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THESIS

AROMA-ACTIVE COMPOUNDS IN THAI CHILI PASTE (NAM PHRIK PHAO) AND THEIR CHANGES IN SPRAY-DRIED POWDERS

PREMSIRI ROTSATCHAKUL

A Thesis Submitted in Partial Fulfillment of
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Three types of Thai chili pastes (CP) were prepared, consisting of an unheated CP (UH-CP), a CP heated at 100°C for 25 min (H25-CP), and a CP excessively heated for 50 min (H50-CP). The aroma-active compounds in the CPs were investigated by two gas chromatography-olfactometry methods: dynamic headspace dilution analysis (DHDA) and aroma extract dilution analysis (AEDA). The result from DHDA indicated that methanethiol, dimethyl sulfide, 2-methylpropanal and allyl mercaptan were the predominant headspace odorants of CPs and these compounds increased with heating time. The result from AEDA showed that 2-vinyl-4*H*-1,3-dithiin and diallyl trisulfide had the highest $\log_3$FD factors in H25-CP. In addition, 3-ethyl-2,5-dimethyl pyrazine and 3-hydroxy-4,5-dimethyl-2(*5H*)-furanone (sotolon) were indicated as potent odorants in basic and acidic fractions of H25-CP, respectively. On the basis of their high FD factors by both GCO methods, the predominant odorants in all CPs were 3-vinyl-4*H*-1,2-dithiin, allyl methyl disulfide, and allyl methyl trisulfide. Furthermore, dimethyl trisulfide and diallyl disulfide had the highest odor-activity values in H25-CP, suggesting that these were also aroma-active compounds in H25-CP. The effect of different combination of maltodextrin DE 18.5 (MD) and gum arabic (GA) carrier materials on the flavor release and flavor retention of chili paste oil (CPO) during storage at 50 °C and 68% RH for 5 weeks were evaluated. The three samples with MD/GA ratios of 1:2, 1:1 and 2:1 w/w were prepared by spray drying. The MD/GA (1:2) sample showed the highest content of the total oil and the encapsulation efficiency of microencapsulated spray-dried CPO powders. Dynamic headspace analysis was applied to determine the rate of release of volatiles from the microencapsulated powders. The combination containing higher proportions of GA provided lower amounts of the release of selected volatiles in the headspace. Better total flavor retentions of detected volatiles at the initial time and after 5 weeks of storage were obtained for a MD/GA ratio of 1:2. The result from quantitative descriptive analysis (QDA) revealed that the aroma intensities of some selected attributes were slightly decreased in each sample at the end of storage time. It can be assumed that spray-dried CPO powders could retain the most aroma intensities during storage time for 5 weeks.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF TABLES (Continued)

LIST OF FIGURES

LIST OF FIGURES (Continued)

LIST OF FIGURES (Continued)

LIST OF FIGURES (Continued)

LIST OF FIGURES (Continued)

LIST OF FIGURES (Continued)

LIST OF ABBREVIATIONS

AEDA	=	Aroma Extract Dilution Analysis
AF	=	Acidic fraction
a_w	=	Water activity
BF	=	Basic fraction
BHT	=	Butylated hydroxytoluene
b.p.	=	Boiling point
CIS	=	Cooled Injection System
CP	=	Thai chili paste
CPO	=	Chili paste oil
DB5	=	DB-5MS column
DHDA	=	Dynamic Headspace Dilution Analysis
DOD	=	Deodorized distilled water
DSC	=	Differential Scanning Calorimetry
EE	=	Encapsulation efficiency
FD factor	=	Flavor Dilution factor
FFAP	=	Stabilwax®-DA capillary column in Table 7-9 and Appendix Table A1 and A2; DB-FFAP capillary column in Appendix Table B3
FID	=	Flame Ionization Detector
GA	=	Gum arabic
GC	=	Gas Chromatography
GC-MS	=	Gas Chromatography-Mass Spectrometry
GCO	=	Gas Chromatography-Olfactometry
H25-CP	=	Typically heating Thai fried chili paste ingredients at 100 °C for 25 min
H50-CP	=	Excessively heating Thai fried chili paste ingredients at 100 °C for 50 min
HP-5MS	=	HP-5MS capillary column
In	=	Insoluble

LIST OF ABBREVIATIONS (Continued)

MD	=	Maltodextrin
MSD	=	Mass Selective Detector
na	=	Not available
nd	=	Not detected
NF	=	Neutral fraction
OAV	=	Odor-Activity Value
ppb	=	Parts per billion
ppm	=	Parts per million
QDA	=	Quantitative descriptive analysis
RI	=	Retention Index
RTX5	=	RTX®-5SILMS capillary column
S	=	Soluble
SL	=	Slightly soluble
SAFE	=	Solvent Assisted Flavor Evaporation
t	=	Odor detection threshold
TDS	=	Thermal Desorption System
T_g	=	Glass transition temperature
UH-CP	=	Unheated Thai fried chili paste ingredients
VSL	=	Very slightly soluble
VS	=	Very soluble

AROMA-ACTIVE COMPOUNDS IN THAI CHILI PASTE (NAM PHRIK PHAO) AND THEIR CHANGES IN SPRAY-DRIED POWDERS

INTRODUCTION

Nam Phrik Phao, Thai chili paste (CP), is widely consumed in Thailand. It is used as a condiment in Thai food such as Tom Yam, stir-fried sea foods or it can be used as a spread for breads or crackers. CP has specific flavors derived from its ingredients and processing. The flavor of CP is one of the most important criteria determining consumer choice and acceptance. Its ingredients include chili powder, garlic, shallot, dried shrimp, tamarind paste, fish sauce, salt, coconut sugar, and soybean oil. Dried shrimp, fish sauce, and shrimp paste provide a protein source and characteristic aroma of dried seafood. Garlic and shallot are the most prevalent culinary herbs that contribute sulfur compounds that usually possess strong pungent and spicy organoleptic characters. In addition, reaction of ingredients via the Maillard reaction system can create additional aroma components during heating.

The characteristic aroma components of CP has not been previously studied. One of the purposes of this dissertation was to identify and compare the chemical nature of aroma-active compounds of unheated CP (UH-CP), CP heated at 100°C for 25 min (H25-CP), and excessively heated CP (100 °C for 50 min; H50-CP). The results will aid in the predominant volatile compounds of CP caused by ingredients or processing may be more clearly detected and characterized.

CP exhibits sensitivity to light, heat, and oxygen. Prolong heating and drying cause loss of the aroma from CP which prevent the use of dried CP in food products such as soups and snacks. Production of CP powder that retains the authentic aroma quality will benefit food industry. Encapsulation has been used in flavor industry to protect aroma compounds from destructive changes. There are no reports on encapsulation of chili paste. In the light of this information, an attempt has been

made to encapsulate chili paste oil by spray drying using gum arabic and maltodextrin as the wall materials. The microcapsules were evaluated for the content and stability of volatiles during storage.

Studies on changes in the aroma profile and flavor retention were undertaken to understand the effect of different wall materials on encapsulated aroma quality of CP.

OBJECTIVES

1. To identify and quantify the aroma-active components formed during heating process of Thai chili paste.

2. To assess the release of the major volatile compounds from the mixtures of gum arabic and maltodextrin in various proportions by dynamic headspace (DHS) gas chromatography (GC) analysis.

3. To study the effects of the different metrices components of wall materials in the retention of selected aroma active compounds of spray dried Thai chili paste oil powders.

4. To compare the aromatic profile of spray-dried CP oil encapsulated in different wall materials by sensory analysis during storage.

LITERATURE REVIEW

Volatile compounds formed during food processing are derived from lipid oxidation and degradation, Maillard reaction, Strecker degradation of amino acids, and caramelization of sugars. The reactive compounds formed in these initial reactions can react to form the important classes of flavor compounds (Ó Hare and Grigor, 2005).

1. Lipid Oxidation and Degradation

Several volatile compounds derived from lipid degradation include aliphatic (and some aromatic) hydrocarbons, aldehydes, ketones, alcohols, fatty acids and other carboxylic acids, esters, lactones, and alkylfurans.

1.1 Hydrolysis

Hydrolysis of lipids releases free fatty acids. Short-chain fatty acids have distinctive odor, the long-chain fatty acids which are more common in meat fat are not significant aroma chemicals, hydrolysis is favored by high activity and acid/base catalysis (Ó Hare and Grigor, 2005).

1.2 Oxidation

The lipids that contribute flavor precursors include triacylglycerols and membrane phospholipids. Phospholipids contain a much higher proportion of unsaturated fatty acids than triacylglycerols and prone to oxidation. Lipid oxidation products have strong and characteristic aromas even at very low concentrations. Lipid oxidation compounds, e.g., *trans,cis*-2,4-deacdienal, *cis*-hex-3-enal, and penta-2,4-dienal contribute to an unpleasant rancid aroma of oxidized fat. Formation of these compounds is favored by low temperature conditions. At high temperatures, thermolytic and oxidative degradation of lipids produce a different balance of compounds, which is normally considered more beneficial (Mistry and Min, 1992).

1.3 Fatty aldehydes

Aldehydes with 6-10 carbons have green, fatty, or tallow aromas. Unsaturated aldehydes may have more interesting aromas. For example, 2,4-decadienal has an aroma of fat-fried food. Alkyl derivatives of heterocyclics also have fried or fatty flavors (Ó Hare and Grigor, 2005).

1.4 Fatty aldehydes

Aldehydes formed during lipid oxidation could react with compounds from Maillard reaction (Mottram, 1998). Fatty aldehydes may contribute the alkyl groups to alkylthiazoles and alkylpyridines (Mottram, 1985). Butyl and pentyl pyrazines could result from the reaction between pentanal or hexanal and dihydropyrazine that formed by the condensation of two aminoketone molecules (Ho *et al.*, 1987). Pentanal may be involved in the formation of 5-butyl-3-methyl-1,2,4-trithiolane (Mottram, 1998). The reaction of pentanal or hexanal with hydrogen sulfide and acetyldehyde has been suggested as the route to butyl and pentyl trithiolanes (Ho *et al.*, 1987).

2. Strecker Degradation

Strecker degradation of amino acids occurs in conjunction with Maillard reaction. In Strecker degradation amino acids are decarboxylated and deaminated in the presence of dicarbonyl compounds forming 'Strecker' aldehydes and aminoketones (Figure 1). Aldehydes are reactive carbonyls that can take part in the Maillard reaction. Aminoketones can involve in cyclization reactions to produce heterocyclics such as pyrazines. Dicarbonyl compounds which participate in Strecker degradation may be derived from lipid oxidation or Maillard reaction (Martins *et al*., 2001).

In degradation of cysteine, the Strecker aldehyde produced is 2-thioethanal (mercaptoacetaldehyde). Further degradation of 2-thioethanal releases carbon dioxide

and hydrogen sulfide. Hydrogen sulfide could reacts with aldehydes, furfurals, and furanones to produce several 'meaty' sulfur compounds (Rizzi, 1999). These compounds have very low odor thresholds.

3. The Maillard Reaction

Maillard reaction that occurs between carbonyls and amino groups is important in the formation of most cooked flavors. The reaction is named after Louis Maillard, who reported on the formation of melanoidins following the reaction of glycine with glucose (Maillard, 1912). The reaction is most famously represented by the classic scheme (Figure 2) proposed by Hodge (1953). The initial stages of the reaction involve the condensation of the carbonyl groups (found on reducing sugars) with an amino compounds (an ε-amino group of a protein-bound lysine residue or the α-amino group of free amino acids) to give Schiff's bases and then glycosylamines. Subsequent stages include Amadori rearrangement, if the starting sugar is an aldose, or Heyns rearrangement, if the starting sugar is a ketose). The rearangements generate thermally unstable intermediates which then undergo dehydrations and deaminations to form various products such as hydroxyketones, dicarbonyl compounds and derivatives of furfurals and furanones. Reactive carbonyls can be formed from lipid degradation, Strecker degradation, and Maillard reaction. A whole series of analogous reactions can occur with thiols derived from cysteine or methionine replacing amino groups as the nucleophile. Water activity, pH and temperature are key parameters in determining the progress of Maillard reaction. Many of the steps are promoted by acid or base catalysis. Water acts as solvent, promoting the reaction by allowing mobility of the reactants. The reaction occurred very slowly at extremely low water activity (Tressl and Rewicki, 1999; Yaylayan, 2003).

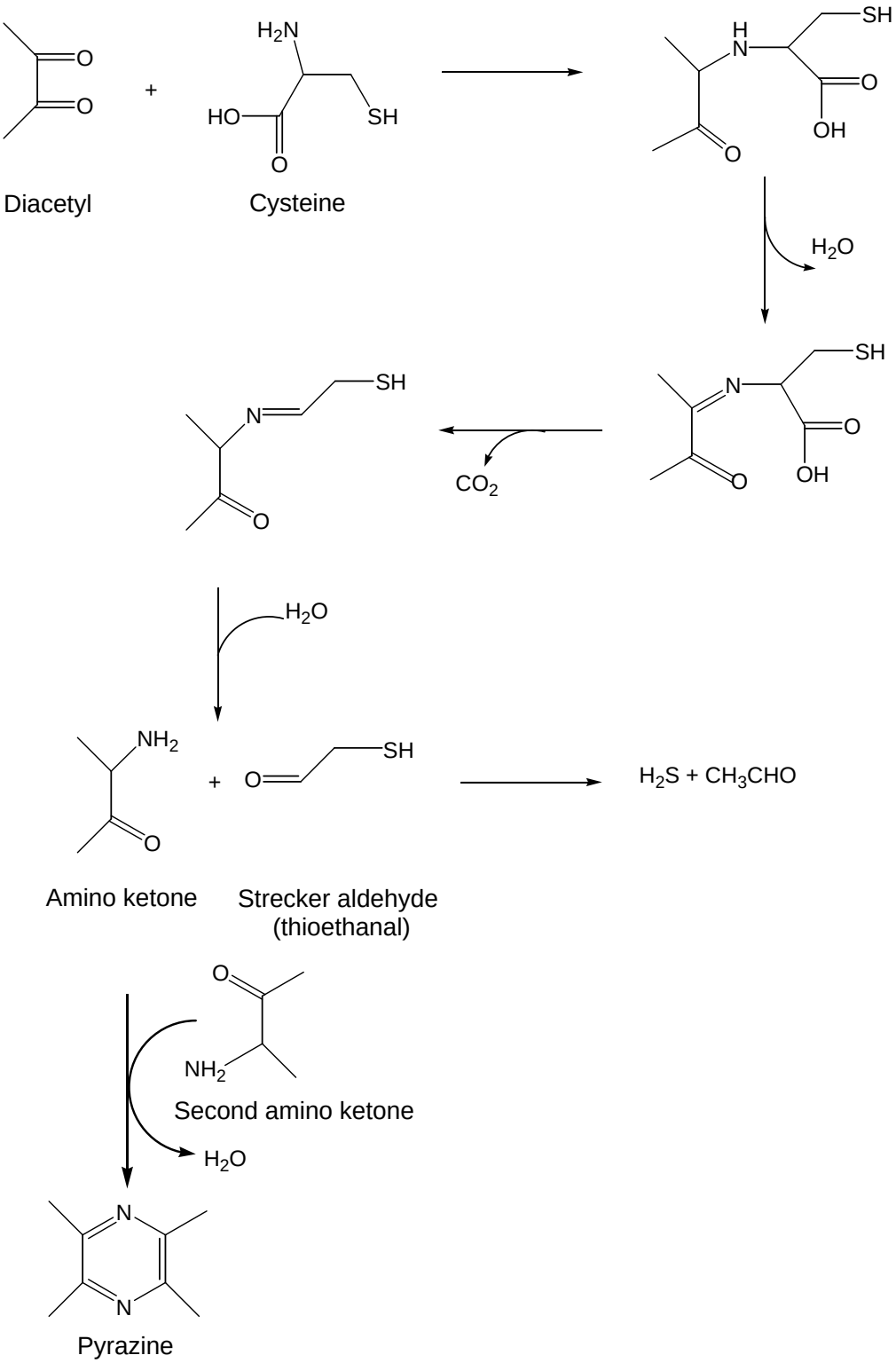


Figure 1 Strecker degradation (illustrated by the reaction of diacetyl with cysteine)

Source: Ó Hare and Grigor (2005)

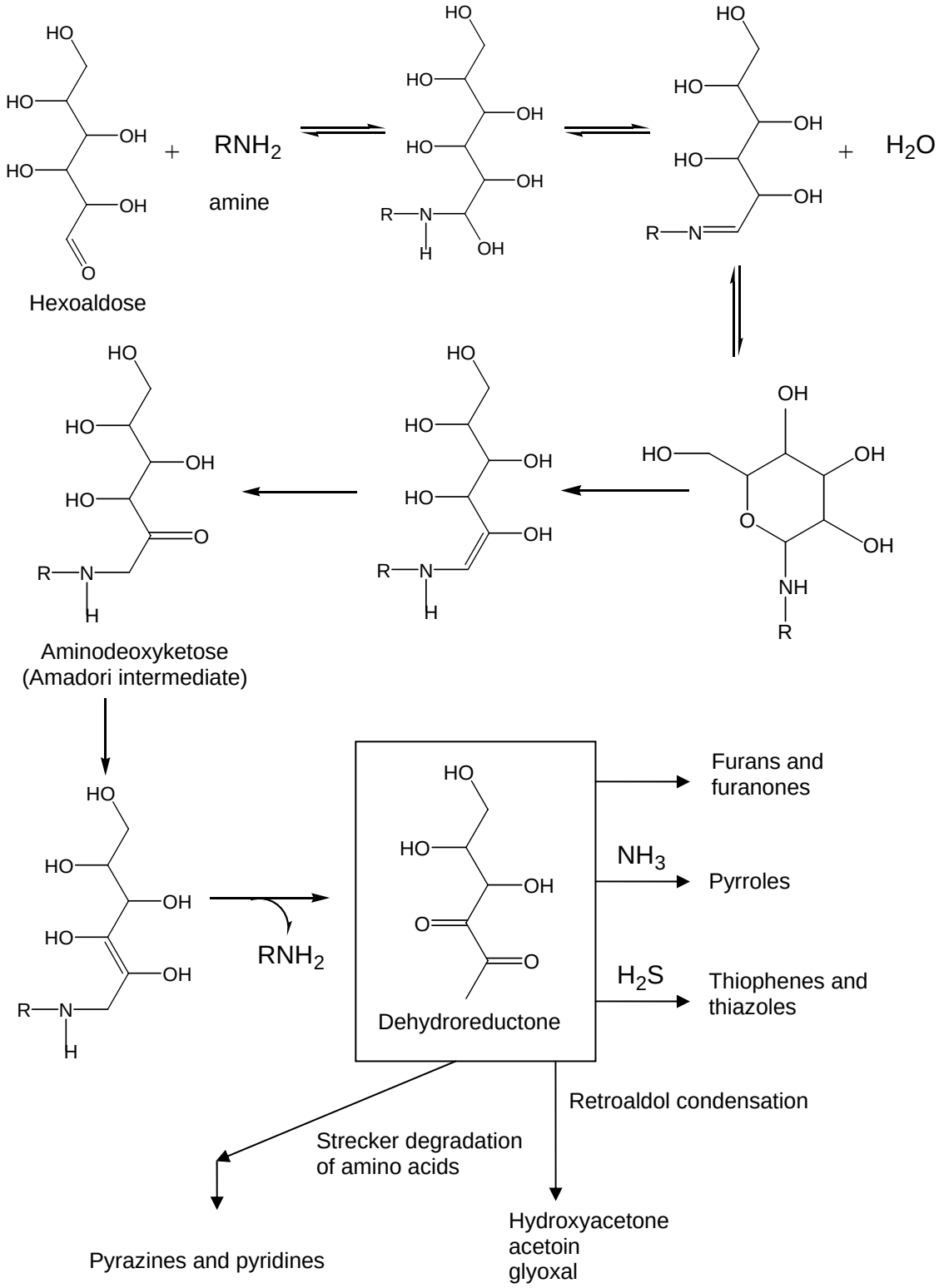


Figure 2 The Maillard reaction

Source: Ó Hare and Grigor (2005)

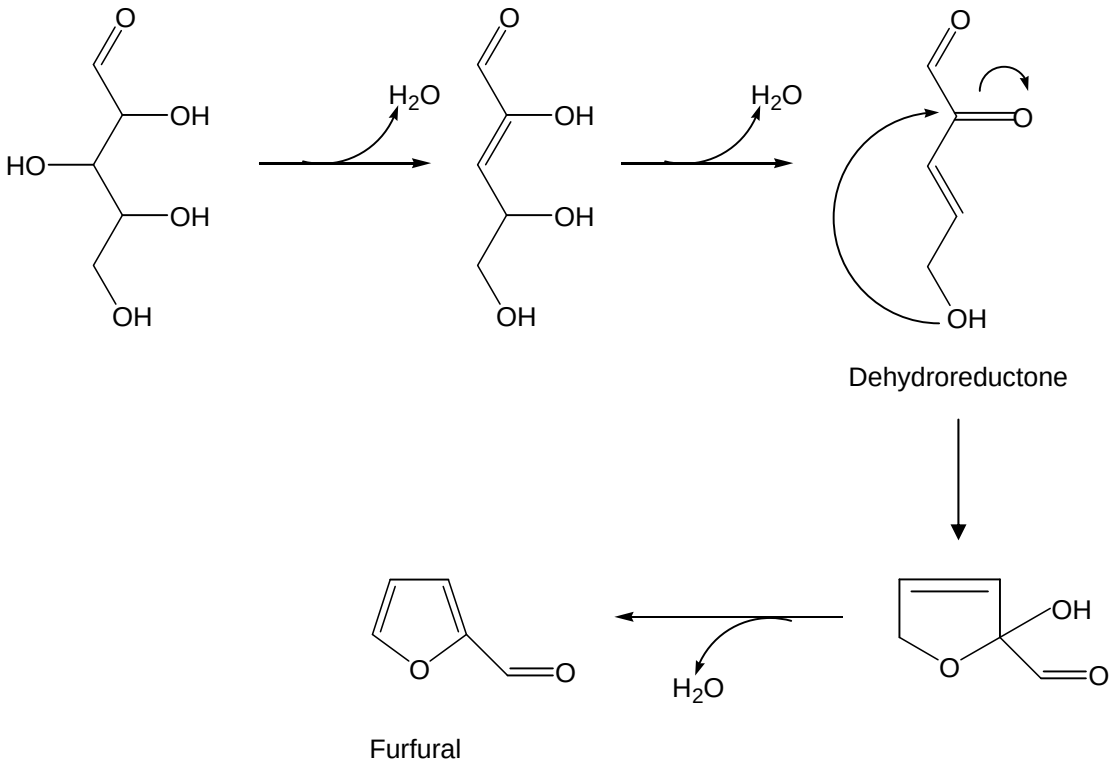


Figure 3 Caramelization

Source: Ó Hare and Grigor (2005)

4. Caramelization

Heating of sugars above 150 °C in the condition that has low water activity generates anhydrides. Anhydrides further dehydrate (Figure 3) to formed furfural (from pentoses) or 5-hydroxymethylfurfural (from hexoses). Continued reaction at high temperature gives rise to aliphatic and aromatic hydrocarbons, furans, aldehydes and ketones including dicarbonyls which may take part in Strecker degradation (Tressl and Rewicki, 1999). Under certain conditions complex alcohols and lactones may be formed.

Aroma Compounds in the Ingredients

The ingredients of CP include dried chili, garlic, shallot, dried shrimp, tamarind paste, fish sauce, salt, coconut sugar, and vegetable oil. The volatile components of the ingredients have been previously studied, especially garlic and shallot.

1. Garlic

Garlic (*Allium sativum* Linn.) has been extensively used worldwide as an important vegetable, spice, and seasoning. Many kinds of garlic products, such as garlic oil, garlic powder, garlic salt, garlic paste, and garlic flakes, have been used in the home or in the food industry. These garlic products can be divided into the following forms according to their preparation methods: raw garlic, dried garlic, boiled garlic, baked garlic, and fried garlic. The difference in preparation methods can result in different flavor sensations of these garlic products.

Intact cloves of garlic contain the colorless and odorless compound, alliin (3-[(S)-2-propenylsulfinyl]-L-alanine). Alliin is converted into allicin (2-propene-1-sulfinothioic acid S-2-propenyl ester) by allinase (cysteine sulfoxide lyase; C-S lyase, EC 4.4.1.4) after the clove has been bruised, cut, or crushed. Allicin is unstable and easily converted into more stable sulfur compounds, e.g. diallyl sulfide, diallyl disulfide, diallyl trisulfide, vinyl dithiines, or ajoenes over time or after heating (Block *et al*., 1986). Allicin and the sulfur compounds produced from allicin have characteristic garlic odors and tastes (Block, 1992).

As shown in Figure 4, during the physical breakdown of garlic cells, alliin, can be transformed to allicin, the primary flavor compounds of garlic, by the action of allinase through the dehydration process, accompanied by the formation of pyruvate and ammonia.

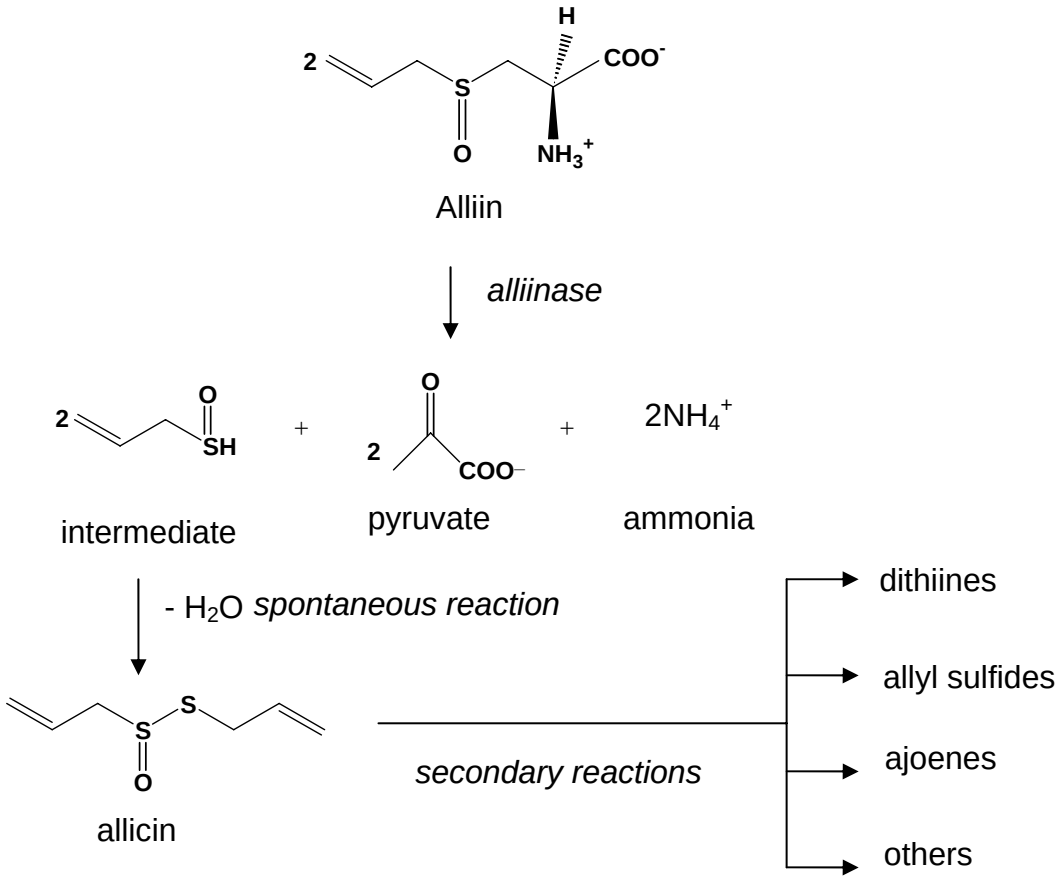


Figure 4 Enzymatic reaction of allinase with alliin of garlic
Source: Lee *et al*. (2003)

Allicin self decomposes to form two isomeric cyclic compounds, 2-vinyl-[4*H*]-1,3-dithiin and 3-vinyl-[4*H*]-1,2-dithiin. The spectra of these three compounds are very similar (Yu and Wu, 1989b). In another pathway as shown in Figure 5, 2-vinyl-[4*H*]-1,3-dithiin and 3-vinyl-[4*H*]-1,2-dithiin are postulated to be dehydration products of allicin by heat treatments (Yu and Wu, 1989a).

