographic efficiency. Injections were splitless. The column effluent was split, 1/3rd of the flow was conducted to the FID and the other 2/3rds to the olfactory port for sniffing with previously mixed humid air. Two assessors, trained in a similar way to Dreher and coworkers[5], evaluated each sample in triplicate on both ZB-5 and DB-Wax columns. Odor descriptors and retention times were recorded for every sample. Assessors rated odor intensity continuously throughout the chromatographic separation process using a linear potentiometer as previously described[6]. Intensities of all odor-active compounds of each GC-O run were normalized so the highest intensity from each assessor was given a score of 10. The normalized intensities of all the runs were then averaged. A peak was considered odor-active only if at least half of the panel responses found a similar odor quality at the same retention time. Identification of volatiles was determined by comparing standard Linear Retention Index (LRI) values from both FID and MS data from samples with those from authentic standards. MS fragmentation patterns were also used to aid identification when available. Identifications were confirmed by comparing odor quality of standards and unknowns at the same retention time. Since the primary goal of this study was to determine character impact volatiles, the aroma character of the unseparated SPME extract was compared to that of the initial samples. The sorbed SPME volatiles were introduced to GC injection port and carried to the sniffer using a short (1 m.) fused silica column to confirm the similarity to the original sample.

Mass Spectrometry

GC-MS was employed to confirm the identities of the odor-active volatiles identified in the GC-O experiments. Headspace SPME volatiles were separated and analyzed using an Agilent GC 7890 quadrupole mass spectrometer and a DB-Wax capillary column (30 m. × 0.25 mm. i.d. × 0.50 µm film thickness). The carrier gas was helium in the constant flow mode of 2 mL/min. The source was set at 200 °C, the transfer line was maintained at 260 °C, and the injector was at 200 °C. The oven temperature program consisted of a linear gradient from 40 to 220 °C at 7 °C/min with a 2 min final hold. Electron ionization in the positive ion mode was used (70 eV), either scanning a mass range from *m/z* 25 to 300 or acquiring data in the selected ion mode. Mass spectra matches were made by comparison with NIST 2005 version 2.0 standard spectra (NIST, Gaithersburg, MD). Only those compounds with spectral fit values ≥ 800 were considered positive identifications. Authentic standards were also used to confirm identifications whenever available.

Sensory Aroma Profile Analysis

The aroma attributes for Thommong leaf and Mangdana beetle were evaluated using panels from Thailand who have had life-long experiences with both samples. One gram of chopped leaf and 3 grams of Mangdana beetle were separately presented to the panel in 40 ml screw cap glass vials at room temperature. The panel consisted of fifteen experienced panelists (age 22-50 years, 13 females and 2 males) and employed consensus sensory description. The eight consensus attributes consisted of: “sweet herbaceous”, “metallic”, “green”, “sweet”, “cheesy”, “creamy”, “cooked” and “savory”. Eight reference solutions for each of the aroma attributes were prepared at 50-fold greater than their odor thresholds in water: 2-hexenyl acetate (sweet herbaceous), (*E*)-2-nonenal (metallic), hexenal (green), vanillin (sweet), butanoic acid (cheesy), cream milk powder (creamy), methional (cooked) and chicken stock (savory). The panelists were asked to rate the intensity of eight attributes in each sample on a scale from 0 to 5 (0 stands for not perceivable and 5 for very high intensity).

RESULTS AND DISCUSSION

GC-O Aroma Active Volatiles

As shown in Table 1, a total of 23 aroma active volatiles were observed in Thummong leaves and 25 aroma volatiles in the Mangdana water beetle using GC-Olfactometry, GC-O. Although a total of 43 aroma active volatiles were observed from both samples, only five volatiles were common to both samples. Aldehydes constituted the major aroma active functional group (17) listed in Table 1, followed by equal numbers of esters, alcohols, and ketones (six each).

The overall intense, sweet, herbaceous character of both samples was due to completely different volatiles. In the case of Thummong leaves the intense herbaceous aroma was due to 11-dodecen-2-one, but in the case of the Mangdana water beetle, the same aroma character was produced by (*E*)-2-hexenyl acetate. Both of these character impact volatiles were confirmed by matching sensory descriptors using GC-O as well as matching retention times of standards as well as corresponding MS spectral matches. A commercial source of (*E*)-2-hexenyl acetate was used as a standard but 11-dodecen-2-one had to be synthesised[4] as no commercial source was available. Both compounds were described as intensely sweet herbaceous which is surprising as they differ profoundly in terms of general structure and functional group (ester versus a ketone). The character impact volatile in Mangdana water beetle has been previously reported as (*E*)-2-hexenyl acetate[2] and was later confirmed as the only character impact volatile in omission studies of an aroma reconstitute model[3]. This volatile also occurs naturally in many fruits such as strawberry, and yellow passion fruit[7,8].