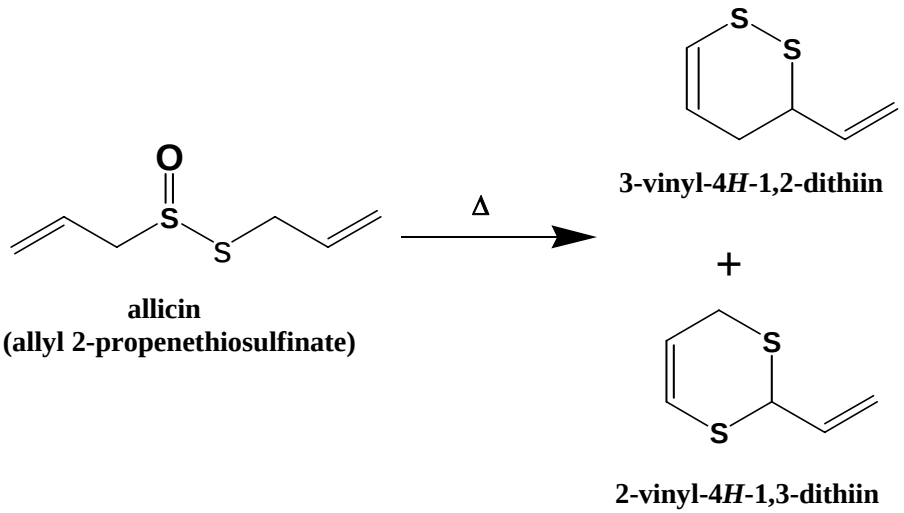


Figure 5 Formation of 2-vinyl-4*H*-1,3-dithiin and 3-vinyl-4*H*-1,2-dithiin from Allicin

Source: Abu-Lafi *et al*. (2004)

Some of the allylthio-containing volatile compounds, especially diallyl sulfide and diallyl disulfide are believed to be responsible for the characteristic of heated garlic flavor (Lawson *et al*., 1991). The most abundant volatile compounds in garlic are diallyl trisulfide followed by diallyl disulfide and methyl allyl trisulfide (Calvo-Gómez *et al*., 2004).

Several studies have been carried out on the volatile compounds formed upon oil-frying, baking, boiling, and microwaving of garlic (Yu *et al*., 1993, 1994a, b, c). Most thermal processes generate 40-60 volatile compounds except for boiling that generates 16 compounds. Baking and microwaving of sliced garlic produce the highest amount of volatiles whereas boiling and baking whole garlic cloves produce the least. The dominant volatile formed in all thermal processes of garlic is diallyl disulfide. Diallyl trisulfide is abundant only in the baked and microwaved garlic slices.

Major compounds of heated garlic oil are diallyl trisulfide, diallyl disulfide, methyl allyl trisulfide, methyl allyl disulfide, diallyl sulfide, methyl allyl sulfide, dimethyl trisulfide, 2-vinyl-[4*H*]-1,3-dithiin and 3-vinyl-[4*H*]-1,2-dithiin. Most of these sulfur-containing compounds come from the decomposition and rearrangement of allicin at high temperature (Yu and Ho, 1993).

Volatile compounds in blanched and thermally treated blanched garlic slices are listed in Table 1 (Yu *et al.*, 1994b). The major volatile compounds generated from thermally degraded nonvolatile flavor precursors are allyl disulfide, 1-propene, acetaldehyde, allyl sulfide, methyl allyl trisulfide, and other poly sulfur-containing cyclic compounds. The major volatile compounds generated from thermal interactions of nonvolatile flavor precursors of garlic and sugars were pyrazines. Formation of pyrazines in these thermally treated blanched garlic slices are proposed through Maillard reactions of the amino-containing precursors and the reducing sugars in garlic tissues. The major volatile compounds in baked blanched and fried blanched garlic slices which are probably generated from thermal interactions of nonvolatile flavor precursors and lipids are pyridines. Thermal interactions of alliin and 2,4-decadienal, the major degradation products of vegetable oil containing linolenic acid, are mainly alkylthiophenes, especially 2-formal-5-pentylthiophene, 2-hexylthiophene, 2-butylthiophene, and 2-hexanoylthiophene, 2-pentylpyridine, and 2-pentylbenzaldehyde (Yu *et al*., 1994c).

Table 1 Important volatile compounds identified in garlic samples

compound	yield (ppm)		
	BBG[a]	FBG[a]	BG[a]
compounds probably generated from thermal degradation of nonvolatile flavor precursors (alliin and deoxyalliin)			
1-propene	7.12	10.55	2.20
acetaldehyde	1.85	25.24	0.70
allyl methyl sulfide	0.06	1.31	nd[b]
allyl sulfide	1.16	8.99	0.09
allyl methyl disulfide	0.80	7.26	0.04
1,2-dithiacylclopent-3-ene	0.77	2.15	0.06
allyl disulfide	9.14	50.74	2.40
allyl methyl trisulfide	1.53	4.34	0.17
3-vinyl-4*H*-1,2-dithiin	0.49	1.44	0.14
2-vinyl-4*H*-1,3-dithiin	0.31	0.72	nd
allyl trisulfide	0.52	0.66	0.06
compounds probably generated from thermal interactions of sugars and nonvolatile flavor precursors (alliin and deoxyalliin)			
2,5-dimethylpyrazine	0.51	0.69	nd
ethylmethylpyrazine	0.35	0.72	nd
3,5-diethyl-2-methylpyrazine	0.47	0.45	nd
compounds probably generated from thermal interactions of lipids and nonvolatile flavor precursors (alliin and deoxyalliin)			
4-heptenal	0.11	0.68	nd
2-ethylpyridine	0.12	0.20	nd
2-pentylfuran	nd	0.22	nd
methylethylpyridine	0.06	0.19	nd
phenylacetaldehyde	0.10	0.44	nd

[a] BBG : baked blanched garlic; FBG: fried blanched garlic; BG: blanched garlic
[b] nd : not detected
Source: Yu *et al*. (1994b)

2. Shallot

Shallot (*Allium ascalonicum* Linnaeus) is normally crushed or cut before deep oil frying to produce flavor.

Volatile oils from raw, baked, and deep-fat-fried shallots have been studied (Wu *et* al., 1982). Baking and deep-frying of shallot increase the amounts of 2,4-dimethylthiophene, 3,4-dimethylthiophene, methyl 1-propenyl trisulfide, and propyl 1-propenyl trisulfide but decrease the amount of saturated alkyl (dimethyl, methyl, propyl, and dipropyl) trisulfides and unsaturated alkyl disulfide. Methyl propyl trisulfide is the most abundant volatile compound in volatile oils of shallot. Methyl propenyl disulfide and propyl propenyl disulfide present in lesser amounts but their changes upon heating were conspicuous (Wu *et al.,* 1982).

The aroma of fried onion could be from 2,4- and 3,4-dimethylthiophenes (Boelens *et al*., 1971). The formation of 2,4-dimethylthiophene was proposed to be formed from alkyl propenyl disulfide through a free-radical mechanism (Boelens and Brandsma, 1972). 3,4-Dimethylthiophene can be formed by the same mechanism. Figure 6 shows a possible mechanism for the formation of 2,4- and 3,4-dimethylthiophene.

Volatile compounds of blanched and thermally treated blanched shallot slices as listed in Table 2 (Chen, 1996). The major volatile compounds from baked blanched and fried blanched shallot slices generated from thermal degradation of nonvolatile flavor precursors of shallot are methyl propyl trisulfide, dimethylthiophenes, methyl propyl disulfide, and dipropyl trisulfide. The major volatile compounds that are probably generated from thermal interactions of nonvolatile flavor precursors of shallot and sugars are pyrazines, especially ethyl dimethyl pyrazines, dimethyl pyrazines, ethyl methyl pyrazines, and trimethylpyrazines. The major sulfur-containing compounds of deep-fried shallots contained propenyl group in their structures, which is apparently different from the allyl group of garlic flavor components (Jirovetz *et al*., 1992).

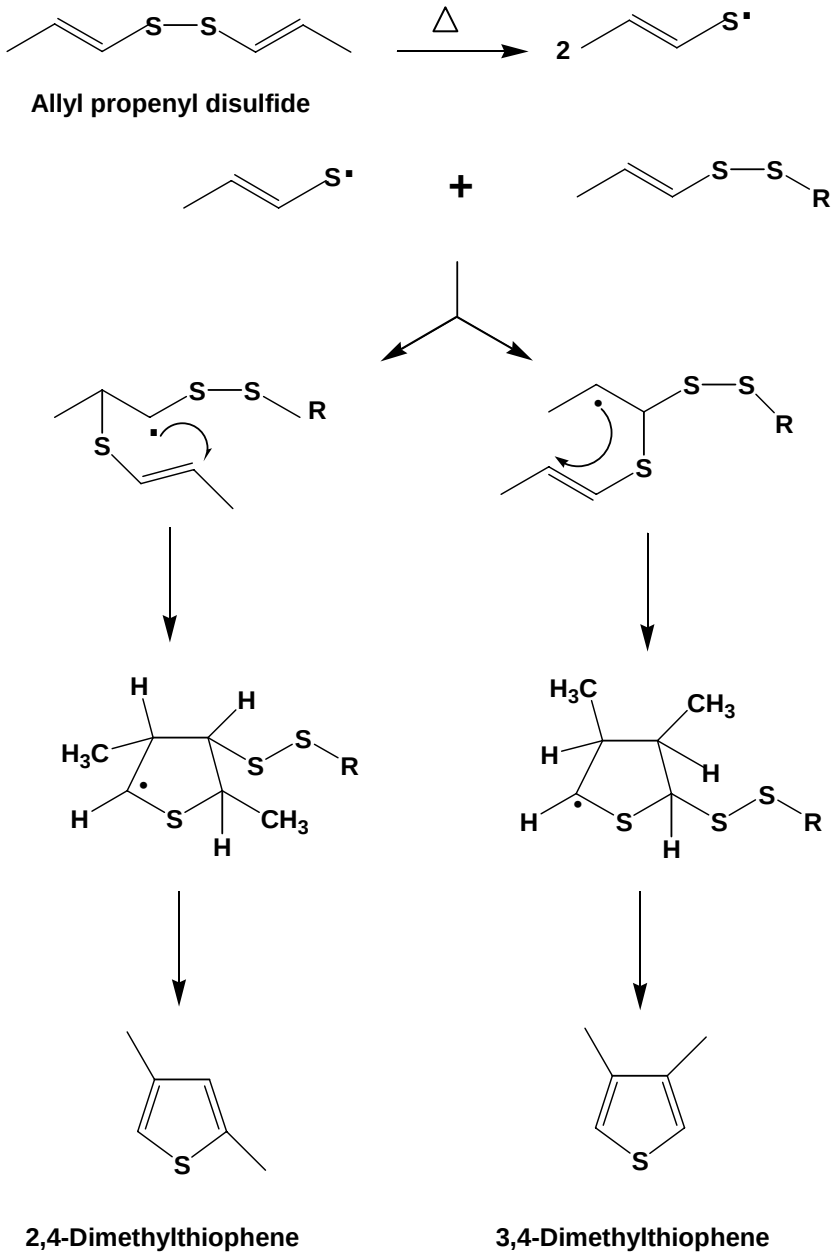


Figure 6 Possible mechanism for the formation of alkylthiophenes in heated shallots

Source: Ho *et al*. (1997)

The analysis of the headspace volatiles above freeze-dried, baked, or fried shallots has also been conducted (Wu and Wu, 1982). While the earlier data showed that over 40% of the volatile oil of fresh shallots was composed of dimethyl and methyl trisulfide, these accounted for only 5% of the headspace of the freeze-dried product. The main volatiles in freeze-dried product is approximately 10% of *cis*- and 10% of *trans*-(1-propenyl) propyl disulfide.

About 50% of baked shallot oil extracts comprised methyl propyl trisulfide, methyl-(1-propenyl) trisulfide, and propyl-(1-propenyl) trisulfide , these compound contributed less than 10% of the headspace volatiles of the freeze-dried product. Dipropyl disulfide and methyl propyl disulfide were consisted only 5% in freeze-dried shallot.

The oil from the deep-fried shallots contained mainly methyl propyl disulfide, methyl propyl trisulfide, isomeric (1-propenyl) propyl trisulfide, and 3,4-dimethylthiophene (in combination more than 50% of total oil); in contrast, a quarter of the headspace volatile comprised dipropyl disulfide and dimethyl trisulfide.

Table 2 Important volatile compounds identified in shallot samples

compound	yield (ppm)		
	BBS[a]	FBS[a]	BS[a]
compounds probably generated from thermal degradation of nonvolatile flavor precursors (alliin and deoxyalliin)			
1-propene	0.43	0.86	nd[b]
methanethiol	0.58	1.96	nd
dimethyl disulfide	0.46	1.91	nd
dipropyl disulfide	1.66	2.00	nd
1-propanethiol	0.29	0.30	0.03
3-methylthiophene	nd	0.78	nd
methyl propyl disulfide	2.49	8.50	0.01
dimethylthiophene	4.83	15.44	nd
1-propenyl methyl disulfide	0.75	5.46	0.08
dimethyl trisulfide	3.52	7.28	nd
1-propenyl propyl trisulfide	nd	1.42	nd
methyl propyl trisulfide	19.12	16.44	nd
dipropyl trisulfide	7.29	3.94	nd
compounds probably generated from thermal interactions of sugars and nonvolatile flavor precursors (alliin and deoxyalliin)			
pyridine	nd	0.41	nd
2-pentylfuran	nd	5.80	nd
methylpyrazine	1.14	5.48	nd
dimethylpyrazine	13.64	59.24	nd
2,3-dimethylpyridine	1.23	nd	nd
ethyl methylpyrazine	6.11	15.38	nd
trimethylpyrazine	4.53	10.77	nd
5-ethyl-2-methylpyridine	nd	0.82	nd
2,6-diethylpyrazine	nd	3.38	nd
ethyl dimethylpyrazine	33.85	29.38	nd

Table 2 (Continued)

compound	yield (ppm)		
	BBS[a]	FBS[a]	BS[a]
compounds probably generated from thermal interactions of sugars and nonvolatile flavor precursors (alliin and deoxyalliin)			
methyl propylpyrazine	4.43	3.31	nd
dimethyl propylpyrazine	2.70	12.83	nd
2,3-dimethyl-5-[1-methylpropyl]pyrazine	1.51	nd	nd
3-ethyl-2,6-dimethylpyridine	0.94	nd	nd

[a] BBS: baked blanched shallot; FBS: fried blanched shallot; BS: blanched shallot

[b] nd.: not detected

Source: Chen (1996)

3. Other Ingredients

Other ingredients for CP are dried chili, dried shrimp, tamarind paste, fish sauce, salt, coconut sugar, and vegetable oil.

Aroma quality of dried chili is similar to that of paprika. It has been reported that the heat generated volatile compounds in spice paprika powder were Strecker aldehydes (acetaldehyde, 2-methylpropanal, 2- and 3-methylbutanal), hexanal, 6-methyl-5-hepten-2-one and β-ionone, especially hexanal could be used as a marker compounds for heat treatment of paprika (Cremer and Eichner, 2000). Additionally, Mazida *et al*. (2005) reported that 2-isobutyl-3-methoxypyrazine, 2,3-butanedione, 3-carene, trans-2-hexenal and linalool were the principal compounds of fresh chili at three ripening sages (green, turning, and red). Zimmermann and Schieberle (2000) also reported that 4-hydroxy-2,5-dimethyl-3-(2*H*)-furanone (HDMF; caramel-like), 3-hydroxy-4,5-dimethyl-2(5*H*)-furanone (sotolon; seasoning-like) and 2- and 3-methylbutanoic acids contribute to the aroma of sweet bell pepper powder.

Fish sauce has 2-methylpropanal, 2-methylbutanal, 2-pentanone, 2-ethylpyridine, dimethyl trisulfide, 3-(methylthio)-propanal (methional), and 3-methylbutanoic acid as the principal contributors to the distinctive odor (Fukami *et al.,* 2002).

Major aroma compounds in tamarind are furfural, 5-methyl-2(3*H*)-furanone, phenylacetaldehyde, and 5-methylfurfural (Zhang and Ho, 1990).

Techniques for Isolation of Aroma Compounds

CP is a complex food system that composed with many ingredients such as chili powder, garlic, shallot, dried shrimp, tamarind paste, fish sauce, salt, coconut sugar, and soybean oil. To achieve the most complete aroma profile, the analyst must have to use more than one extraction technique.

1. Dynamic Headspace

In dynamic headspace, the sample is purged with an inert gas, such as nitrogen or helium, which strips aroma constituents from the sample. The volatile in the purge gas must then be trapped onto a trap containing porous polymers such as Tenax™. To increase the release of volatiles, samples can be heated and/or stirred during collection (Da Costa and Eri., 2005; Reineccius, 2006). Dynamic headspace techniques offer many advantages including elimination of the solvent peak, analysis of only volatiles, and easy sample preparation (Wampler, 1997).

Tenax™ (poly-2,6-diphenyl-*p*-phenylene oxide) is widely used for aroma trapping. It is capable of sorbing a wide range of organic volatiles. Tanex is especially good with aromatics but it is not suitable for very volatile hydrocarbons (pentane and below) or for small alcohols. It has small surface area, therefore, low adsorption capacity. In addition, Tenax has low affinity for polar compounds and high affinity for nonpolar compounds (Reineccius, 2006; Wampler, 1997).

When Dynamic headspace is used for liquid samples, the carrier gas is introduced into the sample, and 'purges' the volatiles out into the headspace and onto the trap. Therefore, for liquid samples, the term 'purge and trap' instead of 'dynamic headspace' is usually used (Da Costa and Eri., 2005) **.**

2. Direct Solvent Extraction and Solvent Assisted Flavor Evaporation (SAFE) Distillation

One of the simplest and most efficient approaches for aroma isolation is direct solvent extraction. The concept of this technique is to transfer the aroma compounds from the sample to the organic solvent. The extract is obtained by mixing and agitating a liquid or solid sample with organic solvent, allowing separation and collection of the solvent phase. The solvent must have a low boiling point so that it can be easily removed from the extract without a significant loss of sample volatiles and not to obscure other components during chromatograph. It must able to extract polar and non-polar components (Da Costa and Eri., 2005). The solvents most commonly used today are diethyl ether, diethyl ether/pentane mixtures, hydrocarbons, Freons, and methylene chloride. The later two have the advantage of being nonflammable. Nonpolar solvents such as Freons and hydrocarbons should be used when the sample contains alcohol. Diethyl ether and methylene chloride are good general purpose solvents. Diethyl ether can form explosive peroxides. For this reason diethyl ether usually contains inhibitors (e.g., BHT) that will show up in the chromatogram. Furthermore, methylene chloride is a satisfactory general purpose solvent, particularly for flavor compounds with an enolone structure (e.g., Maltol and Furaneol). It is somewhat toxic and is an animal carcinogen. To aid in extraction, sodium chloride may be added to aqueous phase to salt out the organics when low-density solvent are employed (Parliament, 1997).

The disadvantages of this technique are: (Da Costa and Eri., 2005)

- Solvent removal may cause loss of some of the more volatile compounds.

- Use of solvents which may be toxic and/or flammable and which may introduce contaminations.
- A need for large amounts of sample in order to produce a strong extract. Obtained extracts may contain high boiling and non-volatile materials and color components, which may lead to further problems in analysis and chromatography.
- Emulsion formation during extraction may occur.
- Solvent peak may cover early eluting volatiles in the chromatogram.

The major limitation of this method is that it is most useful on foods that do not contain any lipids. Lipids in food can be extracted along with aroma constituents, and they must be separated from each other before further analysis. Separation can be done by molecular distillation (high vacuum distillation) or solvent assisted flavor evaporation (SAFE) distillation (Reineccius, 2006).

To overcome the disadvantages of solvent extraction, the extraction may be followed by the SAFE technique. The SAFE technique can be used as a sole extraction technique for aqueous foods, such as milk, fruit pulps, and foods with high oil content (Engel *et al*., 1999). This technique takes the extract of food matrix and strips off the volatiles under high vacuum (10^{-5} Torr) without heating. The extract is collected in a cooled trap, which is cryogenically cooled with liquid nitrogen (Da Costa and Eri., 2005).

The extracts from natural products can be very complex making the GC chromatogram contain many co-eluting compounds. One way to overcome this problem is to fractionate the extract. This is important for gas chromatography-olfactometry (GC-O) analysis. In GC-O, the analyst aims to eliminate co-eluting to reveal organoleptically important compounds that may be hidden under bigger, less-odorous peaks (Da Costa and Eri., 2005). Fractionation of the extracts can be accomplished by washing the extracts with dilute acid and dilute base. The wash solutions is then re-extracted with solvent to obtain the fractions containing only acids or bases (Da Costa and Eri., 2005).

The extract or combined extracts are typically dried over anhydrous sodium sulfate to remove any water present and then filtered. At this stage the concentration of volatiles in the extract is usually too low for analysis. Most of the solvent has to be removed. The initial concentration of the extract (down to 10-15 mL) may be accomplished by Vigreaux column distillation. This can be followed by passing a fine stream of pure nitrogen gas over the surface of the extract to concentrate to a final volume of 1-2 mL (Da Costa and Eri., 2005).

Methods for Analysis of Aroma Extracts

1. Gas Chromatography -Olfactometry (GCO)

GCO is an important analytical tool in flavor research because it characterizes the odors of single compounds or complex mixtures of volatiles emerging from the sniffing port. The human nose is the detector used for evaluating the effluent of the GC column. The nose has a theoretical odor detection limit of about 10^{-19} moles, making GCO a very valuable and sensitive tool for detection of trace amounts of odor active volatiles or off-odors in foods (Mistry *et al*., 1997). In this technique, the sample pass through the column and is splitted to a flame ionized detector (FID) and a sniffing port. At sniffing port, a humidifier system keeps the air moist to reduce nasal dehydration. The odor of each compound as it elutes from the GC is recorded by trained panelists and the aromagram is created (Da Costa and Eri., 2005). The aromagram indicates which peaks have odor as well as lists the odor characteristic of the peaks (Reineccius, 2006).

GCO aids compound identification. The panelist may be able to identify the volatile compounds based on its aroma and GC retention properties (Reineccius, 2006). Occasionally, GCO data may be misleading due to the concentration effect. Odor characteristics of some flavor compounds can vary as a function of concentrations. For example, skatole (3-methyl indole) has fecal odor at high levels but has pleasant, sweet, and warm at very low levels (Reineccius, 2006). Additional errors in the perceived intensity of a GC peak can be due to masking in mixtures.

Finally, in-line condensation of some volatile compounds can result in persistent background odors. The GC column effluent splitter and the transfer lines to the sniffing port should be adequately heated to ensure odor free (Reineccius, 2006).

The evaluation of aroma profile from a single GC run has a drawback. Since it is impossible to precisely evaluate odor intensities during sniffing, the results cannot be used to distinguish key odorants from the little contributing compounds to the overall aroma. The number of the compounds detected depends on the amount of food used to isolate volatile compounds (Schieberle, 1995).

1.1 Aroma Extract Dilution Analysis (AEDA)

To overcome these problems and to gain some information on the odor activities of the odorants detected during sniffing, the aroma extract dilution analysis (AEDA) has been developed by Ullrich and Grosch (1987). This technique involves stepwise dilution (typically 1:2 or 1:3) of the prepared extract with a solvent and evaluation of each dilution by GCO. GCO is conducted up to the dilution that the odorants of interest cannot be perceived in the GC effluent. The highest dilution at which a substance can still be smelled is defined as its flavor dilution (FD) factor. For example, if 1 part of extract is diluted in 2 parts of solvent, the dilution factor is 3. During GCO, if an odor active compound is then detected in the fifth dilution, the FD factor becomes $3^5 = 243$ (or $\log_3$ D factor = 5). It is necessary to perform AEDA using both a polar (e.g. free fatty acid phase, FFAP) and a non-polar (e.g. silicone rubber, SE-54) stationary phases. This is because some odorants are better resolved and less adsorbed by one of the two types of the column (Grosch, 1993; Mistry *et al*., 1997).

The AEDA technique has been applied to determine flavor key constituents in the chromatogram of an extract (Schieberle and Grosch, 1991). This method is used to identified compounds responsible for olfactory differences between samples or differences between the sample and the reference, as in the off-flavor or the maturation studies (Schieberle and Grosch, 1992).

A critical evaluation of AEDA method has revealed the existence of 'gaps in coincident responses.' In this case, the odorant cannot be detected by the panelist at a given dilution level, but it can be perceived by the same panelist at a higher dilution (Guichard, *et al*., 1995). Hanaoka *et al.* (2000) proved that the panelist's breathing rate was statistically correlated with the risk of missing odorants, due to the interruption of smelling when breathing out. Moreover, the disadvantage of AEDA method is that several dilutions must be sniffed until no significant odor detected. This can take several days for one subject. Accounting for the differences between subjects by using several people and replicating their runs would take several weeks to complete (Acree, 1993). The AEDA method is screening methods. It does not absolutely indicate which compounds make the greatest contribution to the aroma of food. This is partly because during GCO of the dilution extracts, the odor compounds are volatilized and then evaluated. In foods, the volatility of aroma compounds depends on their solubility in the food matrix as well as their interactions with the nonvolatile constituents (Mistry *et al*., 1997).

AEDA is considered a screening tool for ranking odor active compounds in foods. It is based on the odor thresholds of compounds in air. In real life, the odor properties of these compounds are influenced by the food matrix. To indicate which of the compounds revealed by AEDA actually contribute to the aroma of a particular food, quantification of the odorants with higher FD factors and calculation of their OAVs are the next steps in the analytical procedure (Grosch, 1993).

1.2 Odor Activity Values (OAVs)

The ratio of the concentration of an odorant to its odor threshold gives the odor activity value (OAV). The OAVs are calculated on the basis of their odor thresholds in a medium predominating in the food of interest (Grosch, 1993). It was found that odorants with high OAVs are important contributors to characteristic flavors of off-flavors and are suitable as indicator substances for the objective determination of flavor differences in food (Grosch, 1993).

The odor threshold is the lowest concentration of a compound that is just enough for the recognition of its odor. The threshold values are frequently determined by smelling (orthonasal value) and by tasting the sample (retronasal value) (Belitz *et al*., 2004). Odor thresholds of key odor compounds are also determined by performing triangle tests nasally or retronasally. Samples of known concentrations of the odor active compound are prepared in a medium that predominates in the food. Then, two blanks and one sample containing known concentration of the odor active compound are presented as a set to a trained panel of at least five judges. These sets of samples are evaluated in decreasing order of concentration to determine the recognition threshold (Mistry *et al*., 1997).

The main limitation of OAVs is the lack of correlation between odor thresholds of compounds determined in air and in the medium that predominates in food. OAVs are mostly calculated on the basis of odor thresholds of single odorants in pure solvents. However, in the food system, the threshold values may be influenced by nonvolatile components such as lipids, sugars or proteins (Schieberle, 1995).

2. Gas Chromatography-Mass Spectrometry (GC-MS)

The high sensitivity inherent to MS (10-100 pg) and its compatibility with GC makes a GC-MS combination extremely valuable. Mass spectrometry is generally used in the flavor research to identify the unknowns and act as a mass-selective detector (Reineccius, 2006). GC-MS can be tentatively identified the unknowns by using a computer-assisted search of a MS spectra library. GC-MS can ascertain the purity of each peak as it elutes from the column. The purity of the peaks can be checked by taking mass spectra at very short increments of time (1 s or less). If a peak is pure, the mass spectra taken throughout the peak should be the same.

Flavor Encapsulation

Flavor plays an important role in consumer satisfaction and influences further consumption of foods. Flavor stability in different foods has been of increasing interest because of its relationship with the quality and acceptability of foods. Manufacturing and storage processes, packaging materials, and ingredients in foods often cause modification in overall flavor by reducing aroma compound intensity or producing off-flavor components (Lubbers *et al*., 1998). Dry packed foods are often formulated with added flavors to enhance their quality. However, many flavor components are volatile liquids which are susceptible to loss by evaporation, oxidation, or ingredient interactions. These effects can alter the quality of a food product, especially when packages are repeatedly opened. The problem can be minimized by the encapsulation of aroma components.

Encapsulation is the technique that a material or a mixture of materials is coated with or entrapped within another material system. The coated material is called active or core material, and the coating material is called shell, wall material, carrier, or encapsulant. The encapsulation of flavors serves to retain the aroma in a food product during storage, protect the flavors from undesirable interactions with food, minimize flavor/flavor interactions, guard against light-induced reactions and/or oxidation, and to effect on a controlled release (Anandaraman and Reineccius, 1986).

The flavor compounds being protected are locked up in a membrane that isolates it from the external medium. The size of microcapsule formed can vary from 1 to 1,000 μm. The simplest of the microcapsules may consist of a core surrounded by a wall of uniform or non-uniform thickness. The core material may be composed of just one or several different types of ingredients and the carrier may be single or multilayered (Madene *et al*., 2006).

The process for encapsulation of sensitive compounds consists of two steps: the first is often emulsification of a core material, such as the lipid-aroma system,

with a dense solution of a wall material such as a polysaccharide. The second is drying or cooling of the emulsions (Madene *et al*., 2006).

The Encapsulation Matrix

The initial step in encapsulating flavor compounds is the selection of a suitable coating material. An ideal coating material should have the following properties: (Shahidi and Han, 1993)

1. Good rheological properties at high concentration and ease of manipulation during the process of encapsulation.
2. Ability to disperse or emulsify the active material and stabilize the emulsion produced.
3. Nonreactivity with the material to be encapsulated both during processing and on prolonged storage.
4. Ability to seal and hold the active material within its structure during processing or in storage.
5. Complete release of the solvent or other materials that are used during the process of encapsulation, under drying, or other desolventization conditions.
6. Ability to provide maximum protection to the active material against environment conditions (e.g., heat, light, humidity)
7. Solubility in solvents acceptable in the food industry, e.g., water, ethanol, etc.
8. Chemical nonreactivity with the reactive material.
9. Ability to meet specified or desired capsule solubility properties and active material release properties.
10. Economy of food-grade substance.

In this study, maltodextrin and gum arabic were selected as wall material for CP encapsulation by spray drying.

1. Maltodextrin

Maltodextrins are formed by partially hydrolyzing flour or starch with acids or enzymes. They are supplied as different dextrose equivalents (DEs) with the average molecular weight decreasing as DE increases. The DE value is a measure of the degree of starch polymer hydrolysis. The value manifests the ability to form matrices that is important in forming wall systems (Shahidi and Han, 1993). In selecting the wall materials for encapsulation, maltodextrin is good compromise between cost and effectiveness. It is bland in flavor, has low viscosity at a high solid ratio and is available in different average molecular weights (Anandaraman and Reineccius, 1986). The major shortcomings of maltodextrins are the lack of emulsifying capacity and the low retention of volatile compounds (Reineccius, 1991).

The retention capacity changes significantly with the difference of DE values. Bangs and Reineccius (1981) showed that retention of twelve flavor compounds depends on the DE of the maltodextrins. Maltodextrins with DE 10 had the best retention and, as DE increased (DE 15, DE 20, DE 25, and DE 36.5), flavor retention decreased. The reason for the poor retention is often cited as their poor film-forming abilities. The wet encapsulation matrix must form a film around the droplets of active material and effectively retain them during the drying process while losing the water. It is considered that since maltodextrins have no emulsification properties, they produce coarse emulsions that result in poor flavor retention during drying (Reineccius and Risch, 1986).

On contrary, the flavor retention during storage increases with the DE values of maltodextrins (Yoshii *et al*., 2001). Maltodextrins vary greatly in protecting encapsulated ingredients from oxidation. Anandaraman and Reineccius (1986) showed that the use of high DE maltodextrins (36.5) as encapsulating agent gave a satisfying protection of the orange peel oil against oxidation by oxygen. These authors explained that high DE maltodextrins had abundant glucose monomer content, which could share hydrogen bond with oxidation products and exhibit an antioxidant effect. The product with the highest DE is extremely stable and would

have a shelf life of years without using an antioxidant (Anandaraman and Reineccius, 1986). It has been considered that the higher DE systems are less permeable to oxygen and therefore offer better protection to encapsulated flavor compounds (Reineccius, 1991). Desobry *et al.* (1997) confirmed this finding when examining the oxidation of β-carotene encapsulated in a maltodextrin matrix system.

2. Gum arabic

Gums and thickeners are generally bland or tasteless, but they can have a pronounced effect on the taste and flavor of foods. In general, hydrocolloids decrease sweetness, with much of the effect being attributed to viscosity and hindered diffusion (Godshall, 1997).

Gum arabic, or gum acacia, is a polymer consisting primarily of D-glucuronic acid, L-rhamnose. D-galactose, and L-arabinose, with about 5% protein. This protein fraction is responsible for the emulsification properties of the gum since it acts as an interface between oil and water (Shahidi and Han, 1993). Gum arabic is most often used as a flavor-encapsulating material. Its solubility, low viscosity, emulsification characteristics and its good retention of volatile compounds make it very versatile for most encapsulation methods. In addition, the wall material is ideally suited to the encapsulation of lipid droplets as it fulfils the roles of both surface-active agent and drying matrix, thus preventing the loss of volatiles in contact with the atmosphere.

However, gum arabic is an expensive ingredient, its production and costs are susceptible to climatic and political turbulence. Viscosity of concentrated solutions is sometimes troublesome for spraying; hence, there is a need to find total or partial substitutes (Turchiuli *et al*., 2005). Mixtures of gum Arabic and maltodextrins have shown promise as high solid carriers, giving acceptable viscosity in studies on microencapsulation of cardamom oil by spray drying (Sankarikutty *et al*., 1988). Thevenet (1995) tested four different ratios of acacia gum to maltodextrin (1:0, 1:1, 1:3, 1:5). An emulsion was prepared by mixing citrus oil (20% w/w of wall solids) into a hydrated gum/maltodextrin blend (40% solids w/w). The blend of maltodextrin

and gum acabic in the ratio of 1:1 gave high volatile retention. Spray-dried particles formed by mixtures of maltodextrins with gum arabic are typically 10-200 μm and the retention of the volatile material, which is normally > 80%, depends on a number of variables including the inlet temperature of the spray drier, the emulsion concentration and viscosity and the proportion of gum arabic to maltodextrins (Williams and Phillips, 2000).

Yoshii *et al.* (2001) studied flavor release from spray-dried maltodextrin/gum arabic or soy matrices as a function of storage relative humidity and showed that the release of ethyl butyrate decreased as the concentration of maltodextrin in the feed liquid increased. Apintanapong and Noomhorm (2003) used different ratios of gum arabic and maltodextrins to investigate the appropriate wall materials for encapsulation, by spray drying, 2-acetyl-1-pyrroline. These authors showed that the 70:30 combinations of gum arabic and maltodextrin gave the best quality capsules.

Encapsulation Techniques

Flavor encapsulation is accomplished by a variety of methods. The two major industrial processes are spray drying and extrusion (Goubet *et al.*, 1998). Freeze drying, coacervation and adsorption techniques are also used in the industry. In this study, the encapsulation method for preparation Thai chili paste microcapsules was spray drying. This technique is physical method. Physical processes for microcapsule formation involve the combination of core and coating materials by mechanical means in an environment that entraps the core by coating.

Spray Drying

Spray drying is the major process employed to produce dry flavorings. It was the first process used in the flavor industry to obtain encapsulated flavorings. The popularity of spray drying is due to the availability of equipments, low process cost, wide choice of carrier solids, good retention of volatiles, and good stability of the finished product (Reineccius, 1989).

Spray drying relies on physical entrapment of volatile flavors in a solid carbohydrate matrix to reduce their mobility. The goal in spray drying is to trap flavors within the solid walls of hollow spheres. During spray drying, highly volatile flavor components are less effectively encapsulated and more quickly lost than less volatile ones (King, 1990). According to Teixeira *et al.* (2004), this technique provides a high retention of aroma compounds during drying. Spray drying can be used for many heat-labile (low-boiling point) materials because of the lower temperatures that the core material reaches (Dziezak, 1988). A good spray-dried encapsulated flavor will not have a strong aroma, will flow freely, have a good shelf life and will disperse evenly and release flavor into the target food matrix (Baines and Knights, 2005).

Spray drying is the atomization of infeed liquid into a spray of droplets which contact hot air in a drying chamber and are converted into a powder. It can be considered as three sequential processes: emulsification, atomization, and drying.

1. Emulsification

The initial step is the selection of a suitable carrier. The ideal carrier should have good film former; have low viscosity at high solid levels (<500 cPs at ≥ 45 % solids levels), exhibit low hygroscopicity, release the coated flavor compounds when reconstituted in a finished food product, be low in cost, bland in taste, and stable in supply, and afford good protection to the encapsulated flavor compounds (Shahidi and Han, 1993).

Once a carrier of blend of carriers has been selected, it must be rehydrated in water. It is desirable to use a particular infeed solids level that is optimum for each carrier or combination of carriers. Research has shown that the infeed solids level is the most important determinant of flavor retention during the spray drying process (Reineccius, 1991). Increasing the solids level up to the point that the additional solids are no longer soluble benefits flavor retention by decreasing the required drying time to form a high solids surface film around the drying droplets. Once the droplet

surface reaches about 10 % moisture, flavor molecules cannot diffuse through this surface film, whereas the relatively smaller water molecules continue to diffuse through this surface film and are lost to the drying air (Menting *et al*., 1970; Kerkhoff and Thijssen, 1974).

A high infeed solids level increase viscosity of the emulsion. When the emulsion has high viscosity, the circulatory movement in the droplets is retarded resulting in a semipermeable membrane forms quickly and thus favors flavor retention. It is possible to pump and atomize infeed materials that contain carrier solid in excess of the solubility limits. Insoluble solids offer no barrier to the diffusion of flavor molecules and thus do not improve flavor retention during drying. It has been found that there is an optimum infeed solids level that is unique to each carrier (Reineccius and Bangs, 1985).

When the carrier has been solubilized, with or without heating, the core system must be added. Traditionally, a 20 % flavor load based on carrier solids is used in spray drying (Shahidi and Han, 1993). The encapsulation efficiency is diminished at higher than 20% volatile load (Bhandari *et al*., 1992). In practice, most experiments reported of using the ratio of carrier and core to 4:1 of support to volatile products (Reineccius, 1988). This ratio is the optimum for support materials like gum arabic and other carbohydrate derivatives. An increase in volatiles retained on the surface of powder particles was observed when the volatiles load was increased from 20% to 25% (Bhandari *et al*., 1992). High flavor loads typical result in unacceptably high losses of flavors in the dryer. Flavor retention decreases with increasing amounts of core-material can be explained in two different ways. First, at higher core-to-shell material ratios, there was less protection afforded for each core-material droplet within the microcapsule. Secondly, the core-material solubilized in the continuous phase may interfere with the drying process and lead to an inferior slid shell (Zilberboim *et al*., 1986).

During emulsion preparation, the mixture must be homogenized prior to spray drying. The emulsion created should have small oil droplets to improve its

stability and prevents droplets coalescence during the drying process. It appears advantageous to efficiently homogenize the infeed material. Water-soluble materials also may be encapsulated by the treatment of homogenization. Instead of having a clearly defined core and coating, the product consists of a homogeneously blended matrix of the polymer entrapping the core. These products are sometimes described as matrix particles or entrapped ingredients. They also are said to be covered with a very fine film of coating (Shahidi and Han, 1993). Reineccius (1988) suggested that a smaller emulsion size yields larger percentage retention of the orange oil with a smaller amount of surface oil, but did not produce a longer shelf life. In addition, Sheu and Rosenberg (1995) showed that the retention of volatiles during microencapsulation by spray drying could be enhanced by reducing the mean emulsion size of the dispersed core material during emulsification. Moreover, it has been reported that aroma compounds are better retained in the smaller emulsion particles than in the larger ones, and consequently, the flavor should evaporate more easily form large emulsion particles during atomization (Soottitantawat *et al*., 2005).

2. Atomization

Two types of atomizers are widely used in industrial manufacturing: the single fluid, high pressure spray nozzle and the centrifugal wheel. Atomization parameters have a significant effect upon the particle size distribution of resultant powders (Shahidi and Han, 1993). Particle size is insignificant if high infeed solids are used (Bomben *et al.*,1973). Although particle size may have a minimal influence on flavor retention during, it is often desirable to produce large particles to aid in dispersion upon reconstitution. Small particles are often difficult to disperse and float on the liquid surface (Jones, 1988).

From a practical point of view particle shape is as important as particle size and drying conditions which yield spherical particles and less of dents will improve the flowability of the finished powder. Spherical particles also pack together better and this increases bulk density making the flavor more compact, less dusty, and easier to bag off and transport. The spherical particles formed in spray drying are not

completely round but are usually partially collapsed taking on the appearance of deflated footballs (Baines and Knights, 2005). Particles that are not spherical are produced when infeed viscosity is too high, atomizer rotation is too slow, or inlet air flow is not high enough. Particle shape does not have a great bearing on flavor retention because the oil-phase flavor material is dispersed throughout the spray-dried particles as fine droplets encased in a honeycomb-like structure (Baines and Knights, 2005). When gum arabic and maltodextrin are used in equal proportions, cracks are not observed. This could be due to the drying conditions employed especially the rate of drying. However, maltodextrin showed an increasing tendency for the development of cracks with increase in concentration even in combination with gum arabic up to 60% level in the mixture (Sankarikutty, *et al*., 1988).

3. Drying

As the atomized particles fall through the gaseous medium, they assume a spherical shape, with the oil encased in the aqueous phase. The rapid evaporation of water from the coating during its solidification keeps the core temperature below 100 °C in spite of the high temperatures used (Brenner, 1983). The particles exposure to heat is in the range of a few seconds at most. Thus, the main advantage of this method is its ability to handle many heat-labile materials. However, because any one material, such as flavor, may contain as many as 20 to 30 different components (alcohols, aldehydes, esters, and ketones) with boiling points ranging form 38 °C to 180 °C, it is possible to lose certain low boiling point aromatics during the drying process (Taylor, 1983).

Flavor compounds can leave the drying droplets before the formation of the crust on the droplets' surface. Further losses can occur by diffusion in the crust solid or through the pores or channels (Rė, 1998). The type of solids, its concentration, and drying temperatures are very important for crust formation. The diffusion coefficients of water and volatiles are reduced as water decreases upon drying. As a result of differences in molecular weight between water and volatiles, the reduction in the diffusivity of the volatiles is more pronounced than that of water. Once the crust has

formed, volatiles diffusivity is so low that the volatiles are entrapped in the drying solid matrix, while water can still diffuse through the crust. The crust therefore becomes effectively a selective membrane (Bylaite *et al*, 2001). An air inlet temperature is selected which favors the formation of a diffusion-resistant semipermeable membrane over the surface of the droplet during the transition between it being an aqueous emulsion and a powder. Water evaporates from the surface of the droplet to create a denser area and a transient membrane which continues to allow the passage of water vapor with a minimum loss of volatile flavor chemicals. The formation of the membrane is dependent on the infeed solids level and the nature of the carriers being used, and it acts to deter the evaporation of flavor molecules by size exclusion. The larger the flavor molecule, the slower the rate of diffusion through the membrane (Rosenberg *et al*., 1990).

The loss of volatiles during the spray drying process can be explained by a mechanism of selective permeability as mention above. This assumes that the retention of the compounds in the atomized drop is a function of parameters related to the relative volatility of the compounds. After the skin has formed, other factors become preponderant in the parameters that control the phenomenon of diffusion through the wall (Bertolini *et al*., 2001). The vapor pressure of the flavor molecule also has a bearing on volatile losses but is considered to be secondary to molecular weight. Thus, low molecular weight flavor compounds with high vapor pressures are lost more readily in spray drying than high molecular weight flavor compounds with lower vapor pressures. The degree of aroma retention is strongly dependent on the moisture content of the final microcapsules and on the humidity of the exhaust air (Madene *et al.,* 2006). On the other hand, the greater polarity, and consequently the greater solubility of the encapsulated compound in an aqueous medium, results in a greater capacity for diffusion through the matrix during the spray drying process, leading to grater losses during the formation of the capsules (Rosenberg *et al*., 1990).

Losses of non-volatile compounds may also occur during drying of the drops. Droplets of non-volatile substances that present at the surface of the drops

leaving the atomizer, or those that migrate to the surface prior to the formation of dry crust around the drying particles, may be swept off the particle surface (Rė, 1998).

Flavor retention is influenced by inlet and exit air temperatures. In general, a high enough inlet air temperature should be used to allow rapid formation of a semipermeable membrane on the droplet surface but yet not so high as to cause heat damage to the dry product or "ballooning" of the drying droplet. Ballooning occurs when steam is formed in the interior of the drying droplet, causing the droplet to puff. The result is a thin-walled, hollow particle that will not retain flavor compounds as well as the non-ballooned counterpart (Verhey, 1972).

The disadvantages of spray drying are that some low-boiling point aromatics can be lost during spray drying and the core material may be on the surface of the capsules. Flavor compounds on the surface could be oxidized and cause flavor changes of the encapsulated product (Dziezak, 1988; Desobry, 1997). Other problems with spray drying are that this technology produces very fine powders, typically in the range of 10-100 μm in diameter. In this case, agglomeration is needed to make the dried material more soluble if it is a liquid application. Agglomeration of spray-dried powders can be accomplished by using fluidized bed process.

Fluidized-bed spray coating is a three-step process. First, the particles to be coated are fluidized in the hot atmosphere of the coating chamber. Then, the coating material is sprayed through a nozzle onto the particles and film formation is initiated, there follows a succession of wetting and drying stages. The small droplets of the sprayed liquid spread onto the particle surface and coalesce. The solvent or the mixtures is then evaporated by the hot air and the coating material adheres on the particles. This technique relies upon a nozzle spraying the coating material into a fluidized bed of aroma particles in a hot environment. The size of the product varies from 0.3 to 10 mm (Jacquot and Pernetti, 2003).

Controlled Flavor Release

Controlled release is a novel technology that can be used to increase the effectiveness of many ingredients and is defined as a method by which one or more active agents or ingredients are made available at a desired site, at a specific time, and a specific rate. With the emergence of controlled-release technology, heat-, temperature-, or pH-sensitive additives can be conveniently used in food systems (Pothakamury and Barbosa-Canovas, 1995). Moreover, the retention of flavor is governed by factors related to the chemical nature of the core, including its molecular weight, chemical functionality, polarity and relative volatility, to the wall material properties and to the nature and the parameters of the encapsulation technology. The entire core material will be within the wall. Controlling the volatiles on the particle surface is important, since any volatiles not encapsulated but absorbed on the surface of the encapsulation matrix, is subject to evaporation and/or oxidation. Therefore, powders with high levels of surface core may be associated with poor stability (Bangs and Reineccius, 1990).

Flavor matrix systems encapsulating volatile compounds, release depends on several mutually dependent processes such as diffusion of the volatile compound through the matrix, type and geometry of the particle, transfer from the matrix to the environment, and degradation/dissolution of the matrix material (Pothakamury and Barbosa-Canovas, 1995). De Roos (2000) showed that two factors control the rate of flavor release from products, the comparative volatility of the aroma compounds in the food matrix and air phases under equilibrium conditions (thermodynamic factor) and the resistance to mass transport from product to air (kinetic factor). The mechanism of release for the capsule may be based on solvent effects, such as melting, diffusion, degradation, or particle fracture.

The advantage of controlled release are: the active ingredients are released at controlled rates over prolonged periods of time; loss of ingredients during processing and cooking can be avoided or reduced; reactive or incompatible components can be separated (Dziezak, 1988). Furthermore, the release characteristics of encapsulated

flavors from the powder are quite important for estimating the storage period, as well as the controlled release applications in food.

1. Release of Flavor by Diffusion

Diffusion is controlled by the solubility of a compound in the matrix and the permeability of the compound through the matrix. Diffusion is important in food because it is the dominant mechanism in controlled release from encapsulation matrices (Cussler, 1997). The vapor pressure of a volatile substance on each side of the matrix is the major driving force influencing diffusion (Gibbs *et al*., 1999). The principal steps in the release of a flavor compound from matrix system are: diffusion of the active agent to the surface of the matrix; partition of the volatile component between the matrix and the surrounding food and transport away from the matrix surface (Fan and Singh, 1989). It should be obvious that if the food component is not soluble in the matrix, then it will not enter the matrix and so diffusion will not take place irrespective of the pore size of the matrix (Reineccius, 1995).

For most physical methods, it is known that the success of encapsulation depends on the formation of a metastable amorphous structure, a glass, with a very low permeability to the organic compounds that are encapsulated within it. In drying processes, the presence of sugar and/or polymers in the encapsulation system reduces the water content. Reduction of water content lowers the glass transition temperature, and the resulting amorphous matrix is impermeable to organic compounds as well as to oxygen. Permeability to water, however, remains finite. This selective diffusion theory of Rulkens and Thijssen (1967) is the basis for encapsulation using spray drying and freeze drying (Karel and Lager, 1988). In spray drying, upon droplet formation rapid evaporation from the surface produces a surface layer in which the selective diffusion mechanism comes into play. In freeze drying, upon water crystallization the nonfrozen solution is viscous and the diffusion of core materials is retarded. At the beginning of freeze drying, the surface of this solution becomes an amorphous solid in which selective diffusion comes into play.

The release of flavor from the spray-dried powder may be considered to be associated with the diffusion mechanisms of both flavor and water, because the solubilization of the wall matrixes with water would be followed by subsequent release of the encapsulated flavor (Soottitantawat *et al.*, 2004). Water uptake at high relative humidity (RH) destroys the capsule structure. The release of microencapsulated ethyl butyrate is closely related with the adsorption of water in the wall materials and hydration of the powder. At the initial stage, water penetrates the surface wall of the spray-dried particle and destroys the microcapsule structure, which leads to the release of the microencapsulated ethyl butyrate (Yoshii *et al*., 2001). Anker and Reineccius (1988), who monitored the formation of limonene oxides in encapsulated spray dried orange peel oil stabilized in the a_w range of 0.001 to 0.536, finding that the slowest rates of formation of limonene oxide occurred at water activities of 0.536. The effect of RH on the release of volatile materials indicates that high retention is maintained as long as the individual structures of the capsule are intact (Rosenberg *et al*., 1990). The rate of release and oxidation of encapsulated D-limonene by spray drying is closely related to the RH and the structure changes (Soottitantawat *et al*., 2004).

Whorton and Reineccius (1995) studied release of volatile compounds from spray-dried maltodextrin and corn syrup solids microcapsules. Water activity varied from a_w of 0.11 to 0.75, and loss of volatiles was followed by gas chromatographic headspace analysis. The content of volatiles in the powder was also quantified. The results showed that flavor loss increased with increasing a_w until structure collapse took place. After collapse, volatile release decreased. The authors concluded that the high release typically occurred between the glass transition temperature and complete collapse. Full collapse was claimed to effectively re-encapsulate the flavors.

In microencapsulation products, the diffusion of molecules has been related to a glass transition phenomenon (Roos, 1995). When the matrix has been rapidly dehydrated and a low water activity has been reached, as for example after spray drying or freeze drying, it is believed that carriers are in the glassy state (Whorton and Reineccius, 1995). This is characterized by a very low mobility of the carrier

molecules. Due to this low mobility, release of the materials is primarily via Fickian diffusion through the pores in the matrix. The amount of volatile released will also depend on the composition of the matrix, pore size, particle size, and the thickness and area of wall around the entrapped volatiles. As a consequence, while the matrix is in the glassy state, release or retention of volatiles depends more upon the rate at which aroma can migrate to the surface than upon the relative volatility of the aroma (Whorton, 1995).

When the plasticizer content, for example water content, increases, a transition occurs from the solid glassy to a liquid-like rubbery state. The hydrogen bonds which are responsible for the main structural forces in dried amorphous products are weakened. Molecular mobility and diffusion increase (Whorton, 1995). The swelling and bridging of the particles or dissolution of the wall polymer can occur depending on the water uptake (Rosenberg *et al*., 1990). The larger volatiles losses are observed at this state of the carrier than the glassy or collapsed ones. These variations in aroma retention could be explained by the destruction of microregions immobilizing the volatiles and by the diffusion of aroma compounds (Rifai and Voilley, 1991). With increasing water content, collapse may occur when plasticization of the carrier decreases the viscosity to the extent that the polymer matrix is unable to support itself against gravity. When it happens, porosity is lost, reducing diffusion through the matrix. In some case, collapse can result in reencapsualation of the aroma compound (Whorton and Reineccius, 1995).

2. Release of Flavor by Degradation

The coating can be fractured or broken open by external forces, such as pressure, shearing, or extractions, or by internal forces, such as would occur in a microcapsule having a permeation-selective coating (Shahidi and Han, 1993).

The release of an active compound from a matrix-type delivery system may be controlled by diffusion, erosion or a combination of both. Homogeneous and heterogeneous erosion are both detectable. Heterogeneous erosion occurs when

degradation is confined to a thin layer at the surface of the delivery system, whereas homogenous erosion is a result of degradation occurring at a uniform rate throughout the polymer matrix (Pothakamury and Barbosa-Canovas, 1995).

3. Release of Flavor by Swelling

In system controlled by swelling the flavor dissolved or dispersed in a polymeric matrix is unable to diffuse to any significant extent within the matrix. When the matrix polymer is placed in a thermodynamically compatible medium, the polymer swells because of absorption of fluid from the medium. The aroma in the swollen part of matrix then diffuses out (Fan and Singh, 1989).

4. Release of Flavor by Melting

This mechanism of release involves the melting of the capsule wall to release the active material. This is readily accomplished in the food industry as there are numerous approved materials for food use that can be melted (e.g., lipids, modified lipids, and waxes). In such applications, the coated particles are stored at temperature well below the melting point of the coating. Aroma is released when the particles are heated to above the melting points during preparation or cooking (Sparks *et al*., 1995).

MATERIALS AND METHODS

Materials

1. Samples

Chili powder, dried shrimp, and shrimp paste were obtained from a local market in Bangkok, Thailand. Garlic, shallot, tamarind paste, fish sauce, salt, coconut sugar, and soybean oil were purchased at a local market in Urbana, IL.