The five aroma volatiles common to both samples consisted of (*Z*)-3-hexenal, methional, (*E*)-2-nonenal, guaiacol, and β-ionone. Both samples had a strong metallic/fatty aroma component due to (*E*)-2-nonenal. The lower molecular weight (*Z*)-3-hexenal produced a stronger (8 versus 2 intensity) green aroma in the water beetle than the leaf. The remaining three common aroma volatiles, methional, guaiacol and β-ionone where all about twice as intense in the water beetle as in the leaf. In fact, the sum of all the aroma active intensities in the water beetle were almost twice as high as the total intensities from the leaves (176 versus 97).

As seen in Table 1, the functional group composition was somewhat different between the leaf and the beetle. The leaves were characterized by fewer aldehydes (seven versus eleven) and more ketones (five versus two). Both samples contained the same number of alcohols and esters (three and three). The water beetle contained three volatile acids whereas the leaf contained none.

In an attempt to compare the overall aroma impressions of the Thummong leaf and the Mangdana water bug using GC-O data, the individual aroma attributes and their corresponding aroma intensities were classified into eight categories. Those aroma volatiles with similar sensory descriptors were placed in the same group. The categories consisted of: fatty/waxy, metallic, green, sweet/herbaceous, cheesy, fruity/floral, cooked, and meaty/sulfur. These aroma quality groupings were chosen to match as closely as possible to the consensus aroma descriptors developed in a parallel sensory study which will be discussed later. These GC-O category results are shown in the radar or spider web graphics in Figure 2A.

Both samples were characterized by having strong fatty/waxy, metallic, green, and fruity/floral characteristics as determined by summing the aroma intensities in each category. Both character impact compounds were major contributors to the sweet herbaceous category and accordingly rated either 9 or 10. However, the relative contribution of the character impact compound was greater in the leaf because it contained less aroma active volatiles in other categories. Both samples contained similar total aroma intensities in the metallic, green, and cooked aroma categories. The water beetle exhibited greater fatty/waxy and cheesy total intensities compared to the leaf. On the other hand the leaf had stronger fruity/floral and meaty/sulfur relative intensities. Overall, the summarized aroma category profiles were reasonably similar for both products.

Sensory Panel Aroma Descriptive Analysis

Average sensory panel descriptive analysis scores for the eight consensus attributes are shown in Figure 2B. There were similar panel scores for only two attributes, sweet herbaceous and sweet. The similar intense sweet herbaceous scores were probably due to the respective character impact volatiles observed in the GC-O studies. The panel indicated that the leaf contained strong green, sweet herbaceous, and metallic aroma attributes. However, the leaf exhibited very weak creamy, cooked, and savory scores. In contrast, the water beetle possessed strong aroma intensities in these same categories, namely; cooked, savory, creamy, and cheesy attributes. These scores on the left hand side of the spider web diagram are so profoundly different between the two samples, suggesting that the panel found these two flavor sources to be different even though the major character sensory attribute was very strong in both cases.

In comparing the GC-O patterns with the sensory patterns (Figures 2A and 2B), it is important to keep in mind that the aroma values are evaluated in very different ways. In GC-O, aromas are evaluated individually whereas sensory analysis evaluates mixtures of aroma compounds. It is probably the reason why some of the sensory panel aroma attributes such as “creamy” or “savory” were not observed in the GC-O study. These attributes were probably not due to a single volatile, but rather combinations of aroma volatiles. Therefore, where there is agreement of GC-O summary data with such sensory attributes as “green”, “metallic”, and “sweet herbaceous”, it is because these sensory attributes are largely due to single components rather than mixtures.

GC-MS Chromatograms

GC-MS was carried out to evaluate all of the major volatiles in the three samples and to confirm the identities of the character impact compounds in the three samples tentatively identified in the GC-O studies. The total ion chromatograms, TIC, of the three samples are shown in Figure 3 with major peaks identified in the figure caption. It can be seen from the relative peak heights that the character impact volatiles for both Thummong leaves (11-dodecen-2-one) and Mangdana water beetle (E-2-hexenyl acetate) were also the dominant peaks. They represent a rare example where the character impact volatile is also the single largest peak in the chromatogram (Fig. 3). The fragmentation patterns of the character impact volatile from Mangdana (A), (*E*)-2-hexenyl acetate, Thummong leaf (B) matched both those in the spectral library and standards, thus confirming their identifications. They have similar smells but are produced from totally different chemical compounds. There are limited reports of the aroma active volatile, 11-dodecen-2-one, but it has been reported as a specific ketone volatile in Changyu XO (a Chinese brandy) comparable to Hennessy XO (a well-known French liquor)[9].