2. Chemical Reagents

2.1 Diethyl ether (anhydrous, 99.8%) (Aldrich Chemical Co. Ltd., USA)

2.2 Hexane (HPLC grade) (Labscan Asia Co. Ltd., Bangkok, Thailand)

2.3 Methanol (HPLC grade) (Aldrich Chemical Co. Ltd., USA)

2.4 Etahnol (Absolute GR, Merck, Darmstadt, Germany)

2.5 Sodium chloride (99%) (Aldrich Chemical Co. Ltd., USA)

2.6 *n*-Alkane standards (C6-C30) (Aldrich Chemical Co. Ltd., USA)

2.7 2-Methyl-3-heptanone (Aldrich Chemical Co. Ltd., USA)

2.8 2-Ethylbutyric acid (Aldrich Chemical Co. Ltd., USA)

2.9 2,4,6-Trimethyl pyridine (Aldrich Chemical Co. Ltd., USA)

2.10 Sodium sulfate anhydrous (granular) (Fisher Scientific, UK)

2.11 Sodium hydroxide (98.7%) (Fisher Scientific, UK)

2.12 Hydrochloric acid (36.5%) (Fisher Scientific, UK)

2.13 Deodorized distilled water

2.14 Authentic standard compounds (Aldrich; Sigma, USA; Fluka, Germany; Lancaster, USA; Mallinckrodt Baker Inc., USA; Bedoukian Research Inc.; Alfa, USA; Pfaltz/Bauer, USA; Firmenich Co.; USA)

2.15 Gum arabic (Jumbo Trading Co., Ltd., Bangkok, Thailand)

2.16 Maltodextrin DE 18.5 (D-Perse 4, Siam Modified Starch, Co., Ltd, Pathumthani, Thailand)

2. Chemical Reagents (Continued)

2.17 Hayashi solvent FM containing 26% methanol and < 0.2 mg H_2O/mL (Hayashi Pure Chemical Ind., Co., Ltd., Osaka, Japan)

2.18 Hydranal composite solvent (Riedel-deHaën, Sigma-Aldrich, Seelze, Germany)

2.19 Nitrogen gas (ultra high purity)

2.20 Helium carrier gas (ultra high purity)

2.21 Hydrogen (ultra high purity)

2.22 Air zero grade

2.23 Liquid nitrogen

3. Equipments and Instruments

3.1 Ultra Turrax Ika T25 basic homogenizer (Janke & Kunkel GmbH & Co., Germany)

3.2 Three-neck glass purge and trap vessel (280-mL volume, jacketed; Scientific Instrument Services, USA)

3.3 Adsorbent trap (Tenax TA 60/80, 200 mg/trap; Supelco, USA)

3.4 Heating and refrigerated circulator (Lauda RM6, Brinkmann, Canada)

3.5 Condensers

3.6 Hot air oven (Memmert, Germany)

3.7 Hot plate stirrer (SS10, Stuart Scientific, UK)

3.8 Roto mix (type 50800, Thermolyne, USA)

3.9 Solvent assisted flavor evaporation (SAFE) system

3.10 High vacuum distillation system equipped with high vacuum pump (BRV10MK, Edwards, UK) and rotary pump (RV5, Edwards, UK)

3.11 Gas chromatography 6890 (Agilent Technologies Inc., USA) equipped with a FID, a cooled injection system (CIS4, Gerstel GmbH & Co., Germany), thermal desorption system (TDS2, Gerstel) and an olfactory detector port (ODP2, Gerstel)

3. Equipments and Instruments (Continued)

3.12 Gas chromatography 6890 (Agilent Technologies Inc., USA) equipped with a FID, an on-column injector and an olfactory detection port (DATU Technology Transfer, USA)

3.13 Gas chromatography 6890 - mass selective detector (MSD 5973; Agilent Technologies Inc., USA)

3.14 Gas chromatography 6890N (Agilent Technologies Inc., China) equipped with a FID, and thermal desorber (QUI-0002 version 5.1, Unity Markes International Limited, UK)

3.15 Spray dryer (Mobile Minor™, Niro Inc., USA)

3.16 Water activity meter (Testo 650, Testo Inc., Germany)

3.17 Differential scanning calorimetry (Pyris 1 DSC, Perkin Elmer, USA)

3.18 Karl Fischer Titration (787 KF Titriho, Metrohm, Switzerland)

3.19 Vacuum oven (VOS-450SD, Eyela, Tokyo, Japan)

3.20 Centrifuge refrigerator (RC 50 plus, Sorvall®, Ct, USA)

3.21 Shaking water bath (Memmert, Schwabach, Germany)

3.22 Incubator (Memmert, Germany)

3.23 Rotary evaporator (R114, Buchi, Switzerland)

3.24 Glasswares; prior to use, all glasswares were baked at 120 °C for at least 2 h.

Methods

1. Sample Preparation

The composition of unheated CP (UH-CP) is presented in Table 3. The procedure of Pojjanapimol *et al.* (2004) was used for the heating of CP. Two hundred grams of the mixed ingredients of CP (Table 3) was placed in a one-litre, three-neck round bottom flask and closed with thermometer, condenser (0 °C), and PTFE stirrer blade assembly (Figure 7). The mixture was heated at 100 °C by using a heating mantle and stirred at 50 rpm for 25 or 50 min to produce H25-CP and H50-CP,

respectively. The mixture was immediately cooled in an ice-water bath. The CPs (200 g of mixed CP ingredients) were placed in Corpak clear standard wide-mount bottles and sealed with Teflon-lined closures and then covered with aluminium foil. Samples were prepared in duplicate and kept at -70 °C until further analysis.

1.1 Compositional analysis of CPs.

Moisture, lipid, protein, ash and crude fiber were determined according to AOAC (2000).

Table 3 Composition of Thai Chili Paste

Ingredients	Percent (w/w)
chili powder	3.74
ground garlic (*Allium sativum* Linnaeus)	16.26
ground shallot (*Allium ascalonicum* Linnaeus)	19.51
minced dried shrimp	4.88
tamarind paste	4.06
fish sauce	2.44
salt	0.33
kaphi (shrimp paste)	5.69
coconut sugar	23.58
soy bean oil	19.51

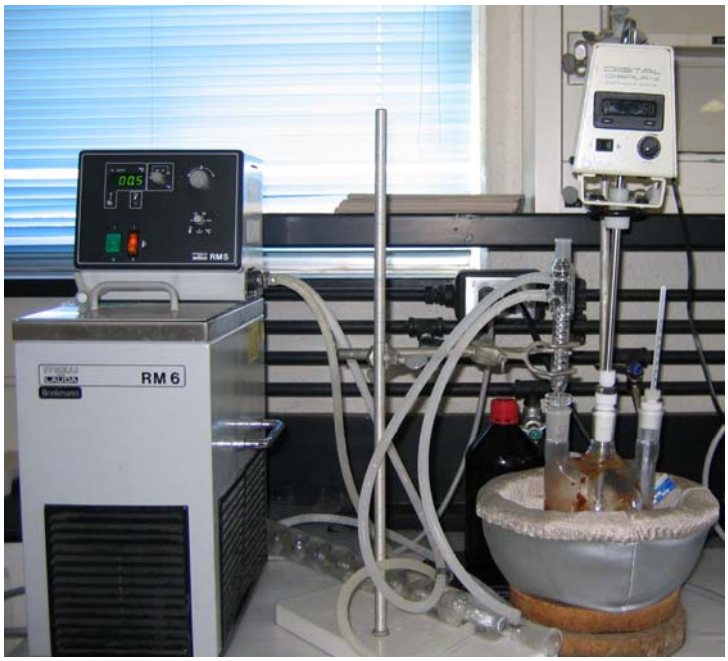


Figure 7 Apparatus used for heating of chili paste

2. Isolation, Determination and Quantification of Aroma Active Compounds in Chili Paste

2.1 Dynamic headspace dilution analysis (DHDA)

A mixture of 5 g of CP, 45 mL of deodorized distilled water, and 5 µL of an internal standard solution (containing 1.62 µg/µL of 2-ethyl butyric acid, 2.44 µg/µL of 2,4,6-trimethyl pyridine and 1.25 µg/µL of 2-methyl-3-heptanone in methanol) was homogenized at 14,000 rpm for 1 min using an Ultra Turrax Ika T25 basic homogenizer (Janke & Kunkel GmbH & Co., Germany). Five milliliter of the mixture was placed in the three-neck glass purge and trap vessel (280-mL volume, jacketed; Scientific Instrument Services, Ringoes, NJ) (Figure 8). The volatiles were purged onto an adsorbent trap (Tenax TA 60/80, 200 mg/trap; Supelco, Bellefonte, PA) using nitrogen (flow rate 20 mL/min, ultra high purity). The vessel was maintained at 25 ºC by circulating water through the external water jacket. DHDA was performed by varying the headspace purge times (25, 5, and 1 min), as described by Cadwallader and Baek (1998). After purging, the desorption tube was removed

from the vessel and dry purged with nitrogen gas (flow rate 50 mL/min) for approximately 5 min at room temperature to remove any moisture in the trap.

The thermal desorption system (splitless-mode; TDS2, Gerstel GmbH & Co. KG, Germany) was employed to desorb the volatiles from Tenax trap. This system was connected to a 6890 GC (Agilent Technologies Inc.) equipped with a cooled injection system (CIS4, Gerstel), a flame ionization detector (FID) and an olfactory detector port (ODP2, Gerstel). Volatiles were desorbed at 220 ºC into the CIS4 inlet held at -150 ºC (solvent vent mode, 50 mL/min helium vent flow) followed by cold splitless injection (inlet heated at 12 ºC/s from -150 to 260 ºC with final hold time of 10 min; purge valve delay was 1.10 min) of the desorbed volatiles into the GC column.

Figure 8 Purge and trap vessel for dynamic headspace sampling of chili paste

Separations were performed on a polar capillary column (Stabilwax-DA, 15 m x 0.32 mm i.d.; 0.5-µm film; Restek, Bellefonte, PA) and a non-polar column (DB-5MS, 15 m x 0.32 mm i.d.; 0.5-µm film; J&W Scientific, Folsom, CA). The GC oven temperature was programmed from 30 ºC to 225 ºC at a rate of 10 ºC/min with initial and final hold times of 5 and 15 min, respectively. Helium was used as a carrier gas at a constant flow rate of 2.1 mL/min. The FID and sniffing port temperatures were 250°C. To prevent drying of the nasal mucosa, humidified air was supplied to the sniff port at 30 mL/min. The end of the capillary column was split between FID and sniff port in the ratio of 1:1 using deactivated fused silica capillaries (1 m x 0.32 mm i.d.; Restek). Analysis of each dynamic headspace dilution was carried out in triplicate and GCO was performed by two experimented sniffers. A flavor dilution factor was calculated for each odorant by dividing the highest purge time tested (25 min) by the purge time at which it was last detected by GCO (e.g., either 25, 5, or 1 min) (Avsar *et al.*, 2004).

2.2 Direct solvent extraction and solvent assisted flavor evaporation

A CP sample (20 g) was weighed into a 250 mL Teflon bottle equipped with Tefzel closures. The extraction was prepared in duplicate. Deodorized distilled water (100 mL) and 20 µL of an internal standard solution (containing 1.62 µg/µL of 2-ethyl butyric acid, 2.44 µg/µL of 2,4,6-trimethyl pyridine and 1.25 µg/µL of 2-methyl-3-heptanone in methanol for acidic, basic, and neutral fractions, respectively) were added to the bottles. The mixture was homogenized at 14,000 rpm for 1 min using an Ultra Turrax Ika T25 basic homogenizer. Diethyl ether (90 mL) and 10 g of sodium chloride (99%) were added to the bottles. The mixtures were agitated for 30 minutes on a Roto mix (Thermolyne, type 50800; Dubuque, IA) at 200 rpm. The bottles were then centrifuged at 3000 rpm for 10 min in order to separate the solvent phases, which were subsequently collected into round bottom flasks. The procedure was repeated two more times with 90 mL of diethyl ether. The solvent phases were combined and then dried over anhydrous sodium sulfate. The extract was concentrated to 100 mL by distillation using a Vigreux column at 40 ºC.

Separation of volatile compounds from CP extract was achieved by using solvent assisted flavor evaporation (SAFE) (Figure 9). The SAFE system was employed to isolate the volatile components of a complex matrix as in the case of CP. This system was selected due to its higher yields of volatile components from fatty matrices (Engel *et al.,* 1999) and reduced thermal alteration of the flavor components of CP during the isolation. The system consisted of two liquid nitrogen-cooled traps (receiving and waste traps), a transfer head and a 1-L round bottom flask. The whole system was operated under high vacuum (approx. 10^{-5} Torr). The CP extract (100 mL) was fed through the upper part of the transfer head and separation occurred when aliquots of the sample were dropped into the round bottom flask partially submerged in a mild (50 ˚C) water bath. The extraction time was approximately 2.5 h. The separated volatiles traveled through the outlet line of the separation head towards the receiving tube, where they were condensed and frozen due to the sudden changed of temperature. The tube was then removed and thawed to proceed with the fractionation of the CP extract.

Figure 9 Solvent assisted flavor evaporation (SAFE)

To separate the acidic, basic and neutral fractions from the extract, the SAFE extract was first concentrated to 30 mL by distillation using Vigreux column at 40 ˚C and then placed in a 125-mL separatory funnel. The extract was washed with aqueous NaOH (1 mol/L, 3 x 30 mL), and the organic layer containing the neutral and basic volatiles was collected. The aqueous layer was washed with diethyl ether (2 x 30 mL) and then acidified to pH 2 with 10% (w/v) aqueous HCl and saturated with NaCl. It was then extracted with diethyl ether (3 x 20 mL). The ether layer containing the acidic volatiles was washed with saturated NaCl (2 x 30 mL).

The organic layer containing neutral and basic volatiles was washed with saturated NaCl (2 x 30 mL) and then extracted with aqueous HCl (1 mol/L, 3 x 30 mL). The organic layer containing the neutral volatiles was collected and was washed with saturated NaCl (2 x 30 mL). The aqueous layer containing basic volatiles was washed with diethyl ether (2 x 30 mL) and then adjusted to pH 9 with 10% NaOH solution, and saturated with NaCl. The mixture was then extracted with diethyl ether (3 x 20 mL). The ether extract was washed with saturated NaCl (2 x 30 mL) and the ether layer containing the basic volatiles was collected.

Each fraction from above was concentrated under a gentle stream of nitrogen gas to 10 mL, dried over anhydrous Na_2SO_4, and further concentrated to 100 µL under a nitrogen gas stream. Samples were prepared in duplicate and kept at -70 ˚C until analysis.

2.3 Aroma extract dilution analysis (AEDA)

Each of the original organic fractions was diluted sequentially with diethyl ether in a ratio of 1:3 according to the aroma extract dilution analysis technique (Grosch, 1993). Each dilution was kept in a 200-µL glass insert put inside a 1-mL amber vial equipped with PTFE-lined screw cap and kept at -70 ˚C until analyzed. This method was employed to detect the potency of the odor-active compounds in each of the organic fractions. The number of serial dilutions analyzed

for each fraction depended on the potency of the compounds and it was carried out until no odor was detected.

The GCO system consisting of a 6890 GC (Agilent Technologies) equipped with a FID, an on-column injector and an olfactory detection port (DATU Technology Transfer, Geneva, NY). Each aroma extract was injected by cool on-column mode (+3 ˚C temperature tracking mode) into a polar capillary column (DB-Wax, 15 m x 0.32 mm i.d.; 0.5-µm film; J&W Scientific) or a non-polar column (RTX®-5SILMS, 15 m x 0.32 mm i.d.; 0.5-µm film; Restek). Column effluent was spit 1:1 between FID and olfactory detection port using Siltek deactivated fused silica tubing (1 m x 0.32 mm i.d.; Restek), with both detector temperatures held at 250 ºC. The GC oven temperature was programmed from 35 ºC to 225 ºC at a rate of 8 ºC/min with initial and final hold times of 5 and 15 min, respectively. Helium was used as a carrier gas at a constant flow rate of 2.2 mL/min. The sniffing port was supplied with humidified air at 30 mL/min. Two experienced sniffers evaluated the neutral, basic, and acidic fractions of CP extract two times on the two different columns (Cadwallader *et al.*, 1995).

2.4 Dynamic headspace analysis/gas chromatography-mass Spectrometry (DHA/GC-MS)

System used for DHA/GC-MS consisted of a thermal desorption system (TDS2, Gerstel) a CIS4 inlet (Gerstel), and a 6890 GC/5973N mass selective detector (MSD; Agilent Technologies). The conditions for thermal desorption and injection was the same as previously described for DHDA.

Separations were performed using either a polar capillary column (Stabilwax-DA, 30 m x 0.25 mm i.d.; 0.5-µm film; Restek) or a non-polar column (RTX®-5MS, 30 m x 0.25 mm i.d.; 0.5-µm film; Restek). The oven temperature was programmed from 35 °C to 225 °C at a rate of 6 °C/ min with an initial and final hold times of 5 and 10 min, respectively. Helium was used as carrier gas at a constant rate of 1.0 mL/min. The MSD conditions were as follows: capillary direct interface

temperature, 280 °C; ionization energy, 70 eV; mass range, 35 to 300 amu; electron multiplier voltage (autotune + 200 V); scan rate, 5.27 scans/s.

2.5 Gas chromatography-mass spectrometry

GC-MS of the organic fractions obtained by SAFE were injected using a cool on-column method (+3 ˚C temperature tracking mode) into a 6890 GC/5973N MSD (Agilent Technologies). Separations were performed using the columns described above for DHA/GC-MS. The oven was programmed from 35 °C to 225 °C at a rate of 4 °C/ min with an initial and final hold times of 5 and 20 min, respectively. Helium was used as carrier gas at a constant rate of 1.0 mL/min. The mass selective detector conditions were the same as described for DHA/GC-MS analysis.

2.6 Identification of odorants

For positive identification, retention indices, mass spectra, and odor properties of unknowns were compared with those of authentic standard compounds analyzed under identical conditions. Tentative identifications were based on comparing mass spectra of unknown compounds with those in the National Institute of Standards and Technology (NIST, 2005) mass spectral database or by matching the retention indices values and odor properties of unknowns against those of authentic standards. For linear retention indices (LRI) calculation, a homologous series of *n*-alkanes (C6-C30 and C6-C22 for polar and non-polar column, respectively) were analyzed under the same chromatographic conditions. The linear retention indices (LRI) or retention index (RI) was calculated followed by (Van den Dool and Kratz, 1963) as showed in Appendix.

2.7 Quantification of selected compounds

An aqueous mimic matrix for standard solutions was composed of 21.67% w/w sucrose, 0.96% w/w fructose, 0.95% w/w glucose, 19.51% w/w fresh soybean oil, and 0.33% w/w NaCl in odorless water with pH adjusted to 5.96 using

10% aqueous HCl. The sugar contents of the metrix imitated the composition of coconut sugar reported by Apriyantono *et al.* (2002). The stock solution for quantification contained 813 µg of dimethyl sulfide, 972 µg of allyl mercaptan, 177 µg of 2-methylbutanal, 247 µg of 3-methylbutanal, 372 µg of allyl methyl sulfide, 408 µg of 2,3-butanedione, 550 µg of dimethyl disulfide, 461 µg of 3,3'-thiobis-1-propene, 521 µg of methyl propyl disulfide, 868 µg of 1,3-dithiane, 472 µg of dipropyl disulfide, 550 µg of dimethyl trisulfide, 509 µg of 3-ethly-2,5-dimethylpyrazine, 735 µg of methional, 1265 µg of diallyl disulfide, 368 µg of benzaldehyde, 545 µg of butanoic acid, 401 µg of phenylacetaldehyde, 481 µg of 3-methylbutanoic acid, 982 µg of phenol, 1334 µg of 2,5-dimethyl-4-hydroxy-3-(2*H*)-furanone(DMHF), and 443 µg of phenylacetic acid per mL in methanol. Preparation of standard solutions was accomplished by addition of 0 (blank), 1 µL, 25 µL, 50 µL, 100 µL, and 500 µL of stock solution to the matrix (20 g). Each mixture was also spiked with 20 µL of internal standard solution. Sample preparation and GC-MS analysis were performed in the same way as described above for the CP samples. Each standard concentration was measured in duplicate. Concentrations of positively identified compounds were determined using MS response factors for each compound relative to the internal standard, calculated as follows:

Concentration of compound i = concentration of internal standard x f_i x peak area of compound i/peak area of internal standard

Where f_i is the MS response factor of compound i calculated using 5-point standard curve. When no commercial standard is available, semi-quantitative estimates of methanethiol, 2-methylpropanal, 3,4-dimethylthiophene, methyl allyl disulfide, methyl allyl trisulfide, dipropyl trisulfide, 3-vinyl-4*H*-1,2-dithiin, 2-vinyl-1,3-dithiane, diallyl trisulfide, and 2-vinyl-4*H*-1,3-dithiin in Table 7 were conducted by using 2-methyl-3-heptanone as the internal standard without considering calibration factors, that is, f_i =1.00.

3. Sensory Evaluation of Chili Paste

The panelist was performed a quantitative descriptive analysis (QDA) to evaluate aroma profile of CPs. Panelists received approximately 20 h of training during which they identified and defined descriptive terms for CPs aroma and determined appropriate aroma references. The samples (5 g) were presented to 10 panelists in glass vials (48 mm i.d. x 58 mm high) with a cap. For the sensory evaluation, the cap was removed and the samples were sniffed by the trained panelists (at room temperature, ca 25 °C), who consisted of 4 male and 6 female graduate students of Kasetsart university between the ages of 22 and 39. Standard references for "dried shrimp", "roasted chili", "garlic", "salt aromatic", "shallot", and "sweet aromatic" were presented at room temperature (Table 4). Aroma intensities were scored on an unstructured 15 cm line scale anchored on the left with "none" and on the right with "very strong" (Meilgaard *et al*., 1999). The previously assigned intensity ratings of the standards were used as references for rating the intensities of the CPs. Rating results from individual panelists were revealed at the end of each sensory analysis session, and final aroma profiles of the CPs were reported on the basis of discussion and consensus rating (average of duplication) by the panel (Zhou *et al*., 2002).

Table 4 Preparation of reference standards for descriptive sensory evaluation of CPs

Descriptor	Reference	Preparation	Rating[a]
Dried shrimp (aromatic associated to dried shrimp products.)	Kung seab sauce	Weighed kung seab sauce (3 g) from local market into a glass vial	10.5
Roasted chili (aromatic associated to roasted chili.)	Ground roasted chili	Roasted chili with a little of soybean oil in a wok for 3 min and then ground and weighed to 0.6 g into a glass vial	10.4
Garlic (aromatic associated to garlic.)	Ground fried garlic	Ground garlic (100 g) and fried with soybean oil (50 g) in a wok for 5 min and then weighed (5 g) into a glass vial	9.7
Salt aromatic	Nam pla wan (grilled prawns with sweet and sour fish sauce)	Weighed nam pla wan (8 g) into a glass vial	9.9
Shallot (aromatic associated to shallot.)	Fried shallot	Weighed fried shallot (0.5 g) into a glass vial	9.9
Sweet aromatic	The solution of palm sugar	Heated and stirred palm sugar (150 g) with distilled water to 115 °C , added fish sauce (5 g) and distilled water (100 g), cooled to room temperature and then weighed to 2 Tbsp. into a glass vial	9.8

[a] Aroma intensity values were rated on an unstructured 15 cm line scale.

4. Encapsulation of Chili Paste Oil

4.1 Chili paste oil preparation

Chili paste was prepared from the formulation listed in Table 3. A wok was heated to about 100 °C. The ground shallot and ground garlic were added and fried for about 90 min until golden brown color was observed. The shrimp paste was added and thoroughly stirred. The fish sauce, tamarind juice, coconut sugar, and salt were then mixed and added in a wok. After that, the minced dried shrimp and chili powder were added and fried. The mixture was then further stir-fried with soybean oil until it was sticky. The total processing time from fried shallot to sticky chili paste in a wok was 25 min. After cooling to room temperature, chili paste was placed in Duran bottles and covered with aluminum foil to prevent from light and stored at -40 °C until using. Chili paste sample were prepared in duplicate.

The oil was separated from chili paste by centrifugation at 3000 rpm (rotor no.22; RC 50 plus, Sorvall®, Ct, USA) for 10 min at 20 °C. The upper oil layer was collected by decantation and pipetting into Duran bottles and stored at -40 °C until using for emulsion preparation.

4.2 Emulsion preparation

A commercial gum arabic was purchased from Jumbo Trading Co., Ltd. (Bangkok, Thailand). D-Perse 4, the 18.5 DE maltodextrin from tapioca starch was purchased from Siam Modified Starch (Pathumthani, Thailand). All the wall polymers were of food grade quality.

The formulation for the emulsion was followed that of Buffo *et al.* (2002). The compositions are listed in Table 5. The total concentration of the dissolved solids in the carrier solution was about 32% w/w on the wet basis (320 g of solid powder and 680 g of water). Three different combinations of solid systems were

prepared. The mass ratios of maltodextrin to gum arabic were 1:2, 1:1, and 2:1. The chili paste oil (CPO) mass ratio to the carrier was 1:4.

Table 5 Formulations for chili paste oil emulsion

Proportion of maltodextrin:gum arabic	Emulsion composition (g)			
	maltodextrin	gum arabic	water	chili paste oil
1:2	106.67	213.33	680	80
1:1	160.00	160.00	680	80
2:1	213.33	106.67	680	80

Emulsions were prepared according to the procedures described by Buffo *et al.* (2002). The odorless-distilled water (680 g) was heated to 82 °C on the hot plate stirrer (SS10, Stuart Scientific, UK). Gum arabic was added slowly to hot water at 82 °C. The mixture was held at this temperature and stirred vigorously with a perforated paddle (100 rpm) coupled with gentle magnetic stirring for 45 min until dissolved. The solution was then added with maltodextrin and stirred with both perforated paddled and magnetic stirrer. The solution was allowed to cool to room temperature, covered and stirred overnight to enhance hydration. After that, chili paste oil (CPO) was added and mixed thoroughly with the carrier solution. The core mass to the carrier ratio was 1:4. The mixture was emulsified immediately by using Ultra Turrax Ika 25 basic homogenizer (Janke&Kunkel GmbH & Co., Germany) operating at 13,000 rpm (rotor no.22; RC 50 plus, Sorvall®, Ct, USA) for 5 min and followed by 19,000 rpm for 5 min.

4.3 Spray drying

The emulsion was stirred slowly at room temperature during spray drying. The emulsion was fed at the rate of 20 mL/min to a Niro Mobile™ spray drier (Niro, Corp., Soeborg, Denmark) equipped with a rotary atomizer. The inlet and outlet temperatures were maintained at 165 and 85 ± 5 °C. The microcapsules were

collected, filled in aluminum pouches and stored at -40 °C in a freezer until further studied. The samples were prepared in duplicate.

5. Moisture Content Determination

The moisture content of the spray-dried, microencapsulated products was determined according to the 787 KF Titrino Karl Fischer Titrator (Metrohm, Herisau, Switzerland). The powder samples, 0.3 to 0.5 g, was weighed into the preweighted vials and mixed with the Hayashi solvent FM containing 26% methanol and < 0.2 mg H_2O/mL (Hayashi Pure Chemical Ind., Co., Ltd., Osaka, Japan) and titrated against a 5 mg/mL Hydranal composite solvent (Riedel-deHaën, Sigma-Aldrich, Seelze, Germany). The titrator was calibrated with water. The water content was reported in %w/w of sample. The samples were measured 3 times for moisture content.

6. Determination of Glass Transition Temperature

Differential scanning calorimetry (Pyris 1 DSC, Perkin Elmer, CT, USA) equipped with an intracooler (2P, Perkin Elmer) was used to measure glass transition temperatures (Tg) of the encapsulated powders following the method modified from Samuhasaneetoo (2004). The instrument was calibrated for the baseline and temperature using indium (mp 156.74 °C, ΔH_{fusion} = 28.45 J/g). Approximately 20 mg (± 1 mg) of the sample was weighed and hermetically sealed in stainless steel DSC pans. An empty pan was used as a reference. The sample was initially scanned from 25 to 160 °C at 10 °C/min, then cooled to -20 °C at 10 °C/min, and reheated to 160 °C at 10 °C/min. All samples were first heated to destroy thermal history of the sample. Glass transition temperatures were determined from onset temperatures of the discontinuities in the heat flow curves compared with the temperature of the second scan. The results were analyzed using a Pyris Software for Windows version 3.72 (Perkin Elmer). Samples were analyzed in duplicate.

7. Determination of Water Activity

The water activity of the encapsulated powders was determined in triplicate at 25 °C using a Water Activity Meter (Testo 650, Testo Inc., Germany).

8. Surface Oil Determination

Surface oil of the encapsulated CPO was determined by washing method (Samuhasaneetoo, 2004). The spray-dried, microencapsulated products (5 g) were extracted by gently stirring with a magnetic stirrer in 50 mL of mixed solvents (hexane:diethyl ether; (1:1)) at room temperature for 30 min in a 250 mL Duran bottle. The solvent was then filtered through Whatman No. 4 filter paper by vacuum filtration. Each filter paper with solid particles was washed with mixed solvents (3 x 10 mL). The solvent was collected in a preweighted round bottle flask and evaporated by using a rotary evaporator at 45 °C. After that, the flask was dried at 70 °C by using vacuum oven (VOS-450SD, Eyela, Tokyo, Japan) at 76 cmHg for 5 h and the oil was weighted.

$$\text{Surface oil (\%)} = \frac{\text{Weight of oil extracted}}{\text{Weight of dry powders}} \text{ X } 100$$

9. Total Oil Determination

Total oil contents of the spray-dried, microencapsulated products were determined according to the method of Samuhasaneetoo (2004). Two grams of spray-dried, microencapsulated CPO product dispersed in 2 mL of ethanol and 10 mL of hydrochloric acid (8 M) was placed in a 250 mL round-bottom flask. The flask was refluxed at 100 °C for 30 min in a heating water bath. The refluxed sample was placed in a 250 mL separating funnel. The flask was washed with 5 mL ethanol. The mixture was combined in the funnel. Fifty milliliters of mixed solvents (hexane:diethyl ether; (1:1)) was then added in the funnel. The CPO was extracted by

shaking the funnel for 5 min and standing for phase separation. The solvent phase was collected and the lower phase was re-extracted two times with 50 mL mixed solvents. The separated solvent was combined and placed in a preweighted round bottle flask and evaporated using a rotary evaporator at 45 °C. After that, the flask was dried at 70 °C by using a vacuum oven at 76 cmHg for 5 h and the oil was weighted.

$$\text{Total oil (\%)} = \frac{\text{Weight of oil extracted}}{\text{Weight of dry powders}} \text{ X } 100$$

$$\text{Encapsulation efficiency (\%)} = \frac{\text{Total oil} - \text{Surface oil}}{\text{Total oil}} \text{ X } 100$$

10. Release of Volatiles by Dynamic Headspace Analysis (DHA)

The release of selected volatiles (diallyl disulfide, benzaldehyde, butanoic acid, 3-methylbutanoic acid and phenol) from microencapsulated CPO powders was determined according to the method of Baranauskiene *et al*. (2007). Five grams of each spray-dried products which has been extracted with mixed solvents (hexane:diethyl ether; (1:1)) for surface oil determination was placed into the three-neck glass purge and trap vessel. The vessel was maintained at 30°C by circulating water through the external water jacket. The headspace volatiles were purged onto absorbent trap (Tenax TA, 200 mg /trap) using nitrogen (flow rate 30 mL/min, ultra high purity). Tenax tubes were changed every 5, 10, 15, 20, 25, 30, 40, 50 and 60 min, and the total purge time was 60 min. One microliter of internal standard solution (1.62 μg/μL of 2-ethylbutyric acid, 2.44 μg/μL of 2,4,6-trimethyl pyridine and 1.25 μg/μL of 2-methyl-3-heptanone in methanol) was injected on Tenax trap prior to desorption. Trapped volatiles were desorbed from Tenax trap by a thermal desorber (QUI-0002 version 5.1, Unity Markes Int. Ltd., Rhondda Cynon Taff, UK) for 3 min at 230°C; the cold trap was held at -10 °C, the trap heating temperature was 260 °C at

100 °C/sec and hold at this temperature for 3 min. After desorption, volatiles were directed through a heated transfer line at 200 °C and analyzed by 6890N gas chromatograph (Agilent Technologies Inc., China). The volatiles were then fractionated on a polar capillary column (Alltech 007-FFAP, 30 m x 0.32 mm i.d.; 0.25-µm film; Alltech Associated Inc., IL, USA). The GC oven temperature was programmed from 35 ºC to 225 ºC at the rate of 4 ºC/min with initial and final hold times of 5 and 20 min, respectively. Helium was used as a carrier gas at the constant flow rate of 3 mL/min. The FID was maintained at 250°C. Identification of flavor components in the chromatogram was accomplished by comparing the retention times to those of pure standards obtained from a separate GC analysis. Quantification of flavor components was done by comparing the sample peak area to that of the pure component (known amount) peak area.

11. Evaluation of Storage Stability

Each spray-dried microencapsulated products was washed with hexane:diethyl ether (1:1) to remove their surface oil and evaporated residue solvent from the powder overnight at room temperature in a hood. Five grams of powder was then spread on petri dish. The dishes were kept in a tight sealed plastic container over saturated sodium chloride solution (RH 68% at 50 °C) and placed in an incubator at 50 °C for 5 weeks. This relative humidity was selected base on the typical humidity in Thailand. The samples were withdrawn every week for flavor retention and sensory evaluation. Pulled samples were packed in the aluminum pouches and stored at -40°C until analysis.

12. Retention of CPO Encapsulated Powders Flavor during Storage

The changes in flavor were determined by quantifying the flavor retained by the complex. Encapsulated powder from samples with different mass ratios of maltodextrin and gum arabic were storage at 50 °C, 68% RH for 5 weeks and washed with mixed solvents. Approximately 2.5 g of the washed powder was dissolved in 50

mL of deodorized distilled water in a 250 mL Telfon bottle and 10 µl of internal standard solution (1.62 µg/µL of 2-ethylbutyric acid, 2.44 µg/µL of 2,4,6-trimethyl pyridine and 1.25 µg/µL of 2-methyl-3-heptanone in methanol) was added. Then, the mixture was added with 5 g of NaCl and extracted with diethyl ether in a shaking water bath at room temperature for 1 h. The solvent was next separated from the aqueous phase by centrifugation at 3000 rpm (rotor no.22; RC 50 plus, Sorvall®, Ct, USA) for 10 min at 20 °C. The upper layer was decanted and pipetted into a 250 mL Duran bottle. The extraction was repeated two more time with 50 mL of diethyl ether. Recovered solvent was pooled for each sample and then concentrated to 50 mL by distillation using a Vigreaux column at 40 °C. The concentrated solvent was then separated volatiles from non-volatiles by using high vacuum distillation operating at 50 °C for 2 h followed by room temperature for 1 h. After distillation, the extract was concentrated to 10 mL under a gentle stream of N_2 (g). The anhydrous Na_2SO_4 was then added and the extract was filtered through a conical test tube containing with 1.5 g of anhydrous Na_2SO_4. The extract was reduced to ~ 0.3 mL under N_2 (g) and transferred to 1 mL vial with 250 µL reduced volume insert. The extract was then further concentrated to 200 µL under N_2 (g). The vial was stored at -40 °C until the analysis for flavor retention by GC-MS. Flavor retention expressed as percentage of the remaining amount of flavor compound in the powder during storage based on the highest original volatiles found in the capsules at the initial time of storage.

12.1 Gas chromatography-mass spectrometry (GC-MS) analysis

The GC-MS system consisting of a 6890 GC (Agilent Technologies) equipped with a GC-5973 mass selective detector (MSD) system (Agilent Technologies Inc.) and an on-column injector. Each aroma extract (1 µL) was injected by cool on-column mode (+3 ˚C temperature tracking mode) into a polar capillary column (DB-FFAP, 30 m x 0.25 mm i.d.; 0.25-µm film; J&W Scientific) and a non-polar column (HP-5MS, 30 m x 0.25 mm i.d.; 0.5-µm film; J&W Scientific). The oven temperature was programmed from 35°C to 225 °C at a rate of 4 °C/min with an initial and final hold times of 5 and 20 min, respectively. Helium

was used as carrier gas at a constant rate of 1.0 mL/min. The MSD conditions were as follows: capillary direct interface temperature, 280 °C; ionization energy, 70 eV; mass range, 35 to 300 amu; electron multiplier voltage (autotune + 200 V); scan rate, 2.78 scans/s.

12.2 Identification of aroma compounds

Positive and tentative identification of aroma compounds retained in the encapsulated CPO powders and the calculation of linear retention indices (LRI) were performed as previously described in the identification of aroma active compounds in CPs.

12.3 Quantification of Aroma Compounds

The relative quantification of aroma compounds retained in the encapsulated CPO powders was done as previously described in the quantification of aroma active compounds in CPs.

13. Sensory Evaluation of CPO Powder Solution

The panelist was performed a quantitative descriptive analysis (QDA) to evaluate aroma profile of CPO powder solution. Panelists had a 20-h training during which they identified and defined aroma descriptive terms for CPO powder solution and determined the appropriate aroma references.

Fifteen grams of powders was dissolved in 300 mL of deodorized distilled water in a 500 mL glass beaker to obtain 5% total solid concentration. The mixtures were mixed using a magnetic stirrer at 50 °C until the powders were fully hydrated. The CPO powder solutions (20 mL) were placed in glass vials (48 mm i.d. x 58 mm high) with caps, kept to room temperature for 1 h to equilibrate between liquid and gaseous phases.

For the sensory evaluation, the caps were removed and the headspace of the samples were sniffed at room temperature, ca 25 °C, by the trained panelists consisting of 3 male and 9 female graduate students ages between 23-32 year olds from Kasetsart university. Standard references for "chili", "shrimpy", "garlic", "salt aromatic", "sweet aromatic" and "overall aroma intensity" were presented at room temperature (Table 6). Aroma intensities were scored on an unstructured 15 cm line scales anchored with "none" on the left and with "very strong" on the right. (Meilgaard *et al*., 1999). The previously assigned intensity ratings of the standards were used as references for rating the intensities of the CPO powder solution. Rating results from individual panelists were revealed at the end of each sensory analysis session, and final aroma profiles of the CPO powder solution were reported on the basis of discussion and consensus rating (average of duplication) by the panel (Zhou *et al*., 2002).

Table 6 Preparation of the reference standards for descriptive sensory evaluation of CPO powder solution.

Descriptor	Reference	Preparation	Rating[a]
Chili (aromatics associated to the solution of roasted chili smell)	The solution of roasted chili	Ground roasted chili (0.5 g) stirred in 250 mL of deodorized distilled (DOD) water with magnetic stirrer at room temperature for 5 min and placed (15 g) into a sealed glass vial	8.7
Shrimpy (aromatics related to the solution of dried shrimp)	The solution of minced dried shrimp	Washed dried shrimp with warm water and fried them until dried in a hot wok, ground with blender, minced dried shrimp (0.5 g) mixed with 20 mL of DOD water in a sealed glass vial	7.8

Table 6 (Continued)

Descriptor	Reference	Preparation	Rating[a]
Garlic (aromatics associated to garlic)	Ground fried garlic	Placed ground garlic fried with soybean oil from local market (0.5 g) in a sealed glass vial	9.9
Salt aromatic	The solution of deoiled chili paste (opened a cap 3 min before sniffing)	Deoiled chili paste (25 g) stirred in 250 mL of DOD water with magnetic stirrer at room temperature for 10 min and placed (15 g) into a sealed glass vial,	7.9
Sweet aromatic	The solution of mixed boiled sugars and shrimp	Stirred 1 cup of water, 12 teaspoons of sugar and 2 teaspoons of coconut sugar until boiled, added boiled shrimp and stirred until its volume was decreased by one-third of the original, cold to room temperature and placed (4 teaspoons) into a sealed glass vial	7.9
Overall aroma intensity	The solution of deoiled chili paste	Mixed deoilled chili paste (25 g) with 250 mL of DOD water only 2 min and filtered the solution, placed (15 g) into a sealed glass vial	7.5

[a] Aroma intensity values were rated on an unstructured 15 cm line scale.

14. Statistical Analysis

Complete randomize design (CRD) was applied as an experimental design for study the aroma-active compounds in Thai chili paste and their changes in a spray-dried powder

Statistical analysis software (SPSS version 12.00, SPSS Inc., Chicago, IL) was employed for analyzing the statistical results obtained from two replications. Mean with standard deviation for each treatment was calculated. The analysis of variance (ANOVA) and the Duncan's multiple range test (DMRT) were used for comparing differences of the mean values at the 0.05 confidence level.

15. Place and Duration

15.1 Places

- Department of Food Science and Technology, Faculty of Agro-Industry, Kasetsart University, Bangkok, Thailand.
- Department of Food Science and Human Nutrition, University of Illinois at Urbana-Champaign, Illinois, USA.

15.2 Duration

June 2004 –April 2008

RESULTS AND DISCUSSION

1. Effect of Thermal Processing on the Changes of Aroma-Active Compounds in Thai Chili Paste.

Thai chili paste (CP) samples with different degree of heating were prepared by excessively heating CP ingredients at 100 °C for 50 min (H50-CP), typically heating CP ingredients at 100 °C for 25 min (H25-CP), and unheated CP (UH-CP).

Proximate composition of the traditional CP, H25-CP, was determined by an AOAC methods (AOAC, 2000). The sample was composed of 59.61% carbohydrate, 24.29% lipid, 6.61% protein, 4.43% moisture, 3.78% ash, and 1.28% crude fiber.

Two complementary techniques, dynamic headspace dilution analysis (DHDA) and aroma extract dilution analysis (AEDA), were applied for the comprehensive gas chromatography-olfactometry (GCO) analysis of the aroma components. DHDA is suitable for the analysis of the odorants of high and intermediate volatility. AEDA is generally restricted to the analysis of the intermediate and semi-volatile odorants, since the highly volatile compounds may be lost during extraction, distillation, and sample workup procedures. In the present study, a total of 63 and 56 odorants were detected by DHDA (Table 7) and AEDA (Table 8), respectively. Thirty-six common odorants were detected by both methods. Twenty-seven odorants (nos. **1**-**6**, **9**, **11**, **13**, **18**, **19**, **25**, **29**, **32**, **34**, **35**, **37**, **39**, **43**, **48**, **51, 54**, **58**, **62**, **64**, **65**, and **68**) were detected only by DHDA. AEDA revealed additional 19 odorants (nos. **12**, **28**, **33**, **38**, **40**, **42**, **44**, **52**, **57**, **59**, **71**-**73**, **77**-**79**, **81**-**83**) that were not detected by DHDA.

1.1 Aroma-Active Compounds Evaluated by Gas Chromatography Olfactometry-Dynamic Headspace Dilution Analysis (GCO-DHDA)

In UH-CP, 26 odorants were detected with FD factors of 25, including 13 sulfur-containing compounds. Several sulfur compounds detected were the decomposition products of thiosulfinates, derived from amino acid flavor precursors such as S-allyl-L-cysteine sulfoxide (alliin) (Wu *et al*., 1982; Block *et al*., 1986). Alliin is the most predominant amino acid flavor precursor of garlic (Block *et al*., 1986). The enzyme allinase hydrolyzed alliin to pyruvate, ammonia, and thiosulfinate particularly allicin, after tissue disruptions (Block *et al*., 1986). Allicin is highly unstable and can decompose or rearrange to form secondary products. A variety of sulfides are known to contribute to the characteristic flavor and taste of garlic (Block *et al*., 1986; Yu and Wu, 1989a).

The major garlic-derived odorants in UH-CP were identified as dimethyl sulfide (no. **3**), allyl methyl disulfide (no. **24**), dimethyl trisulfide (no. **31**), diallyl disulfide (no. **41**), allyl methyl trisulfide (no. **50**), 3-vinyl-4*H*-1,2-dithiin (no. **61**), and 2-vinyl-4*H*-1,3-dithiin (no. **70**). These compounds have sulfurous, garlic-like, and meaty aroma notes. It has been reported that allyl mercaptan (no. **6**), which can be generated from alliin through a hydrolysis process or free-radical rearrangement (Yu *et al*., 1994c), imparted a characteristic meaty, garlic-like and bologna-like aroma. Allyl mercaptan was reported as a character-impact odorant in bulgogi (Ko *et al*., 2005). Another potent sulfur-containing compound, methyl propyl disulfide (no. **21**), was reported as a character-impact component of onion (Nagodawithana, 1995). In addition, the sulfur compounds with propyl group has been reported as the major sulfur-containing compounds in shallot volatiles (Block, 1992; Nagodawithana, 1995). (*E*)-propenyl propyl disulfide (no. **37**) was the major volatile identified in the distilled shallot oil. 2,4-Dimethylthiophene (no. **18**) was considered to be the typical constituent of fried shallot (Kuo *et al*., 1990; Ho *et al*., 1997).

The sulfur-containing compounds with the highest FD factors of 25 in all CP samples were dimethyl sulfide (no. **3**, fresh corn), allyl mercaptan (no. **6**,

meaty/bologna-like), 3,3'-thiobis-1-propene (no. **17**, sulfurous), methyl propyl disulfide (no. **21**, sulfurous/meaty), allyl methyl disulfide (no. **24**, sulfurous), dimethyl trisulfide (no. **31**, sulfurous/garlic), methional (no**. 36**, potato), (*E)*-propenyl propyl disulfide (no. **37**, sulfurous/garlic), diallyl disulfide (no. **41**, pungent/fresh garlic), allyl methyl trisulfide (no. **50**, eggy/meaty/garlic), and 3-vinyl-4*H*-1,2-dithiin (no. **61**, pungent/garlic). Among these, dimethyl trisulfide was the most potent aroma-active compound with the highest odor-activity value (OAV), followed by allyl mercaptan, diallyl disulfide, and dimethyl sulfide (Table 9). These compounds have extremely high OAVs due to their relatively high concentrations and very low detection threshold values. Moreover, allyl methyl disulfide, allyl methyl trisulfide, and 3-vinyl-4*H*-1,2-dithiin had the highest FD factors of 25 in all treatments, which was consistent with their high relative abundance values (Table 9). Their OAVs, however, could not be calculated because the odor detection threshold values for these compounds are unavailable.

Other predominant aroma compounds with high FD factors of 25 in all CP samples were 2/3-methylbutanal (no. **7/8;** dark chocolate; detected as a coeluted peak), 2,3-butanedione (no. **10**, buttery), 1-octen-3-one (no. **26**, mushroom), (*Z*)-1,5-octadien-3-one (no. **32**, metallic), and *β*-damascenone (no. **69**, applesauce). 2- and 3-Methylbutanal (dark chocolate-like) are Strecker aldehydes derived from isoleucine and leucine, respectively (Belitz and Grosch, 1997). 2,3-Butanedione is a characteristic component of cooked foods and can be thermally generated through the Maillard reaction (Hodge, 1967). Although the FD factors of 2/3-methylbutanal and 2,3-butanedione in the three CP samples were not different, the concentrations of these compounds increased significantly with increasing heating times ($P < 0.05$) and showed the highest concentration in H50-CP (Table 9). Therefore, these odorants seemed to be partially responsible for the thermally-induced changes in CP flavor. 1-Octen-3-one and 1,5-octadien-3-one were reported to contribute to the aroma of fresh crustaceans and these compounds and can be formed by lipid degradation of arachidonic acid and linolenate, respectively (Baek and Cadwallader, 1997). *β*-Damascenone has a very low odor threshold in water (2 pg/g) (Leffingwell, 2004). This compound is believed to be a product of the oxidative degradation of the

carotenoid neoxanthin (Isoe *et al.,* 1973). It can be formed by the hydrolysis of glycosides in some natural products such as grapes (Shure and Acree, 1994). Pojjanapimol (2004) has been reported that *β*-damascenone, heat induced compound, occurred in holy basil essential oil, which produced by thermal process and holy basil leaves that heating in water and oil. On the contrary, this compound was not detected in the fresh holy basil leaves.

The heating of CP lead to the generation of thermally-derived aroma compounds such as methanethiol (no. **1**, sulfurous/rotten), 2-methylpropanal (no. **4**, dark chocolate), 1-propanethiol (no. **5**, meaty), thiophene (no. **11**, garlic), 2-methyl-3-furanthiol (no. **19**, meaty), 3,4-dimethylthiophene (no. **22**, garlic/fishy), octanal (no. **25**, orange oil), benzaldehyde (no. **47**, chili/nutty), butanoic acid (no. **53**, cheesy), and (*E,Z*)-2,6-nonadien-1-ol (no. **64**, cucumber). The FD factors of these compounds increased as a result of heating and they had the highest FD factors in H50-CP. Among these odorants, methanethiol (b.p.= 6 °C) was detected only by DHDA. A semiquantitative estimate of this compound concentration was made due to difficulties in accurately preparing the reference standard. Methanethiol and 2-methylpropanal had low odor thresholds and relatively high concentrations in three CPs (Table 9); thus they may contribute to the overall aroma of the CPs. It was reported that Strecker degradation of valine can lead to the formation of 2-methylpropanal (Belitz and Grosch, 1997). 2-Methylpropanal has been identified as a character-impact odorant in fish sauce (Fukami *et al.,* 2002). 2-Methyl-3-furanthiol (b.p. = 57 °C) was detected only by DHDA on the nonpolar RTX5 column and was not detected by AEDA of the solvent extracts. 2-Methyl-3-furanthiol has been identified as an important odorant in a beef-like process flavor (Baek *et al.,* 2001). This compound can be perceived at a very low concentration because of its low odor detection threshold (*t* = 0.005-0.01 ppb in water; MacLeod and Ames, 1986). The compounds (*E*)-2-decenal (no. **54**, cilantro/fatty), 3-methylbutanoic acid (no. **56**, sweaty/dried fruit), and diallyl trisulfide (no. **66**, pungent/garlic) were detected at higher FD factors in H25-CP than in UH-CP and H50-CP. It was reported that 3-methylbutanoic acid is produced from leucine catabolism *via* transamination, followed by oxidation steps (Thierry *et al.,* 2004).

1.2 Aroma-Active Compounds Detected by Gas Chromatography-Olfactometry Aroma Extract Dilution Analysis (GCO-AEDA)

The medium and higher boiling odorants were extracted from the CP matrix by direct solvent extraction with diethyl ether. The volatiles were then isolated from the non-volatile material by high-vacuum distillation, followed by fractionation to acidic, basic, and neutral fractions.

The results of AEDA for the neutral, basic, and acidic fractions for all CPs are summarized in Table 8 and concentration results for selected odorants presented in Table 9. The concentration of aroma-active compounds on DHDA is higher than on AEDA because AEDA requires solvent extraction, distillation, and evaporation steps. Volatile compounds, as well as their relative abundances, could be changed, and some highly volatile compounds might be lost all together. AEDA revealed 12, 14, and 5 potent odorants with $\log_3$FD factors between 5 and 12 were detected in UH-CP, H25-CP, and H50-CP, respectively. Among these, allyl methyl disulfide (no. **24**, pungent/fresh garlic), dimethyl trisulfide (no. **31**, sulfurous/garlic), methional (no. **36**, potato), diallyl disulfide (no. **41**, pungent/fresh garlic), allyl methyl trisulfide (no. **50**, eggy/meaty/garlic), dipropyl trisulfide (no. **59**, meaty/garlic), 3-vinyl-4*H*-1,2-dithiin (no. **61**, pungent/garlic), diallyl trisulfide (no. **66**, pungent/garlic) and 2-vinyl-4*H*-1,3-dithiin (no. **70**, pungent/garlic) were the predominant odorants having high $\log_3$FD factors in all CPs. Based on results of AEDA, 2-vinyl-4*H*-1,3-dithiin (no. **70**) was the most potent odorant with the highest $\log_3$FD factors of 9, 12, and 9 in UH-CP, H25-CP, and H50-CP, respectively.

Table 7 Potent headspace odorants of Thai chili pastes determined by dynamic headspace dilution analysis.

No.[a]	Compound	RI[b]		Odor description[c]	FD factor[d]		
		FFAP	RTX5		UH-CP	H25-CP	H50-CP
1	methanethiol[e]	696	<500	sulfurous, rotten	5	5	25
2	acetaldehyde[e]	734	<500	pungent, yogurt	25	1	1
3	dimethyl sulfide[e]	760	<500	fresh corn	25	25	25
4	2-methylpropanal[e]	779	549	dark chocolate	5	5	25
5	1-propanethiol[e]	838	616	meaty	5	5	25
6	allyl mercaptan[e]	904	608	meaty, bologna	25	25	25
7/8	2/3-methylbutanal[e]	918	645	dark chocolate	25	25	25
9	allyl methyl sulfide[e]	946	681	meaty, fishy	5	25	25
10	2,3-butanedione[e]	990	593	buttery	25	25	25
11	thiophene[e]	996	na[g]	garlic	nd[h]	5	25
13	ethyl 3-methylbutanoate[e]	1061	851	fruit, berry	5	1	1
14	dimethyl disulfide[e]	1107	775	sulfurous, rubbery	1	1	1
15	hexanal[e]	1086	796	green, cut-grass	5	5	5
16	*(Z)*-3-hexenal[e]	1148	794	green, cut-leaf	5	5	1
17	3,3'-thiobis-1-propene[e]	1188	868	sulfurous	25	25	25
18	2,4-dimethylthiophene[f]	1195	873	sulfurous, rubbery	25	5	5
19	2-methyl-3-furanthiol[f]	na	875	meaty	nd	5	25
20	*(Z)*-4-heptenal[e]	1237	909	crabby, fishy, fatty	25	5	5
21	methyl propyl disulfide[e]	1240	920	sulfurous, meaty	25	25	25

Table 7 (Continued)

No.[a]	Compound	RI[b]		Odor description[c]	FD factor[d]		
		FFAP	RTX5		UH-CP	H25-CP	H50-CP
22	3,4-dimethylthiophene[f]	1258	880	garlic, fishy	1	5	25
23	1,3-dithiane[e]	1277	931	sulfurous, rubbery, salty	5	5	25
24	allyl methyl disulfide[f]	1288	958	pungent, fresh garlic	25	25	25
25	octanal[e]	1290	1011	orange oil	1	5	25
26	1-octen-3-one[e]	1296	976	mushroom	25	25	25
27	2-acetyl-1-pyrroline[f]	1335	921	popcorn	5	25	25
29	unknown	1345	na	roasted	5	5	5
30	dipropyl disulfide[e]	1372	1138	sulfurous, garlic	5	5	5
31	dimethyl trisulfide[e]	1388	969	sulfurous, garlic	25	25	25
32	(*Z*)-1,5-octadien-3-one[e]	1402	986	metallic	25	25	25
34	2-isopropyl-3-methoxypyrazine[e]	1429	1187	green, earthy, soil	5	5	1
35	propyl hexanoate[e]	1442	na	floral, melon	nd	5	5
36	methional[e]	1456	902	potato	25	25	25
37	(*E*)-propenyl propyl disulfide[f]	1450	na	sulfurous, garlic	25	25	25
39	unknown	1461	na	garlic, green, oily, nutty	25	5	1
41	diallyl disulfide[e]	1478	1080	pungent, fresh garlic	25	25	25
43	(*Z*)-2-nonenal[e]	1502	1159	hay	25	5	1
45	(*E*)-2-nonenal[e]	1513	1171	stale, hay	25	25	1
46	2-isobutyl-3-methoxypyrazine[e]	1521	1174	bell pepper	25	25	1

Table 7 (Continued)

No.[a]	Compound	RI[b]		Odor description[c]	FD factor[d]		
		FFAP	RTX5		UH-CP	H25-CP	H50-CP
47	benzaldehyde[e]	1533	941	chili, nutty	nd	25	25
48	(*Z*)-3-nonenal[e]	1540	1095	melon, cucumber	5	5	5
49	(*E,Z*)-2,6-nonadienal[e]	1586	1150	cucumber	25	5	5
50	allyl methyl trisulfide[f]	1592	1144	eggy, meaty, garlic	25	25	25
51	unknown	1606	na	mushroom	nd	1	1
53	butanoic acid[e]	1618	na	cheesy	nd	5	25
54	(*E*)-2-decenal[e]	1636	1260	cilantro, fatty	nd	5	1
55	phenylacetaldehyde[e]	1649	1046	dried rose, plastic	1	5	5
56	3-methylbutanoic acid[e]	1655	na	sweaty, dried fruit	1	5	1
58	unknown	1671	na	garlic, meaty	25	25	25
60	(*E,E*)-2,4-nonadienal[e]	1706	1215	fried, fatty	5	5	5
61	3-vinyl-4*H*-1,2-dithiin[f]	1721	1193	pungent, garlic	25	25	25
62	unknown	1730	1330	metallic	nd	5	5
63	2-vinyl-1,3-dithiine[f]	1742	1202	pungent, garlic	5	5	25
64	(*E,Z*)-2,6-nonadien-1-ol[e]	1755	1162	green, cucumber	1	5	25
65	2-acetyl-2-thiazoline[e]	1761	1097	popcorn	nd	nd	1
66	diallyl trisulfide[f]	1805	1305	pungent, garlic	5	25	5
67	(*E,E*)-2,4-decadienal[e]	1820	1319	fried, fatty	1	1	5
68	unknown	1825	na	brothy	5	5	1

Table 7 (Continued)

No.[a]	Compound	RI[b]		Odor description[c]	FD factor[d]		
		FFAP	RTX5		UH-CP	H25-CP	H50-CP
69	*β*-damascenone[e]	1859	1390	apple sauce	25	25	25
70	2-vinyl-4*H*-1,3-dithiin[f]	1847	1222	pungent, garlic	25	5	5
74	phenol[e]	1974	na	medicinal, antiseptic	1	5	5
75	*trans*-4,5-epoxy-(*E*)-2-decenal[f]	2006	1378	metallic	5	1	1
76	4-ethylguaiacol[f]	2027	1288	smoky, seasoning	1	1	1
80	*o*-aminoacetophenone[e]	2179	1351	corn, tortilla	1	1	1

[a] Numbers correspond to those in Table 8, Table 9, Appendix Table A1 and Appendix Table A2. [b] Retention indices (RI) calculated from GCO results on FFAP (Stabilwax®-DA) or RTX5 (RTX®-5SILMS) column. [c] Odor description at the sniffing port during GCO. [d] Flavor dilution factor determined on Stabilwax-DA column, except compound no. 19; FD factor = highest purge time tested (25 min) divided by lowest purge time in which odorant was last detected by GCO (25, 5, or 1 min); UH-CP = unheated CP, H25-CP = CP heated at 100°C for 25 min, and H50-CP = CP heated at 100 °C for 50 min. [e] Compound positively identified (RI, odor, MS). [f] Compound tentatively identified (RI, odor). [g] na = not available. [h] nd = no odor detected.