Commercial Mangdana flavoring

A commercial Mangdana flavoring was also evaluated using GC-O and GC-MS. In the case of GC-O, only a single aroma active peak was observed at a retention time different than either of the other two character impact volatiles. The TIC chromatogram shown in Figure 3C is also very simple, consisting of essentially one peak. The fragmentation pattern of this peak matched almost perfectly with hexenyl acetate. Standard hexenyl acetate produced the same GC-O retention time and sensory response. Thus the commercial product is not natural nor nature identical. From a chemical point of view it is interesting in that it uses a chemical analogue of the water beetle character impact volatile, (*E*)-2-hexenyl acetate, without the double bond which is probably a more stable and/or a less expensive material.

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FIGURE CAPTIONS

Figure 1 Synthesis of 11-dodecen-2-one

Figure 2 **(A)** Spider graph of GC-O aroma active compounds from Table 1 which have been grouped according to similar odor. The group values are graphed as percent of total intensity from each product for each category. Mangdana beetle (---) and Thummong leaves (**—**) **(B)** Average sensory panel Quantitative Descriptive values comparing scores from Mangdana beetle (---) and Thummong leaves (-).

Figure 3 Comparison of the character impact volatile from Mangdana beetle (A), Thummong leaves (B) and commercial Mangdana flavoring (C). a = hexanal, b = (*E*)-2-hexenal, c = **(*E*)-2-hexenyl acetate**, d = (*E*)-2-hexenol, e = (*E*)-2-hexenyl butyrate, f = 2-hexenoic acid, g = (*E*)-2-heptenal, h = *cis*-rose oxide, i = nonanal, j = (*E*)-2-nonenal, k = 2-undecanone, l = **11-dodecen-2-one**, m = carvone, n = (*E,Z*)-2,4-decadienal, o = **hexyl acetate**

Table 1 Aroma active volatiles in Mangdana water beetle and Thummong leaves

	Identification	LRI		MS	Odor Descriptors	Intensity		Aroma Group
		Wax	DB-5	Ident.		Bug	Leaf	
1	Acetaldehyde	718	539	TIC	solvent like		2	
2	ethyl 2-methylbutanoate	1063	847	TIC	fruity		2	F
3	Hexanal	1076	795	TIC	green	6		C
4	(*Z*)-3-hexenal	1128	795	EIC	green	8	2	C
5	ethyl pentanoate	1142	899	TIC	fruity		1	F
6	(*E*)-2-hexenal	1215	855	EIC	green, metallic, sweet	9		C
7	ethyl hexanoate	1244	998	TIC	fruity		2	F
8	Octanal	1279	982	EIC	citrusy	8		F
9	(*E*)-2-heptenal	1284	954	TIC	green		2	C
10	1-octen-3-one	1292	977	EIC	mushroom	6		B
11	(*E*)-2-hexenyl acetate	1323	1020	TIC	sweet herbaceous	9		D
12	2-methyl-3-furanthiol*	1326	861	*	cooked, meaty		4	G
13	cis-rose oxide	1363	1157	TIC	floral		3	F
14	(*E*)-2-heptenyl acetate	1366		TIC	green, fatty	8		C
15	1-hexanol	1393	865	TIC	green, leaf		7	C
16	dimethy trisulfide*	1400	985	*	sulfury, cabbage-like		10	H
17	(*E*)-2-hexen-1-ol	1403	880	TIC	green, leafy	6		C
18	(*Z*)-3-hexen-1-ol	1407	847	TIC	fresh, green, grassy		3	C
19	nonanal	1410	1100	TIC	green, soapy		1	C
20	acetic acid	1439	680	TIC	vinegar	10		E
21	methional*	1442	909	*	cooked potato	8	4	G
22	cis-linalool oxide	1467	1214	TIC	floral, green		4	F
23	(*E*)-2-hexenyl butanoate	1471	1193	TIC	floral	5		F
24	Decanal	1501	1201	TIC	fresh mint, citrusy	5		C
25	(*Z*)-2-nonenal	1519	1149	EIC	fatty, metallic, geranium	9		A
26	(*E*)-2-nonenal	1529	1161	TIC	metallic, fatty	10	10	B
27	1-octanol	1578	1078	EIC	fatty	7		A
28	(*E,Z*)-2,6-nonadienal	1594	1161	TIC	green, metallic		5	C
29	(*E,E*)-2,4-octadienal	1607	1110	EIC	fatty	4		A
30	butanoic acid	1617	1609	TIC	cheesy, sweaty	8		E
31	2-undecanone	1618	1300	TIC	waxy, fatty sweet		9	A
32	(*E*)-2-decenal	1665	1250	EIC	green, fatty	5		C
33	11-dodecen-2-one	1676	1287	TIC	sweet herbaceous		10	D
34	(*E,E*)-2,4-nonadienal	1710	1220	EIC	fatty	5		A
35	R-(-)-carvone	1745	1245	TIC	minty		2	C
36	2-acetyl-2-thiazoline*	1750	1109	*	cooked jasmine rice	6		G
37	(*E,Z*)-2,4-decadienal	1796	1313	TIC	fatty, green		4	A
38	(*E,E*)-2,4-decadienal	1806	1284	EIC	fatty , cooked grain	4		A
39	hexanoic acid	1837	1085	TIC	sweaty, cheesy	9		E
40	Guaiacol	1851	1091	EIC	smoke, medicine	4	2	G
41	β-damascenone	1855	1395	EIC	sweet honey		3	D
42	β-ionone	1934	1496	EIC	raspberry, floral	9	5	F
43	2-hexenoic acid	1958	1614	EIC	musty, fatty, sweaty	8		A