The FD factors and the concentrations of the thermally generated aroma compounds (nos. **8**, **10**, **27**, **36**, and **77**) increased as a result of heating. It was found that excessive heating of CP (e.g. H50-CP) caused the formation of more intense potato and caramelized notes as a result of increases in methional (no. **36**, potato) and 4-hydroxy-2,5-dimethyl-3-(2*H*)-furanone (no. **77**, HDMF; caramel/burnt sugar). HDMF can be generated from hexoses (for example, glucose or fructose) during the Maillard reaction (Schieberle, 1992; MacLeod, 1998). Methional has been associated with the Strecker degradation of methionine (Tressl *et al.,* 1989). The OAVs and FD factors were consistent for methional and HDMF. Based on its high OAV, HDMF was regarded as the predominant thermally-induced aroma compound of CP.

The FD factors of sulfur-containing compounds (nos. **21**, **24**, **30**, **31**, **41**, **50**, **59**, **61**, **63**, **66**, and **70**) in neutral fraction were highest in the CP produced by intermediately heating treatment (H25-CP), then these compounds decreased as a result of excessive heating (i.e., H50-CP). 3-Vinyl-4*H*-1,2-dithiin (no. **61**) and 2-vinyl-4*H*-1,3-dithiin (no. **70**) were postulated to be thermal degradation products of allicin (Yu and Ho, 1989a). Because of their high FD factors ($\log_3$FD factors ≥ 7) and aroma properties, 2-vinyl-4*H*-1,3-dithiin (no. **70**), diallyl trisulfide (no. **66**), allyl methyl trisulfide (no. **50**), 3-vinyl-4*H*-1,2-dithiin (no. **61**) and diallyl disulfide (no. **41**) are considered predominant odorants of H25-CP. These compounds are responsible for garlic, sulfurous, pungent, and meaty aroma characters. They have been reported to be the major volatile compounds in thermally-treated garlic products (Yu and Ho, 1993; Yu *et al.,* 1993, 1994c) and may be formed by decomposition and rearrangement of allicin at high temperature (Yu and Ho, 1993). Quantitative data in Table 9 showed that compounds nos. **41**, **50**, and **66** exhibited the highest concentration in H25-CP. Moreover, it has been reported that methyl propyl disulfide (no. **21**), dipropyl disulfide (no. **30**) and dipropyl trisulfide (no. **59**) were the character-impact flavor compounds in onion and shallot products (Wu *et al*., 1982; Ohsumi *et al.,* 1993). Although dimethyl disulfide had a $\log_3$FD factor of only 3 in H25-CP, this compound may also contribute to flavor of CP due to its high OAV.

The FD factors of benzaldehyde (no. **47**), (*E,E*)-2,4-decadienal (no. **67**) and *β*-damascenone (no. **69**) increased as a result of heating leading to higher chili/nutty, fatty/fried and apple sauce notes, respectively, in H25-CP. The FD factors of these compounds decreased due to decomposition caused by excessive heating (i.e., H50-CP). (*E,E*)-2,4-Decadienal has been reported as an oxidation product of linoleic acid and plays an important role in the flavor of deep-fried food (Ho *et al.,* 1987). (*E,E*)-2,4-Decadienal and *β*-damascenone, which have extremely low odor detection threshold values of 0.07 and 0.002 in water, respectively (Leffingwell, 2004), were not detected by GC-MS and were, therefore, only tentatively identified using the GCO data.

3-Ethyl-2,5-dimethylpyrazine (no. **33**) and 2,3-diethyl-5-methylpyrazine (no. **42**) were found to be the predominant compounds in the basic fraction of H25-CP. These compounds have characteristic potato and roasted odors. It has been reported that these pyrazines are typical Maillard reaction products formed by interaction of carbonyl (reducing) compounds and amino-containing compounds (Tai and Ho, 1999). In particular, 3-ethyl-2,5-dimethylpyrazine was present in the three treatments at concentrations higher than its odor detection threshold value. Because of its high FD factor in the basic fraction, *o*-aminoacetophenone (no. **80**, corn/tortilla-like) was regarded as an important character impact component of H25-CP. This compound was previously identified as a predominant odorant in tortilla-type corn products and it may be generated from alkali degradation of tryptophan (Buttery and Ling, 1995).

With the exception of HDMF, the FD factors of the acidic odorants differed only slightly among the three CPs. Sotolon (no. **81**, curry), phenylacetic acid (no. **82**, rosy) and vanillin (no. **83**, vanilla) had relatively high $\log_3$FD factors of 3 to 4 in all three CPs. Butanoic (no. **53**) and 3-methylbutanoic acids (no. **56**) were detected at higher $\log_3$FD factors in H25-CP, and this finding is consistent with the quantitative data (Table 9). 3-Methylbutanoic acid has been previously reported as a major volatile component of chili (Zimmermann and Schieberle, 2000).

Table 8 Potent odorants (Log_3FD factors ≥ 2) in Thai chili pastes determined by aroma extract dilution analysis

No.[a]	Compound	Fraction[i]	RI[b]		Odor description[c]	Log_3 FD factors[d]		
			WAX	RTX5		UH-CP	H25-CP	H50-CP
7	2-methylbutanal[e]	NF	915	650	dark chocolate	<1	2	1
8	3-methylbutanal[e]	NF	920	646	dark chocolate	1	3	3
10	2,3-butanedione[e]	AF	982	na[g]	buttery	2	3	3
12	ethyl 2-methylbutanoate[e]	NF	1046	na	floral, sweet	2	<1	<1
14	dimethyl disulfide[e]	NF	1077	769	rubbery, garlic	2	3	3
15	hexanal[e]	NF	1086	796	green, bug	1	2	1
16	(*Z*)-3-hexenal[e]	NF	1149	801	green, bug	2	<1	<1
17	3,3'-thiobis-1-propene[e]	NF	1190	854	garlic, rubbery	1	2	1
20	(*Z*)-4-heptenal[e]	NF	1233	904	fishy, marine	4	3	2
21	methyl propyl disulfide[e]	NF	1251	914	sulfurous, meaty	4	4	2
22	3,4-dimethylthiophene[f]	NF	1266	886	garlic salt, rubbery	5	4	4
23	1,3-dithiane[e]	NF	1290	931	garlic	1	<1	2
24	allyl methyl disulfide[f]	NF	1294	954	pungent, fresh garlic	5	6	4
26	1-octen-3-one[e]	NF	1307	979	mushroom	5	4	2
27	2-acetyl-1-pyrroline[f]	BF	1320	918	popcorn	<1	1	2
28	unknown	NF	1326	na	garlic salt, fishy, meaty	5	nd[h]	nd
30	dipropyl disulfide[e]	NF	1387	1135	sulfurous, garlic	nd	5	1
31	dimethyl trisulfide[e]	NF	1380	981	sulfurous, garlic	5	6	5
33	3-ethyl-2,5-dimethylpyrazine[e]	BF	1428	1064	green, potato, roasted	nd	3	<1

Table 8 (Continued)

No.[a]	Compound	Fraction[i]	RI[b]		Odor description[c]	Log_3 FD factors[d]		
			WAX	RTX5		UH-CP	H25-CP	H50-CP
36	methional[e]	NF	1448	897	potato	4	5	6
38	unknown	NF	1455	na	green, nutty	<1	3	<1
40	unknown	NF	1462	na	roasted, meaty, dried shrimp	4	6	2
41	diallyl disulfide[e]	NF	1473	1080	pungent, fresh garlic	5	7	4
42	2,3-diethyl-5-methylpyrazine[e]	BF	1473	1151	roasted, potato	2	3	1
44	diethylmethylpyrazine[f]	BF	1504	na	roasted	2	nd	nd
45	(*E*)-2-nonenal[e]	NF	1549	1164	floral, melon	4	nd	nd
46	2-isobutyl-3-methoxypyrazine[e]	NF	1536	1177	chili	3	4	1
47	benzaldehyde[e]	NF	1537	953	chili, roasted	1	5	2
49	(*E,Z*)-2,6-nonadienal[e]	NF	1596	1154	green, cucumber	4	4	1
50	allyl methyl trisulfide[f]	NF	1606	1169	eggy, meaty, garlic	6	9	6
52	unknown	NF	1614	na	potato, mushroom	2	3	1
53	butanoic acid[e]	AF	1628	na	cheesy	3	4	3
55	phenylacetaldehyde[e]	NF	1655	1043	rosy	3	3	2
56	3-methylbutanoic acid[e]	AF	1673	na	sweaty, dried fruit	4	5	4
57	unknown	NF	1666	na	melon, floral	2	4	<1
59	dipropyl trisulfide[f]	NF	1683	1356	meaty, garlic	6	6	4
60	(*E,E*)-2,4-nonadienal[e]	NF	1711	1335	nutty	nd	1	<1
61	3-vinyl-4*H*-1,2-dithiin[f]	NF	1756	1194	pungent, garlic	8	8	2
63	2-vinyl-1,3-dithiane[f]	NF	1769	1202	garlic	1	5	2
66	diallyl trisulfide[f]	NF	1811	1301	pungent, garlic	6	11	4

Table 8 (Continued)

No.[a]	Compound	Fraction[i]	RI[b]		Odor description[c]	Log_3 FD factors[d]		
			WAX	RTX5		UH-CP	H25-CP	H50-CP
67	(*E,E*)-2,4-decadienal[e]	NF	1817	1335	nutty, oily	1	3	<1
69	*β*-damascenone[e]	NF	1843	1374	apple sauce	2	3	1
70	2-vinyl-4*H*-1,3-dithiin[f]	NF	1858	1236	pungent, garlic	9	12	9
71	hexanoic acid[e]	AF	1862	na	sweaty	2	2	2
72	unknown	AF	1879	na	brothy, spicy	nd	nd	2
73	unknown	AF	1914	na	spicy	nd	2	nd
74	phenol[e]	NF	2001	na	inky, metallic	5	<1	<1
75	*trans*-4,5-epoxy-(*E*)-2-decenal[f]	NF	2019	1381	metallic	3	2	1
76	4-ethylguaiacol[f]	NF	2043	1288	smoky, seasoning	4	3	2
77	4-hydroxy-2,5-dimethyl-3-(2*H*)-furanone (HDMF)[e]	AF	2028	na	caramel, burnt sugar	2	4	5
78	*p*-cresol (4-methylphenol)[e]	NF	2074	na	phenolic, dung, stable	1	2	1
79	unknown	BF	2105	1342	minty	1	2	1
80	*o*-aminoacetophenone[e]	BF	2236	1299	corn tortilla	1	3	2
81	3-hydroxy-4,5-dimethyl-2(*5H*)-furanone (sotolon)[e]	AF	2211	na	curry, spicy	3	4	3
82	phenylacetic acid[e]	AF	2554	na	rosy	3	3	3
83	4-hydroxy-3-methoxybenzoic acid (vanillin)[e]	AF	2561	na	vanilla	3	4	3

[a] Numbers correspond to those in Table 7, Table 9, Appendix Table A1 and Appendix Table A2. [b - h] Foot notes same as Table 7.

[i] Fraction containing odorant; AF = acidic fraction, BF = basic fraction and NF = neutral fraction.

Table 9 Concentrations and odor activity values of the selected potent odorants[a] in Thai chili pastes

No.[b]	Compound	Concentration ± SD (ng/g)[c]			Odor threshold	Odor activity value (OAV)[e]		
		UH-CP	H25-CP	H50-CP	(ng/mL)[d]	UH-CP	H25-CP	H50-CP
1	methanethiol[g,h]	212 ± 13a	792 ± 100b	908 ± 35b	0.02[j] (0.06[k])	10600 (3533)	39600 (13200)	45400 (15133)
3	dimethyl sulfide[f,h]	63 ± 7a	408 ± 81b	507 ± 60b	0.3[j] (1.2 [k])	210 (53)	1360 (340)	1690 (423)
4	2-methylpropanal[g,h]	397 ± 40a	1019 ± 68b	1327 ± 165b	0.1[j] (3.4[k])	3970 (117)	10190 (300)	13270 (390)
6	allyl mercaptan[f,h]	141 ± 5a	242 ± 11a	477 ± 72b	0.005[l]	28200	48400	95400
8	3-methylbutanal[f,h]	139 ± 14a	2052 ± 196b	3144 ± 154c	0.2[j] (13[k])	695 (11)	10260 (158)	15720 (242)
9	allyl methyl sulfide[f,h]	77 ± 12a	401 ± 50b	706 ± 132c	na[m]			
10	2,3-butanedione[f,h]	94 ± 6a	114 ± 7ab	172 ± 32b	2.3[j] (10[k])	41 (9)	50 (11)	75 (17)
14	dimethyl disulfide[f,i]	8 ± 0.05a	27 ± 0.1c	26 ± 0.2b	0.16[l]	50	169	163
17	3,3'-thiobis-1-propene[f,h]	105 ± 13a	390 ± 25b	555 ± 53c	32.5[j]	3	12	17
21	methyl propyl disulfide[f,h]	50 ± 3a	100 ± 1b	148 ± 19c	na			
22	3,4-dimethylthiophene[g,h]	78 ± 2a	155 ± 2b	298 ± 37c	na			
23	1,3-dithiane[f,h]	894 ± 431a	1736 ± 83b	2006 ± 98b	na			
24	allyl methyl disulfide[g,h]	2759 ± 78a	6552 ± 189b	8182 ± 201c	na			
30	dipropyl disulfide[f,i]	1.75 ± 0.03a	2.17 ± 1.41a	1.77 ± 0.05a	na			
31	dimethyl trisulfide[f,h]	1555 ± 35a	4644 ± 49c	4450 ± 6b	0.005[j] (2.5[k])	311000 (622)	928800 (1858)	890000 (1780)
33	3-ethyl-2,5-dimethylpyrazine[f,i]	0.4 ± 0.02a	0.9 ± 0.2b	0.5 ± 0.01a	0.4[l] (24 [l])	1 (0.02)	2 (0.04)	1 (0.02)
36	methional[f,i]	0.2 ± 0.00a	1.06 ± 0.1b	1.22 ± 0.2b	0.2[j] (0.2[j])	1 (1)	5 (5)	6 (6)

Table 9 (Continued)

No.[b]	Compound	Concentration ± SD (ng/g)[c]			Odor threshold	Odor activity value (OAV)[e]		
		UH-CP	H25-CP	H50-CP	(ng/mL)[d]	UH-CP	H25-CP	H50-CP
41	diallyl disulfide[f,h]	4205 ± 64a	8202 ± 192b	8000 ± 408b	4.3[l]	978	1907	1860
47	benzaldehyde[f,h]	27 ± 6a	47 ± 12a	45 ± 6a	350[j]	0.08	0.13	0.13
50	allyl methyl trisulfide[g,h]	1706 ± 18a	1835 ± 129a	1604 ± 126a	na			
53	butanoic acid[f,i]	42 ± 1a	157 ± 15b	64 ± 0.1a	240[j]	0.18	0.65	0.27
					(135[k])	(0.31)	(1.16)	(0.47)
55	phenylacetaldehyde[f,i]	11 ± 0.01b	11 ± 1b	2 ± 0.02a	4[j]	3	3	0.5
56	3-methylbutanoic acid[f,i]	98 ± 1a	196 ± 7c	117 ± 0.2b	120[j]	0.8	2	1
					(22[k])	(4)	(9)	(5)
59	dipropyl trisulfide[g,i]	0.3 ± 0.00ab	0.35 ± 0.00b	0.27 ± 0.04a	na			
61	3-vinyl-4*H*-1,2-dithiin[g,h]	1108 ± 30a	1319 ± 12b	1461 ± 51c	na			
63	2-vinyl-1,3-dithiane[g,i]	3 ± 0.01a	11 ± 0.1b	3 ± 0.03a	na			
66	diallyl trisulfide[g,h]	343 ± 2a	959 ± 9c	887 ± 23b	na			
70	2-vinyl-4*H*-1,3-dithiin[g,h]	259 ± 3b	226 ± 10a	223 ± 4a	na			
74	phenol[f,i]	22 ± 0.04b	1 ± 0.1a	2 ± 0.02a	5500[i]	0.05	0.04	0.04
77	4-hydroxy-2,5-dimethyl-3-(2*H*)-furanone (HDMF)[f,i]	0.4 ± 0.01a	2.1 ± 0.5b	2.8 ± 0.15b	0.03[k]	13	70	93
					(25[j])	(0.02)	(0.08)	(0.11)
82	phenylacetic acid[f,i]	11 ± 0.2a	14 ± 2a	11 ± 2a	1000[i]	0.01	0.01	0.01

[a] Compounds having high FD factors and detectable by GC-MS were chosen for quantitative analysis. [b] Numbers correspond to those in Tables 7, Table 8, Appendix Table A1 and Appendix Table A2. [c] Average concentration ± standard deviation (n=2). Means in a row followed by different letters are significantly different ($P < 0.05$). [d] Odor detection threshold in water. Numbers in parentheses are odor detection thresholds in sunflower oil. [e] Odor activity value (OAV) was calculated by dividing compound concentration by its published odor detection threshold in water or in sunflower oil. [f] Concentration of compound is based on calibration against authentic reference standard (see text). [g] Semiquantitive concentration = concentration of internal standard x peak area of compound/peak area of the internal standard (see text). [h] Concentration determined by dynamic headspace analysis/GC-MS. [i] Concentration determined by direct solvent extraction-solvent assisted flavor evaporation/GC-MS. [j] Odor threshold reported by Leffingwell (2004). [k] Odor threshold reported by van Gemert (2003). [l] Odor threshold reported by Burdock (2004). [m] na = not available.

1.3 Sensory Evaluation

The results of quantitative descriptive analysis of CP samples are presented in Figure 10 and the statistical analysis is shown in Appendix Table A3. It was recognized that the heating process of CPs increased the intensity of dried shrimp, roasted chili, salt aromatic, and sweet aromatic notes. On the other hand, shallot note was not affected by heating. Garlic note is the predominant aroma notes in UH-CP. The intensity of garlic note decreased after the heat treatment. This result is not consistent with that obtained by DHDA and AEDA. In DHDA and AEDA, aroma compounds are separated by GC columns and individually evaluated by the panelists. In quantitative descriptive analysis, all aroma compounds are evaluated at once. Therefore, garlic note could be masked by the other aromas. Garlic note might not have actually decreased by heating process but perhaps it was simply dominated by other aroma notes in CP system. The pattern of aroma profiles of H25-CP and H50-CP were similar. There were no significant differences in the intensity of dried shrimp, roasted chili, garlic, and shallot notes between H25-CP and H50-CP. It can be presumed that these attributes were not affected by a long time of heat processing because the panelists could not detect the difference in the intensity of these attributes between H25-CP and H50-CP. However, H50-CP had a stronger salt aromatic note than HP25-CP. This implies that the excessive heating time of CP sample did not affect the overall aroma profiles of CPs.

Based on high FD factor and odor description of DHDA and AEDA data, 4-hydroxy-2,5-dimethyl-3-(2*H*)-furanone (no. **77**, HDMF; caramel/burnt sugar) probably contributed to the term sweet aromatic. The presence of 3-ethyl-2,5-dimethyl pyrazine (no **33,** green/roasted), 2,3-diethyl-5-methylpyrazine (no. **42,** roasted), 2-isobutyl-3-methoxypyrazine (no. **46,** bell pepper), benzaldehyde (no. **47,** chili/nutty)**,** and sotolon (no. **81,** curry/spicy) might contribute to the term roasted chili.

Furthermore, on the basis of its odorant of these aroma active compounds, methyl propyl disulfide (no. **21,** sulfurous/meaty), 3,4-dimethylthiophene (no. **22**, garlic/fishy), 1,3-dithiane (no. **23,** sulfurous/salty), allyl methyl trisulfide (no. **50,** meaty/garlic), and dipropyl trisulfide (no. **59**, meaty/garlic) were probably responsible for the combination of garlic, dried shrimp, and salt aromatic note.

The dried shrimp and salt aromatic terms could have been caused by 1-propanethiol (no. **5,** meaty), allyl mercaptan (no. **6,** meaty), allyl methyl sulfide (no. **9,** meaty/fishy), 2-methyl-3-furanthiol (no. **19,** meaty), and (*Z*)-4-heptenal (no. **20,** fishy/fatty).

The garlic and shallot terms might have been due to the presence of thiophene (no. **11,** garlic), 3,3'-thiobis-1-propene (no. **17,** sulfurous/ garlic), allyl methyl disulfide (no. **24,** fresh garlic), dimethyl di -and trisulfide (no. **14**, sulfurous/garlic and **31,** sulfurous/garlic), dipropyl disulfide (no. **30**, sulfurous/garlic), (*E*)-propenyl propyl disulfide (no. **37**, sulfurous), diallyl di- and trisulfide (no. **41,** fresh garlic and **66,** garlic), 3-vinyl-4*H*-1,2-dithiin (no. **61,** garlic), 2-vinyl-1,3-dithiine (no. **63,** garlic), and 2-vinyl-4*H*-1,3-dithiin (no. **70**, garlic).

However, there is a limit in the comparison of individual odorants with aroma attributes because the synergistic and antagonistic effects between the odorants and/or other constituents in the CPs are not considered. Therefore, it is necessary to investigate the flavor recombination experiments in detail to further clarify which aroma-active compounds were related to the CP aroma.

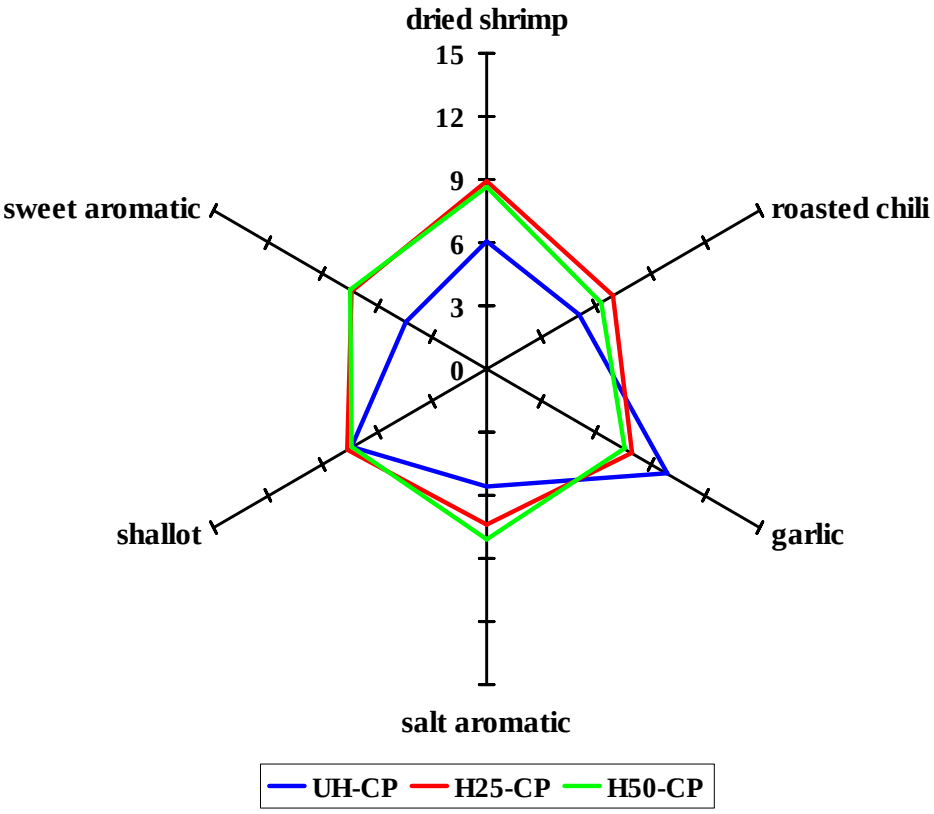


Figure 10 Descriptive aroma profiles of Thai chili pastes (CPs); UH-CP = unheated CP, H25-CP = CP heated at 100 °C for 25 min, and H50-CP = CP heated at 100 °C for 50 min.

2. Microencapsulation of chili paste oil by spray drying using maltodextrin and gum arabic as the wall materials.

Chili paste oil (CPO) was microencapsulated by spray-drying using maltodextrin DE 18.5 (MD) and gum arabic (GA) as the wall materials. Three different MD:GA ratios, 1:2, 1:1 and 2:1, were used. The powder was prepared at a constant total solid content of 32% w/w and the ratio of wall materials to the CPO was 4:1. Most spray-dried products are prepared with solids to flavor ratio of 4 to 1 (Reineccius, 1988). This ratio has been reported to give high flavor retention for wall materials such as gum arabic and other carbohydrate derivatives (Bhandari *et al*.,

1992, Beristain *et al*., 2001). In this study, total oil, surface oil and encapsulation efficiency (EE) are shown in Table 10.

Table 10 Total oil, surface oil and encapsulation efficiency of CPO powders as a function of maltodextrin DE 18.5 and gum arabic ratio.

Matrix component Maltodextrin:Gum arabic	Total oil (g/100g)	Surface oil (g/100g)	Encapsulation efficiency (%)
1:2	18.94 ± 0.12 b	4.15 ± 0.14 a	78.11 ± 0.89 b
1:1	17.53 ± 0.35 a	4.17 ± 0.10 a	76.19 ± 0.10 a
2:1	18.32 ± 0.17 ab	4.63 ± 0.05 b	74.73 ± 0.49 a

[a] Mean ± standard deviation (n=2). Means in a column followed by different letters are significantly different (P<0.05).