Volatile compounds identified by matching: retention characteristics on both wax and DB-5 columns, their sensory aroma characteristics, and their MS spectra with those of authentic standards. TIC = full spectrum identification match, EIC = extraction ion chromatograms retention time match using a single base ion. * Tentatively identified on the basis of aroma descriptors and retention time matches with literature values. Aroma group categories: A = fatty/waxy, B = metallic, C = green, D = sweet/herbaceous, E = cheesy, F = fruity/floral, G = cooked, H = meaty/sulfur.

Figure 1.

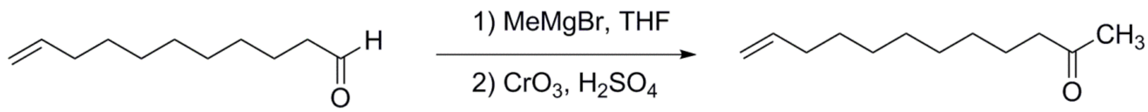


Figure 2.

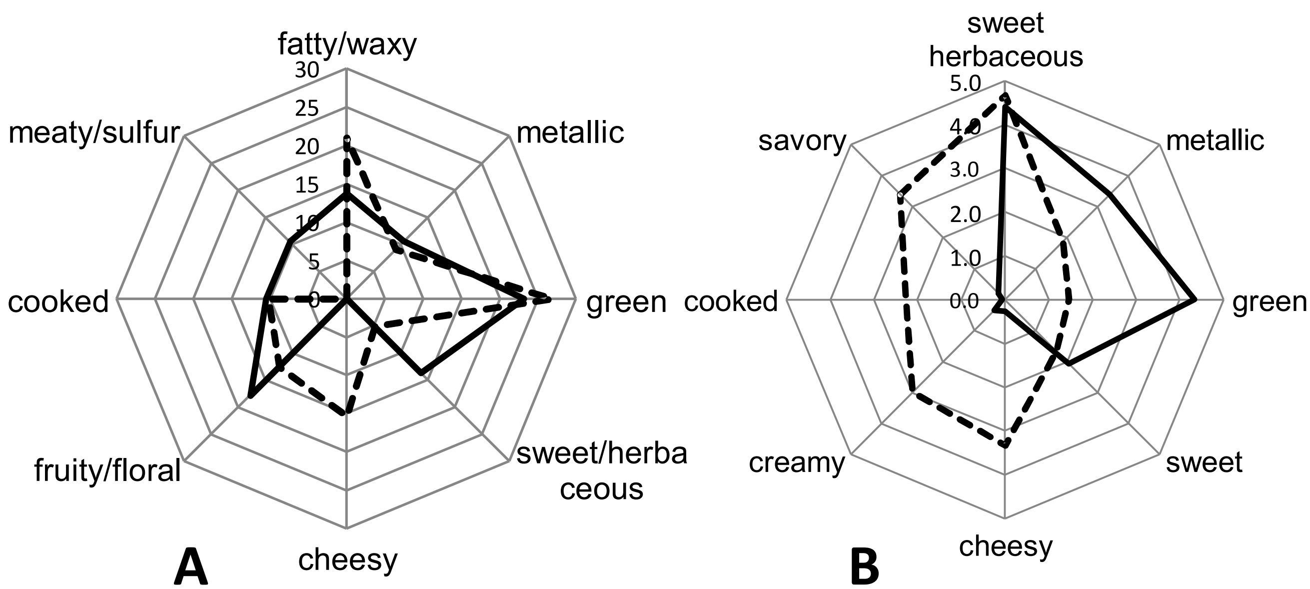
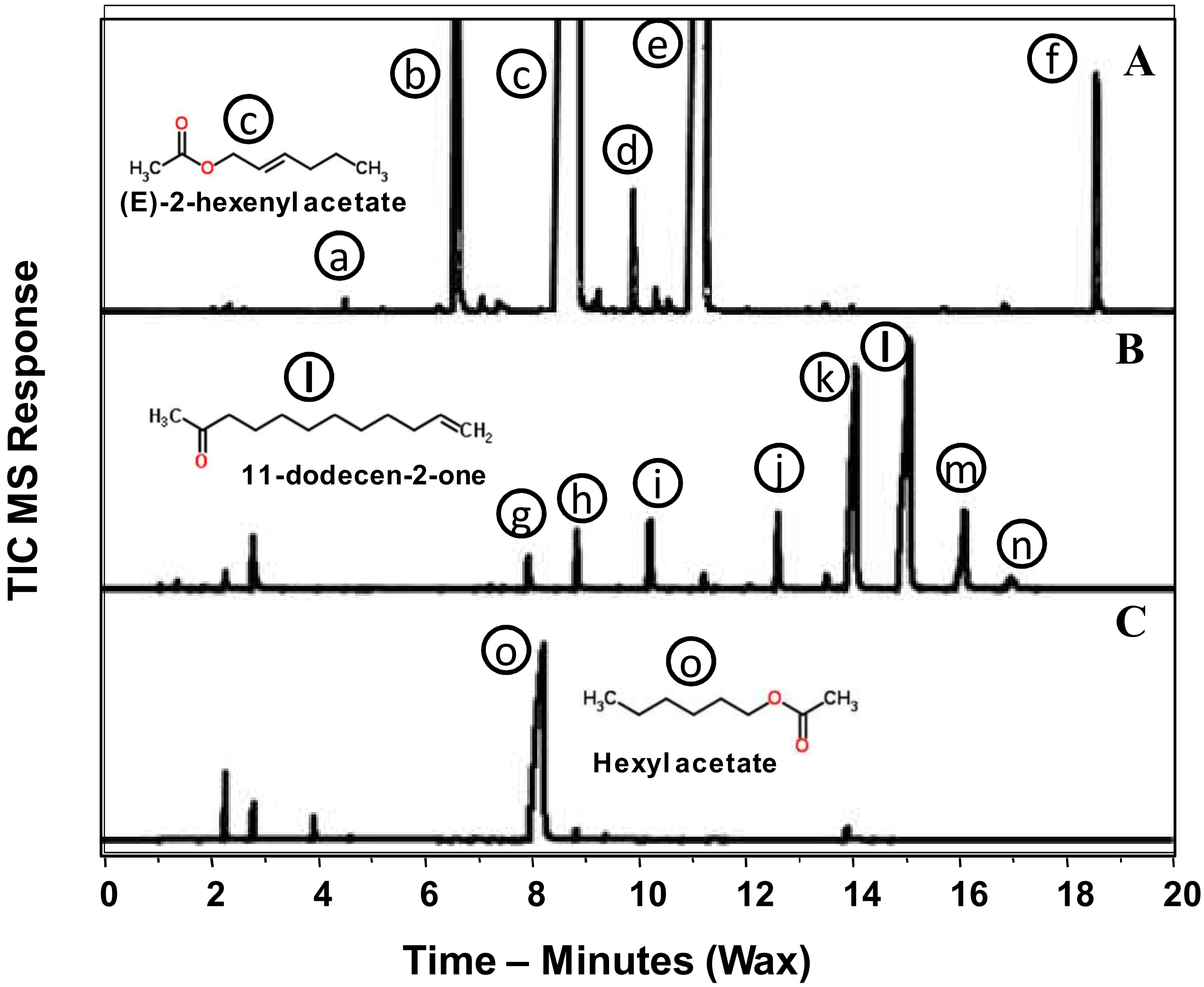


Figure 3.


A
B
C
a
b
c
d
e
f
g
h
i
j
k
l
m
n
o
(E)-2-hexenyl acetate
11-dodecen-2-one
Hexyl acetate
TIC MS Response
0
2
4
6
8
10
12
14
16
18
20
Time – Minutes (Wax)

TOC graphic