2.1 Total oil

Total oil was determined by refluxing and solvent extraction method. The total oil contents of the three spray-dried, microencapsulated products ranged from 17.53% to 18.94% (Table 10). The 1:2 ratio of MD to GA sample had higher total oil than the 1:1 and 2:1 ratios of MD to GA. No significant differences (P>0.05) were found on the total oil content within each of the MD/GA ratios of 1:1 and 2:1.

2.2 Surface oil

The surface oil can be easily oxidized to form off-flavor compounds. The amount of surface oil in spray dried powder is quite important for stable storage (Soottitantawat *et al.*, 2003). The surface oil content was determined by washing method. In the 2:1 ratio of MD to GA, surface oil content was the highest whereas in the MD/GA ratios of 1:2 and 1:1, surface oil contents were similar (Table 10).

2.3 Encapsulation efficiency (EE)

The encapsulation efficiency (EE) of spray-dried powders increased from 74.73% to 78.11% (Table 10). The highest EE was 78.11% for the 1:2 MD/GA mixture. By contrast, the lowest EE was observed at a MD/GA ratio of 2:1. It has been reported that the film forming property and the fine stable emulsion are requirements for the efficient encapsulation. The film former must also be capable of forming fine oil-in-water emulsion (Trubiano, 1995). Maltodextrin offers the advantages of being relatively inexpensive, flavorless, and low in viscosity at high solid content. However, it lacks lipophilic characteristic and does not have emulsion-stabilizing property (Pegg and Shahidi, 1999). The superior properties of gum arabic can be explained by its emulsification property and its tendency to form films at the interfaces between the emulsion phases (Thevenet, 1995). It was seen that the combination containing higher proportions of gum arabic provides better EE value. As mention above, the higher surface oil content and lower EE value of the 2:1 ratio of MD/GA product indicated that this sample was least able to encapsulate the CPO during spray-drying.

2.4 Water activity

The water activity of spray-dried encapsulated CPO at 25 °C remained between 0.17% and 0.20% (Table 11). The results showed no effect of the different combinations of maltodextrin and gum arabic on the water activity of the powders.

2.5 Moisture content

Results of the moisture analyses showed that the percentage moisture in the spray-dried powders varied from 2% to 5% (Table 11). Similar results were obtained by Reineccius *et al*. (1995) for moisture contents of spray-dried gum acacia and maltodextrin powders. The moisture contents of the product powders with higher ratios of gum arabic were higher, while the moisture content of the products decreased when the amount of maltodextrin increased. These results indicated that gum arabic

exhibited greater hygroscopicity than maltodextrin. For all combinations of the carrier solid, the increasing moisture content resulted in an increasing water activity.

Table 11 Water activity, moisture content and glass transition temperature of the spray-dried encapsulated chili paste oil with a varying ratio between maltodextrin DE 18.5 and gum arabic.[a]

Matrix component Maltodextrin:Gum arabic	Water activity at 25 °C	Moisture content (%)	Glass transition (°C)
1:2	0.20 ± 0.07 a	4.12 ± 0.75 b	82.87 ± 2.65 a
1:1	0.17 ± 0.00 a	2.77 ± 0.30 a	93.92 ± 4.25 b
2:1	0.18 ± 0.06 a	3.62 ± 0.79 ab	96.97 ± 2.24 b

[a] Mean ± standard deviation (n=2). Means in a column followed by different letters are significantly different (P<0.05).

2.6 Glass transition temperature (Tg)

As environmental relative humidity at ambient condition (68-73% RH, 25-30 °C), glass transitions of spray-dried powders occurred at 82.87 to 96.97 °C (Table 11). All samples generally present relatively high Tg values. Moreover, all Tg values were above the storage condition (68% RH, 50 °C) and the storage was done below Tg; therefore, no collapse would have been expected. Thus, the samples should be in an amorphous state when stored in the controlled condition at the same relative humidity of ambient condition because there were no sign of caking and collapse taking place in the matrix during storage for 5 weeks. In the glassy state, the amorphous matrix is considered to be relatively stable (Roos and Karel, 1990). Furthermore, Levi and Karel (1995) suggested that flavor compounds in amorphous matrixes are retained at temperature below Tg. In this study, the Tg increased when the proportion of maltodextrin increased from 1:2 to 1:1 ratio of MD-GA mixtures at the same relative humidity because of lower water content. It has been reported that

the Tg increased as the moisture content within each matrix decreased because of the plasticization role of water (Roos, 1995). On the other hand, the 1:1 and 2:1 MD-GA mixtures had no significant differences (P>0.05) in the Tg values.

2.7 Flavor release of selected volatiles from spray-dried microencapsulated CPO by dynamic headspace analysis (DHA)

DHA technique was used to determine the release of volatiles from the spray-dried microencapsulated CPO, with surface oil removed, at 30 °C as a function of purge times for up to 60 min. In this study, five major volatile compounds in CP were selected for determination of flavor release. There were butanoic acid, 3-methylbutanoic acid, diallyl disulfide, benzaldehyde, and phenol. The molecular weight, boiling point and solubility in water of these compounds are presented in Appendix Table B1.

The recoveries of volatile compounds that were released into the headspace of each spray-dried microencapsulated products are shown in Figure 11a-11e. The results revealed that volatiles from different samples were released at different rates by each of the microencapsulated products and observed to increase during storage. The rate of release could depend on various factors, such as different chemical structures and properties of wall materials, the binding capacity of matrices to lock CPO droplets, different physicochemical properties of individual aroma components, and storage conditions (Baranauskiené *et al*., 2007).

The total amount of volatiles released during 5 min of purge treatment varied from 6.20 ng/g for 3-methylbutanoic acid of the MD/GA (1:2) sample to 25.59 ng/g for phenol of the MD/GA (2:1) sample (Appendix Table B2). The range of oil volatile concentrations during 60 min purge was from 35.51 ng/g for butanoic acid of the MD/GA (1:1) sample to 261.67 ng/g for benzaldehyde of the MD/GA (1:1) sample. The highest release rates of butanoic acid, 3-methylbutanoic acid, diallyl disulfide and phenol from the powders were observed during the first 5 min of purging period. Among the selected volatiles, benzaldehyde was released at the

highest rate. The most intensively permeability of this compound from microencapsulated products was observed during the first 30 min of the purge treatment (Figure 11d). This compound was released from 17.49 ng/g for the MD/GA (2:1) sample to 261.67 ng/g for the MD/GA (1:1) sample (Appendix Table B2). This could be caused by partially water soluble compounds. It was found that the dissolved flavor could increasingly diffuse through the wall matrices, resulted in a higher release of flavor (Soottitantawat *et al.*, 2005). However, the release rate of this compound from all three samples were not significantly different ($P>0.05$) during purging (Appendix Table B2).

The MD/GA (2:1) sample exhibited higher release of butanoic acid during purging than other samples (Figure 11a). It might be caused by the high percentage of proteins in arabinogalactan protein fraction and glycoprotein fraction of gum arabic. . These proteins have both hydrophilic and hydrophobic properties (Thevenet, 1995). Therefore, the partial replacement of MD by GA had more effectively extended flavor release. In comparison between the release rates of aliphatic carboxylic acid compounds of a MD/GA (2:1) sample, 3-methylbutanoic acid had lower release rate than butanoic acid (Figure 11a and 11b). This was possible because 3-methylbutanoic acid had higher molecular weight than butanoic acid. Molecules that have higher molecular weight also have larger size and slower diffusion rate (Reineccius, 1988).

The release rates of 3-methylbutanoic acid and diallyl disulfide were not significantly different ($P>0.05$) among the samples (Appendix Table B2). On the other hand, phenol, a water soluble compound, was released from the MD/GA (2:1) sample at a slower rate than from the MD/GA (1:2) sample (Figure 11e). This is because the hydrophilic MD could well retain the water soluble aroma (Pegg and Shahidi, 1999).

The most effective matrix to retain CPO volatiles was the MD/GA (1:2) sample (Appendix Table B2). The amount of emitted volatiles in the headspace of this spray-dried product varied from 6.20 to 213.93 ng/g. Furthermore, the amount of

the released volatiles of the MD/GA (1:1) sample (7.30 to 261.67 ng/g) was similar to the MD/GA (2:1) sample (8.95 to 251.85 ng/g).

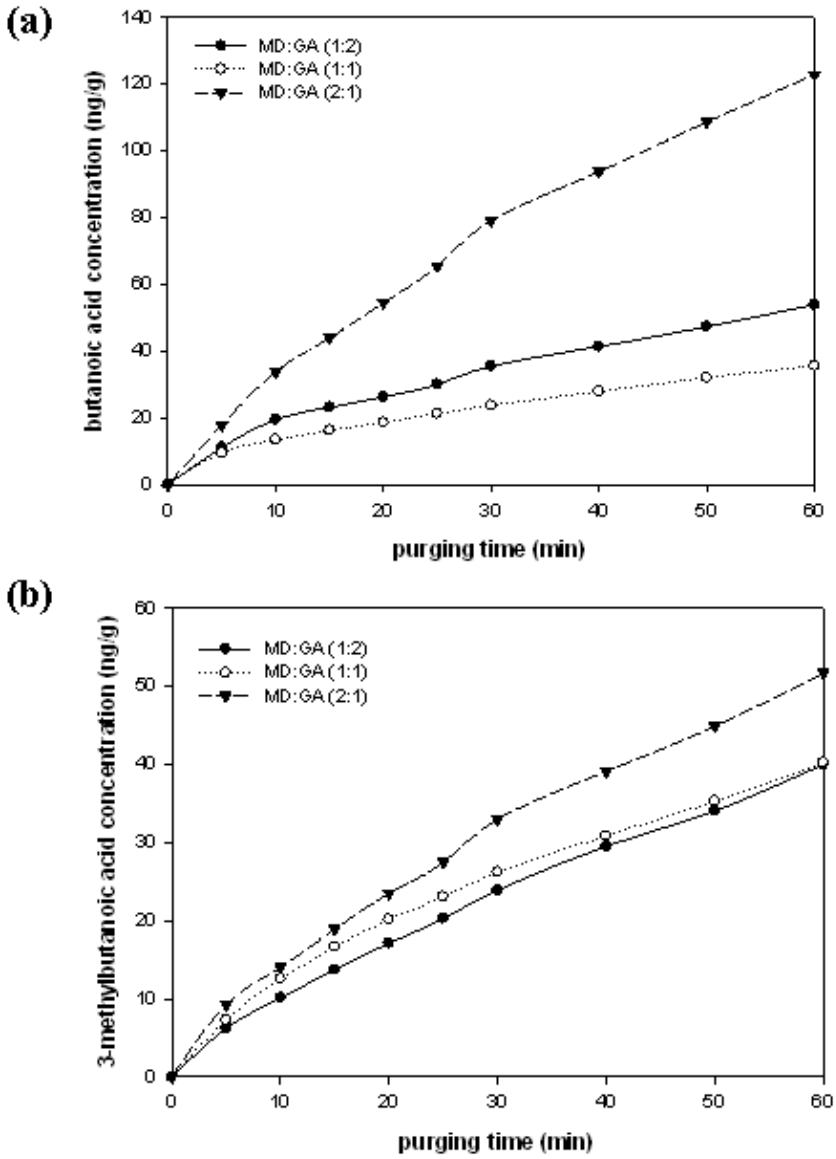


Figure 11 Amounts of major volatiles released from CPO encapsulated with various ratios of MD:GA at different purging time in the dynamic headspace analysis; (a) butanoic acid, (b) 3-methylbutanoic acid, (c) diallyl disulfide, (d) benzaldehyde and (e) phenol.

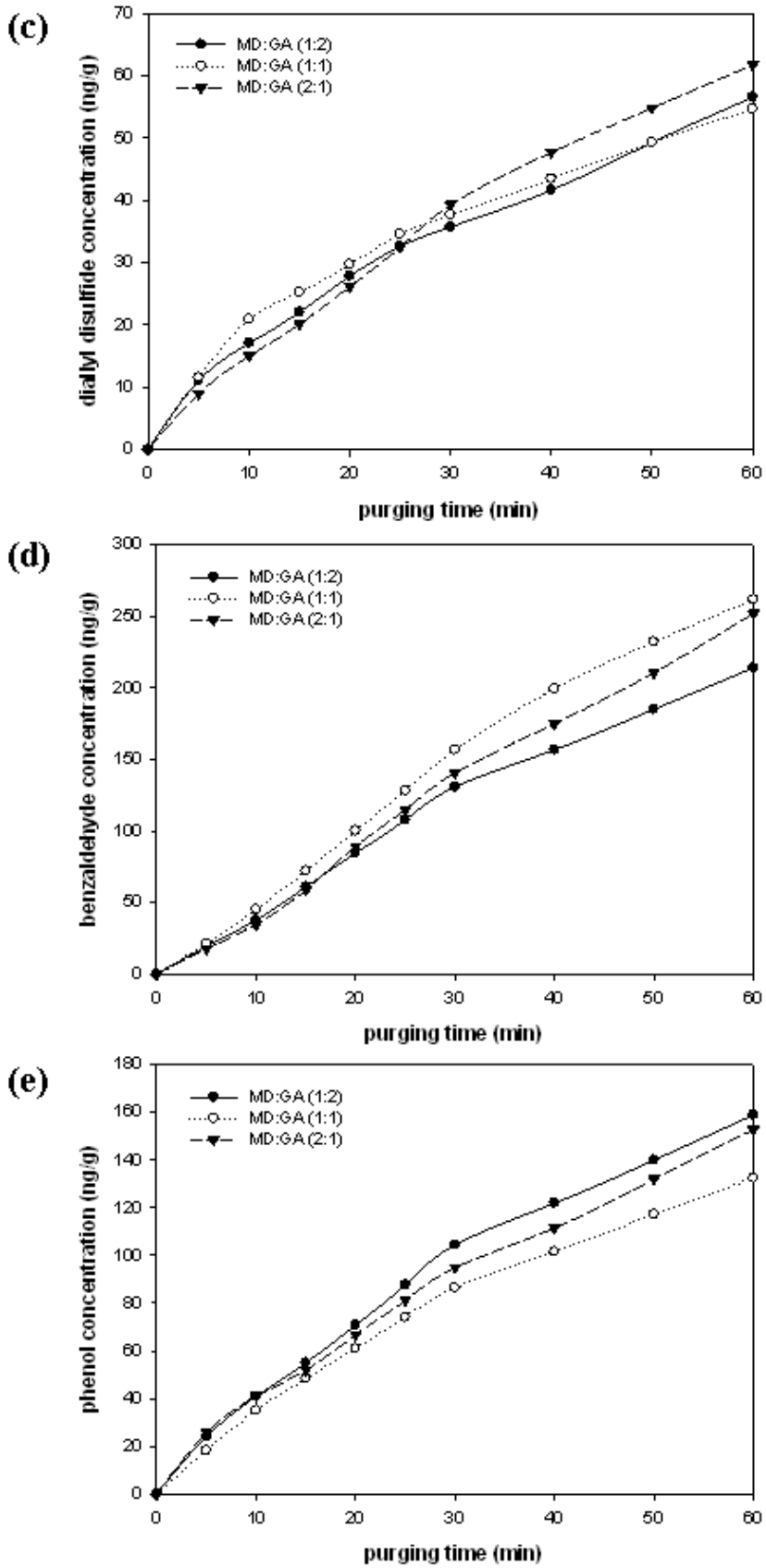


Figure 11 (Continued)

2.3 Flavor retention of selected volatile compounds in CPO powders

CPO encapsulation was prepared by different proportions of maltodextrin DE 18.5 and gum arabic. Maltodextrin DE 18.5 was chosen because higher dextrose-equivalent (DE) maltodextrin offered better protection for encapsulated compounds. Protection from oxidation is probably because of a more dense of capsule. The higher DE maltodextrins have abundant glucose monomers that can bind hydroperoxides and thereby exhibit an antioxidant effect (Anandaraman and Reineccius, 1986; Wagner and Warthesen, 1995).

Microcapsules were dried using spray drying. After that, powders were washed with mixed solvents (hexane:diethyl ether; (1:1)) to remove their surface oil and to oxidative compounds that generated from unencapsulated oil during storage. The accelerated condition of storage experiment was carried out at 50 °C with 68% RH for 5 weeks. The samples were drawn every week for flavor quantification. The flavors were extracted from the complex by using diethyl ether followed by the isolation of volatiles by high vacuum distillation. The aroma extracts were analyzed by GC-MS. Flavor retention expressed as percentage of the flavor content in the powder during storage based on the highest content of the compound among three spray-dried samples at the initial time of storage.

The retentions of each selected volatile compounds are shown in Figure 12a-12p and Appendix Table B3. The molecular weights, odor thresholds in water, boiling points and water solubility property of the selected volatiles are presented in Appendix Table B1. Seventeen major volatile compounds in CPO were selected to study for their retentions based on two criteria. First, volatiles had low odor thresholds and second, volatiles had high FD factors as indicated in DHDA and AEDA data.

The retention of selected volatile compounds decreased through 5 weeks of storage except hexanal, which increased during storage (figure 12c) might have originated from soybean oil, which is the component of CP ingredients. It has been

reported that hexanal is a principal component in both unheated and heated soybean oil (Selke and Frankel, 1987; Wu and Chen, 1992). Hexanal was probably degraded from the autoxidative reaction of 13-hydroperoxide of linoleate (Frankel, 1985). It is the indicator for the development of off-flavor in vegetable oil (Warner *et al*., 1978). After 4 weeks of storage, hexanal in the CPO encapsulated with 2:1 MD/GA showed higher content compared to the sample that were encapsulated with 1:2 MD/GA (Figure 12c). The content of hexanal in a MD/GA (2:1) sample at the end of storage was more than 70 times higher than the initial. It can be assumed that increasing the proportion of MD in the wall system provided less protection against oxygen. In related research, Thenevet (1995) reported that the orange oil in spray-dried encapsulated powders was stable against oxidation in pure GA and in the GA/MD systems containing higher proportions of GA.

At the initial time of storage, the lowest amount of detected volatiles in all samples was dimethyl disulfide (0.38 ng/g), followed by 3-methylbutanal (0.83 ng/g) in a MD/GA (2:1) sample (Appendix Table B3). After 2 weeks, all three samples could not retain dimethyl disulfide in the capsules (Figure 12b). Moreover, 3-methylbutanal was only retained in the MD/GA (1:2) sample at the end of storage (Figure 12a). This is because dimethyl disulfide and 3-methylbutanal are highly volatile, very slightly soluble in water, and have low boiling points. The low flavor retention of these compounds could be cause by the highly volatile compounds might be lost by evaporization during the spray-drying process that used high temperature condition (drying temperature: inlet, 165 °C; outlet, 85 °C). The loss of volatiles during the spray-drying process can be explained by the mechanism of selective permeability. This assumes that the retention of the compound in the atomized drop is a function principally of parameters related to the relative volatility of the compounds. After the skin that constitutes the wall of the covering has been formed, other factors emerge and become preponderant in the parameters which control the phenomenon of diffusion through the wall (Thijssen and Rulkens, 1968). On the other hand, the greater polarity, and consequently the greater solubility of the encapsulated compounds in an aqueous medium, results in a greater capacity for diffuse through the matrix during the spray-drying process, leading to greater losses

during the formation of the capsules (Rosenberg *et al*., 1990; Voilley, 1995). Similarly, it has been reported that the highly volatile flavor components (those with low boiling points) are less effectively encapsulated and more quickly loss than less volatile components (Zeller *et al*., 1999). Furthermore, retention of flavor in the spray-dried powders is governed by factors related to the chemical characteristic of the flavor compounds, including its molecular weight, chemical functionality polarity and relative volatility (Bertolini *et al.,* 2001).

Phenylacetaldehyde was found in spray-dried CPO powders only at the beginning of storage time (Appendix Table B3). This compound could not be retained during storage. Moreover, benzaldehyde was also not detectable after 5 weeks of storage (Figure 12i). These results could be explained by the greater solubility of these polar compounds in water. The polar volatile is more soluble in water and can also diffuse more easily through the matrix (Rosenberg *et al*., 1990).

At the initial time of storage, the highest content of detected volatiles was observed for diallyl trisulfide (222.31 ng/g), followed by benzaldehyde (87 ng/g) and 2-vinyl-4*H*-1,3-dithiin(68.4 ng/g) in the MD/GA (1:2) sample (Appendix Table B3). Additionally, the highest contents of 1,3-dithiane (5.03 ng/g), allyl methyl disulfide (2.90 ng/g), diallyl disulfide (32.85 ng/g) were presented in a MD/GA (1:2) sample. On the other hand, the lowest contents of 1,3-dithiane (1.92 ng/g), allyl methyl disulfide (2.23 ng/g), dimethyl trisulfide (10.52 ng/g), 3-vinyl-4*H*-1,2-dithiin (7.23 ng/g) and 2-vinyl-4*H*-1,3-dithiin (45.23 ng/g) were detected in a MD/GA (2:1) sample (Appendix Table B3).

Excellent flavor retentions at the end of storage were achieved for allyl methyl disulfide (48.23 %), dimethyl trisulfide (43.08 %) and 3-methylbutanoic acid (73.19 %) in a MD/GA (1:2) sample (Figure 12 (e, g, k, and p) and Appendix Table B3). Furthermore, among the sulfur containing compounds detected in this study, 1,3-dithiane (22.65%), 3-vinyl-4*H*-1,2-dithiin (31.40%) and 2-vinyl-4*H*-1,3-dithiin (17.06%) exhibited the highest retention with a MD/GA (1:2) sample (Appendix Table B3). By contrast, the percentage of flavor retentions of 1,3-dithiane (14.17%),

allyl methyl disulfide (23.90%), dimethyl trisulfide (15.52%), and 3-vinyl-4*H*-1,2-dithiin (17.45%) were lower in a MD/GA (2:1) sample than in that of the 1:2 and 1:1 ratios of MD/GA samples. Moreover, 3,4-dimethylthiophene was detectable after 4 weeks of storage only in the case of MD/GA (1:2) sample (Figure 12f). The loss of 3,4-dimethylthiophene was possible due to the second lowest boiling point of this volatile among the identified sulfur containing compounds.

It seems that the retention of the low water solubility compounds (benzaldehyde, allyl methyl disulfide, dimethyl trisulfide and 3-methylbutanoic acid) and water insoluble compounds (diallyl di and trisulfide) in spray-dried CPO powders increased when the GA fraction increased. 2-Vinyl-4*H*-1,3-dithiin and 3-vinyl-4*H*-1,2-dithiin also showed high retention in a MD/GA (1:2) sample at the end of storage. The solubility of these compounds, however, are not available. It has been reported that a remarkable production of 2-vinyl-4*H*-1,3-dithiin and 3-vinyl-4*H*-1,2-dithiin from allicin is observed when less-polar solvents are used and they are rich in the oil macerate of raw garlic (Iberl *et al*., 1990). Therefore, these compounds might be less water soluble compounds. Furthermore, among the sulfur containing compounds studied, these vinyldithiins present high molecular weights. In general, an increase in the size of the molecule reduces diffusion and, as a consequence, retards migration of the compounds to the surface of the matrix, increasing their retention (Reineccius, 1988; Rosenberg, 1990).

The retention of volatiles in spray-dried powders was strongly dependent on the type of solid matrix used. The previously published studies showed that the retention of volatiles compounds increase with increase GA in the MD/GA systems (Sankarikutty *et al*., 1988; Apintanapong and Noomhorm, 2003). GA has emulsification properties and its tendency to form films at the interfaces between the emulsion phases and its improved emulsifying/stabilizing properties (Bhandari *et al*., 1992). Improved film forming or increased areas of hydrophobicity in the drying matrix would be expected to improve volatile retention. On the other hand, MD lacks lipophilic properties and emulsion-stabilizing effects on water insoluble volatile

compounds and, consequently, do not retain water insoluble aromas well (Rosenberg *et al*., 1990; Pegg and Shahidi, 1999; Reineccius *et al*., 2003).

During the beginning of storage, the content of butanoic acid (54.23 ng/g) was highest in a MD/GA (2:1) sample (Appendix Table B3). This might be caused by the water solubility of butanoic acid. However, polar volatiles that are more soluble in water, can diffuse more easily through the matrix causing lower retention during storage (Rosenberg *et al*., 1990). This finding suggested that the retention of butanoic acid was lower in a MD/GA (2:1) sample (23.71%) as compared with a MD/GA (1:2) sample (34.84%) at the end of storage (Appendix Table B3).

However, the amount of hexanoic acid (insoluble straight chain carboxylic acid) in all samples were not significantly different ($P>0.05$) during 5 weeks (Appendix Table B3). Furthermore, phenol was 71.98-74.60% retained after 5 weeks of storage (Appendix Table B3). In the study of flavor release, it was found that the MD/GA (2:1) sample showed lower release rate than the MD/GA (1:2) sample but in this study, there were no significant different ($P>0.05$) in the remaining content of this compound among spray-dried CPO samples at the end of storage.

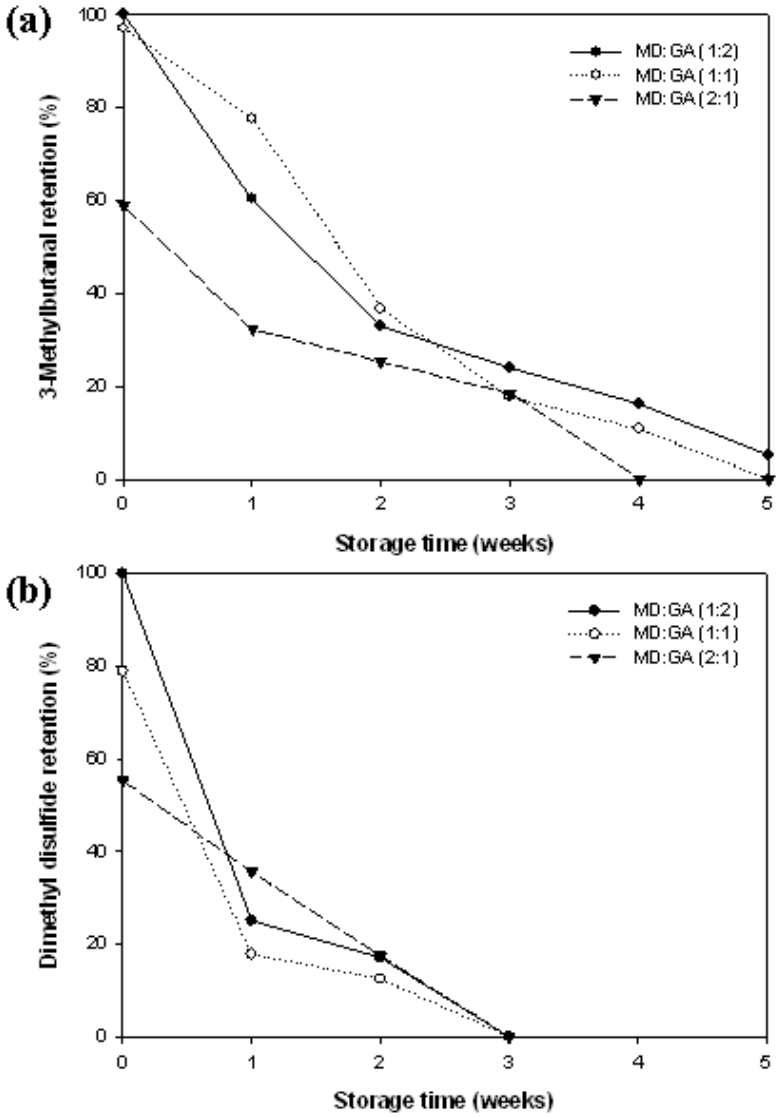
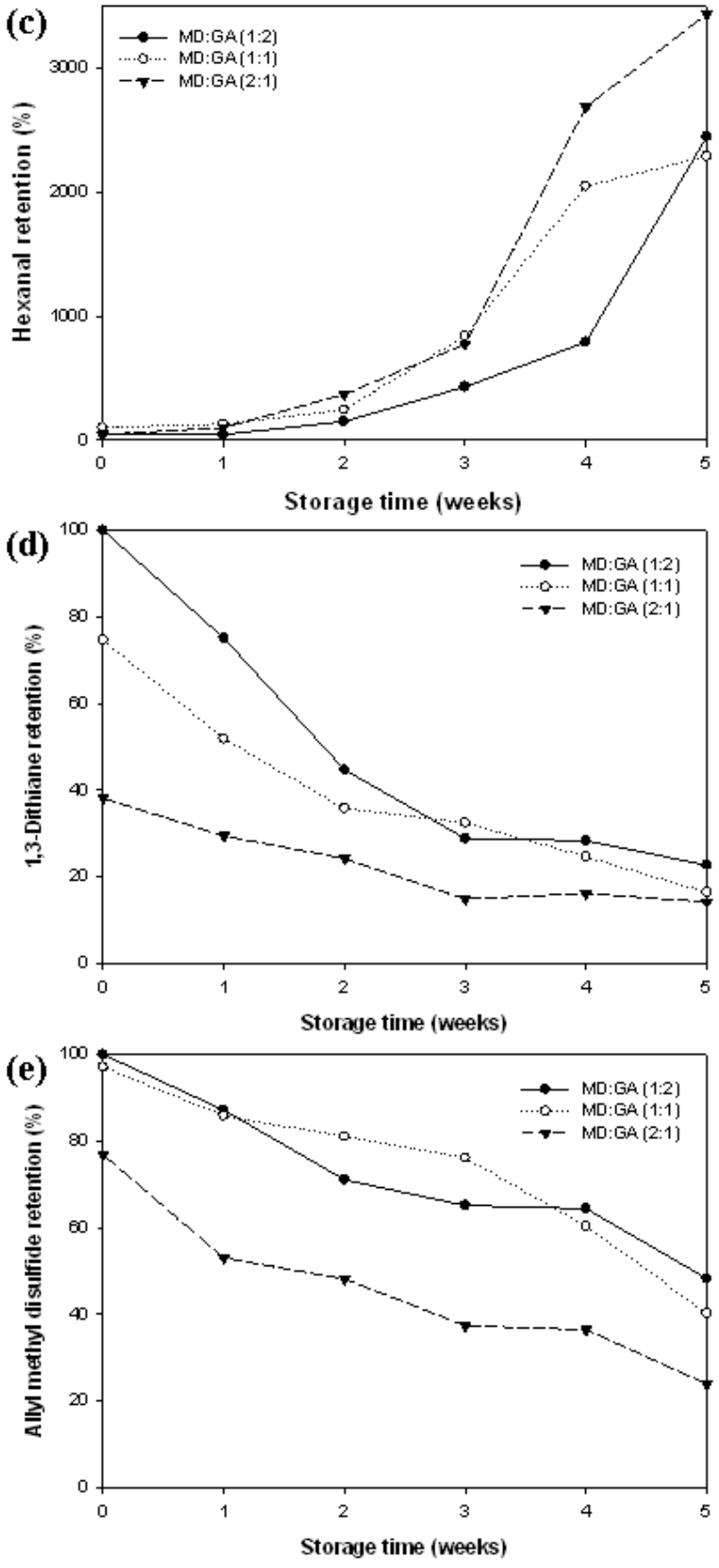


Figure 12 Flavor retention of selected volatile compounds in spray-dried CPO microcapsules with various ratios of MD:GA during storage at 50 °C and 68% RH for 5 weeks: (a) 3-methylbutanal; (b) dimethyl disulfide; (c) hexanal; (d) 1,3-dithiane; (e) allyl methyl disulfide; (f) 3,4-dimethylthiophene; (g) dimethyl trisulfide; (h) diallyl disulfide; (i) benzaldehyde; (j) butanoic acid; (k) 3-methylbutanoic acid; (l) 3-vinyl-4*H*-1,2-dithiin; (m) diallyl trisulfide; (n) 2-vinyl-4*H*-1,3-dithiin; (o) hexanoic acid; and (p) phenol



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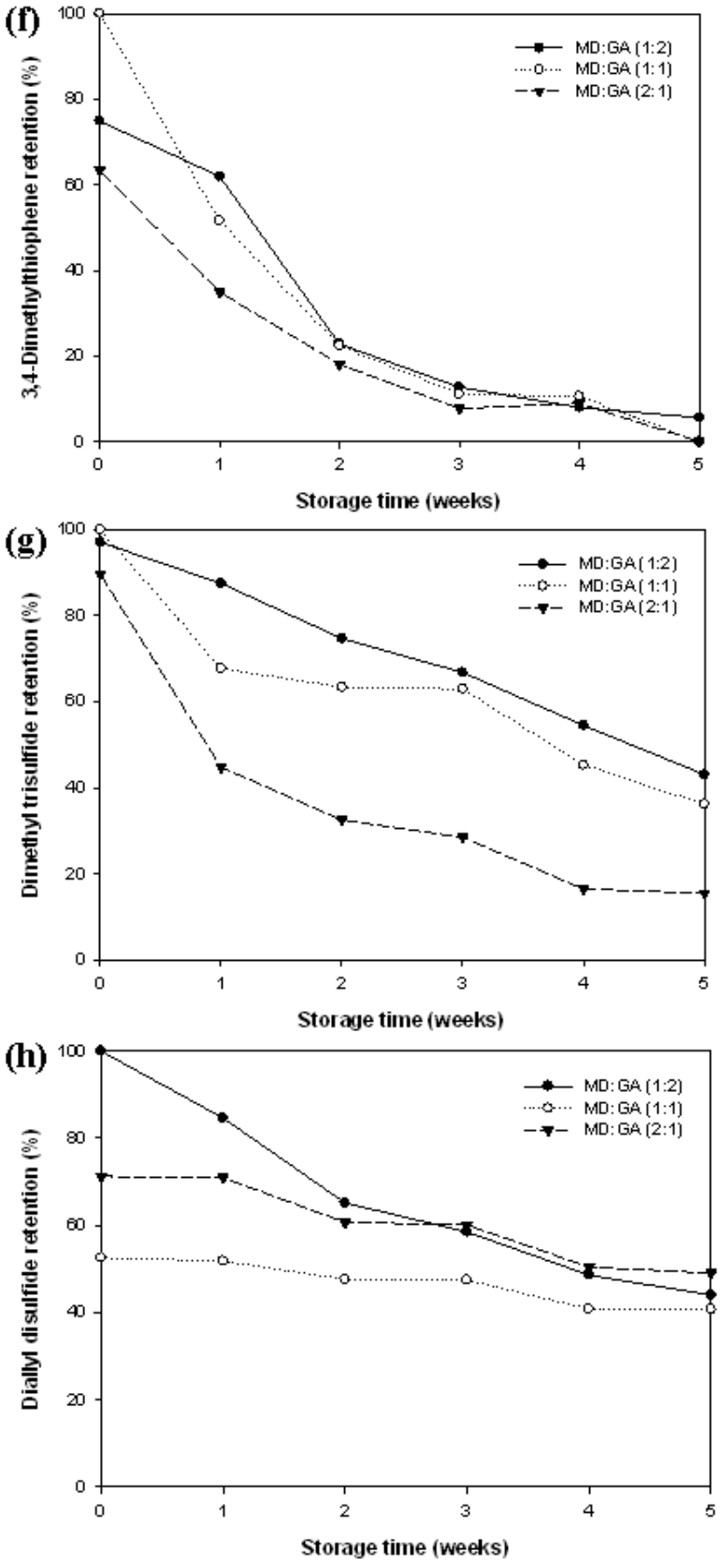

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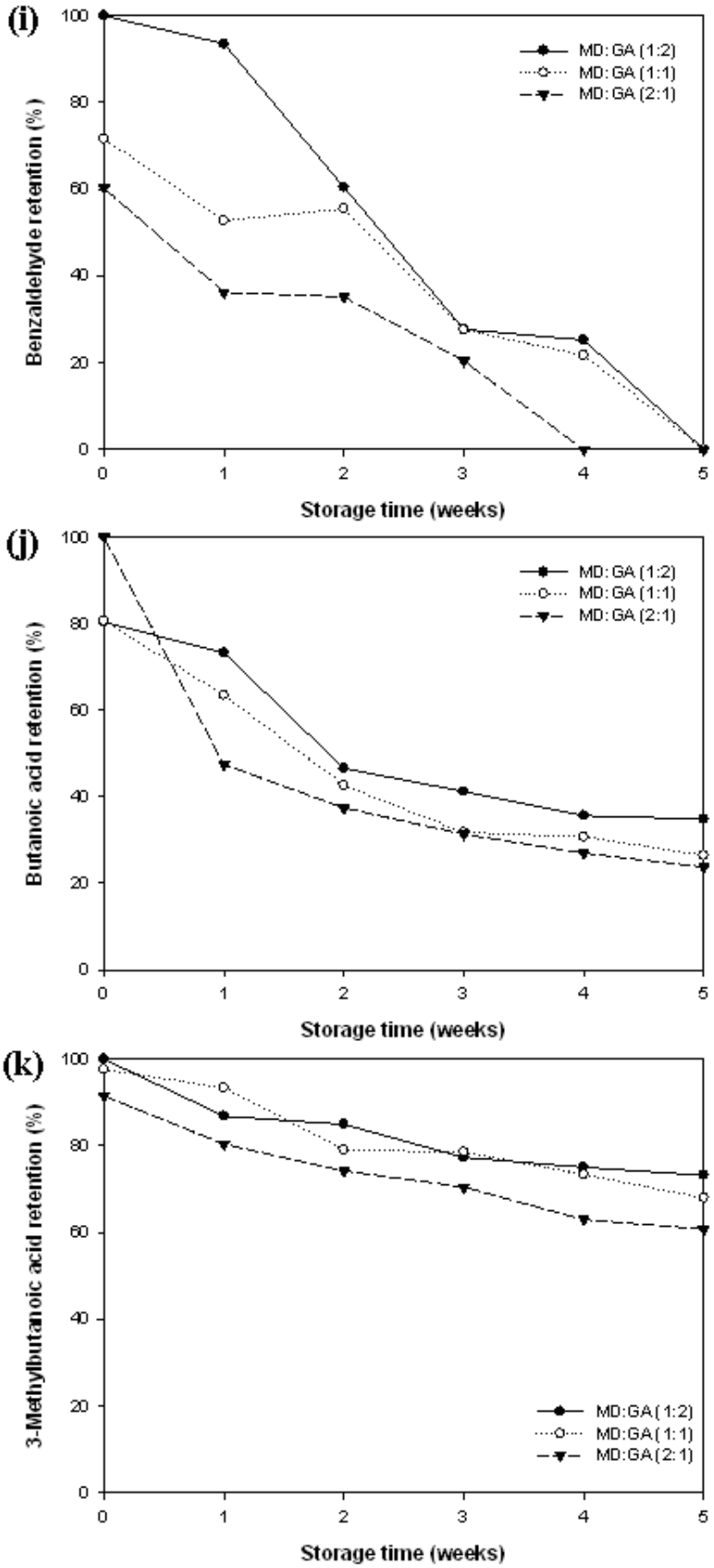

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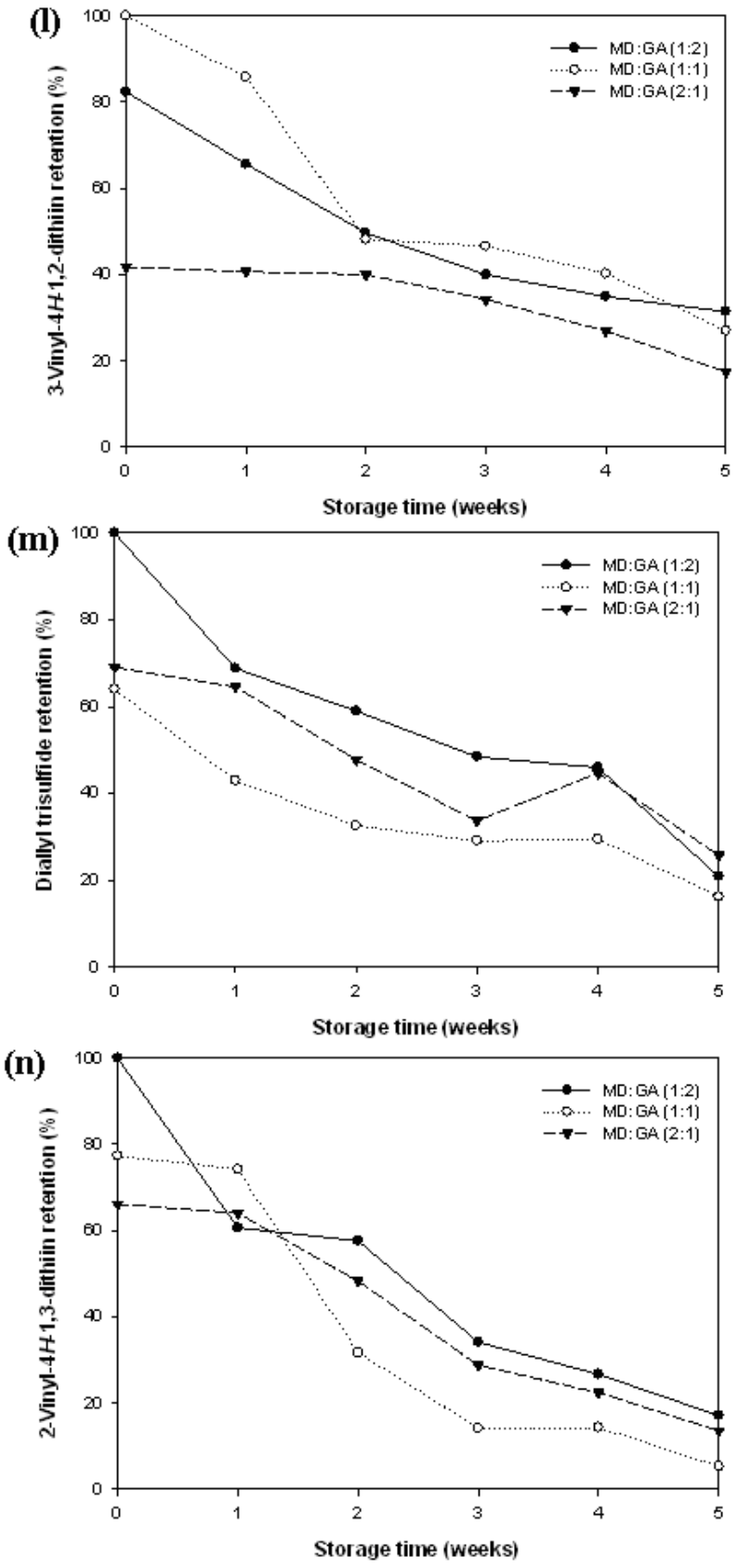

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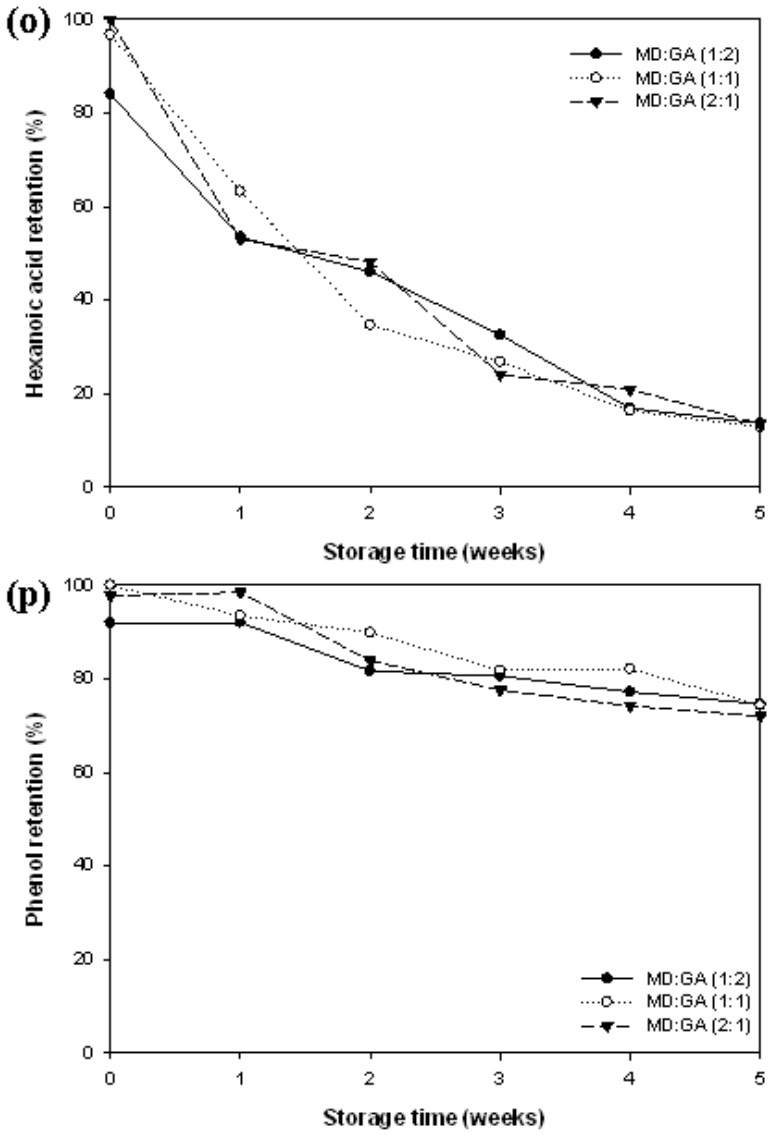

Figure 12 (Continued)

2.4 Sensory evaluation of spray-dried microencapsulated CPO

Quantitative Descriptive Analysis (QDA) was used to evaluate changes of aroma profiles of spray-dried CPO powders during storage at 50 °C and 68% RH for 5 weeks. The powder had been washed with solvent mixture before storage. The samples were drawn every week for the sensory evaluation. The CPO powder solutions (5% in deodorized distilled water) were placed (20 mL) in a glass vial with a cap and presented to the trained panelists (3 male and 9 female panelists, ranging in age from 23-32 years old). Six aroma descriptors of CPO powder solution were selected by panelists. There were chili aroma, shrimpy aroma, garlic aroma, salt aromatic, sweet aromatic and overall aroma intensity. Perceived intensities were scored on an unstructured 15- cm line scales. The changes of these aroma attributes in the CPO powders during storage are presented in Figure 13 a-f and Appendix Table B4.

The results from QDA showed that among six aroma attributes in all treatments at the initial time of storage, the overall aroma intensity had the highest intensity (6.49-6.66 points), whereas the sweet aromatic had the lowest intensity (5.17-5.31 points) (Appendix Table B4). At this point, no significant differences (P>0.05) were found in the chili note, shrimpy note, garlic note, salt aromatic, sweet aromatic, and overall aroma intensity among three samples detected by the panelists (Figure 13 a-f). At the end of storage time, the intensity of shrimpy note of the 2:1 ratio of MD/GA was rated slightly higher than the MD/GA (1:1) sample (Figure 13b). Moreover, a MD/GA (1:2) sample had slightly lower garlic aroma than the 1:1 ratio of MD/GA (Figure 13c). However, there were no significant differences in the intensities of chili note, salt aromatic, sweet aromatic and overall aroma intensity among all samples after week 5 of storage. This reveals that the panelists could not detect the variation in the intensities of most attributes both at the beginning and the end of storage in the samples that were encapsulated with different combinations of MD and GA.

The intensity of sweet aromatic remained constant throughout the storage time in all samples (Figure 13e). Moreover, the constant of aroma intensity values during storage were observed for chili aroma in the 1:2 and 2:1 ratios of MD/GA samples, shrimpy aroma in the 1:2 and 1:1 ratios of MD/GA samples, garlic aroma in the 1:1 and 2:1 ratios of MD/GA samples, salt aromatic in the MD/GA (1:1) sample and overall aroma intensity in the MD/GA (2:1) sample (Figure 13 a-d and f). Storage time affected the intensities of shrimpy note and salt aromatic in the MD/GA (2:1) sample with week 5 being slightly lower than week 0 of storage but these were not significantly different in the MD/GA ratios of 1:2 and 1:1 (Figure 13 b and d). After 5 weeks of storage, the perceived aroma intensities of garlic note and overall aroma intensity of a MD/GA (1:2) sample were slightly lower than at the beginning time of storage, whereas there were no significant differences in the intensities of these attributes between week 0 and week 5 for the 1:1 and 2:1 ratios of MD/GA samples (Figure 13 c and f). This may be implied that the intensities of most attributes had the trend to remain constant in each spray-dried CPO powder samples during storage time for 5 weeks.

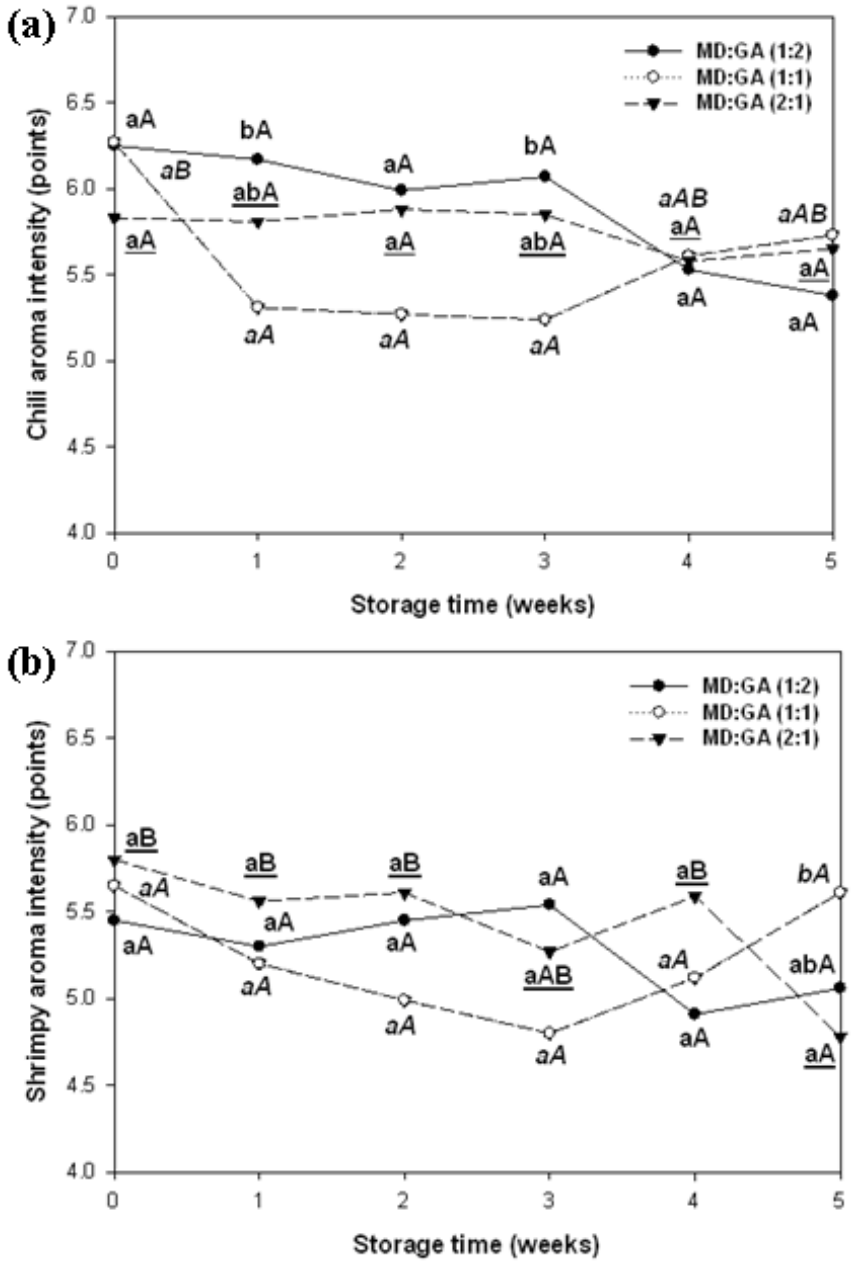

Figure 13 Changes in (a) chili aroma, (b) shrimpy aroma, (c) garlic aroma, (d) salt aromatic, (e) sweet aromatic and (f) overall aroma intensity of spray-dried microencapsulated CPO with various ratios of MD:GA throughout storage at 50 °C and 68% RH for 5 weeks (0-15 points). a and b means within each week with different letters are significantly different (P<0.05); A and B means within each treatment with different letters are significantly different (P<0.05).

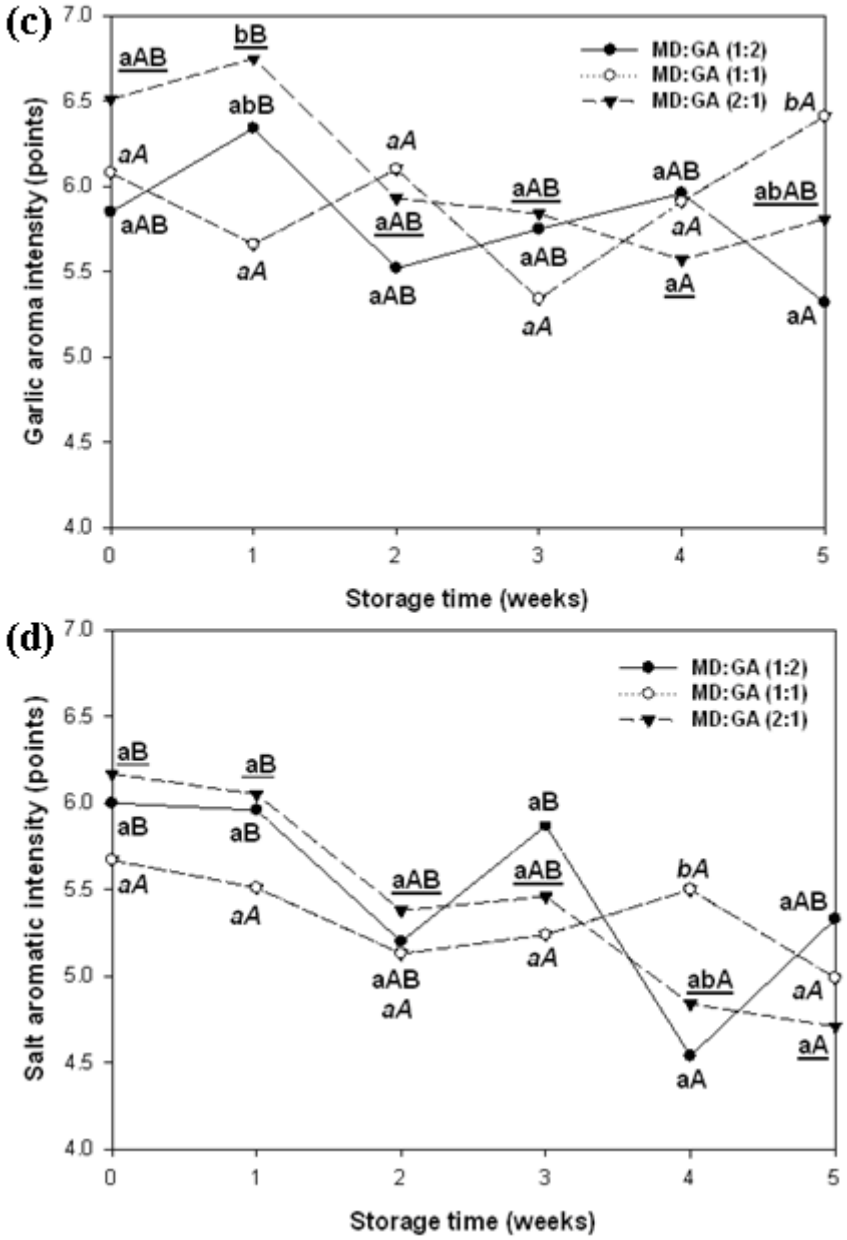

Figure 13 (Continued)

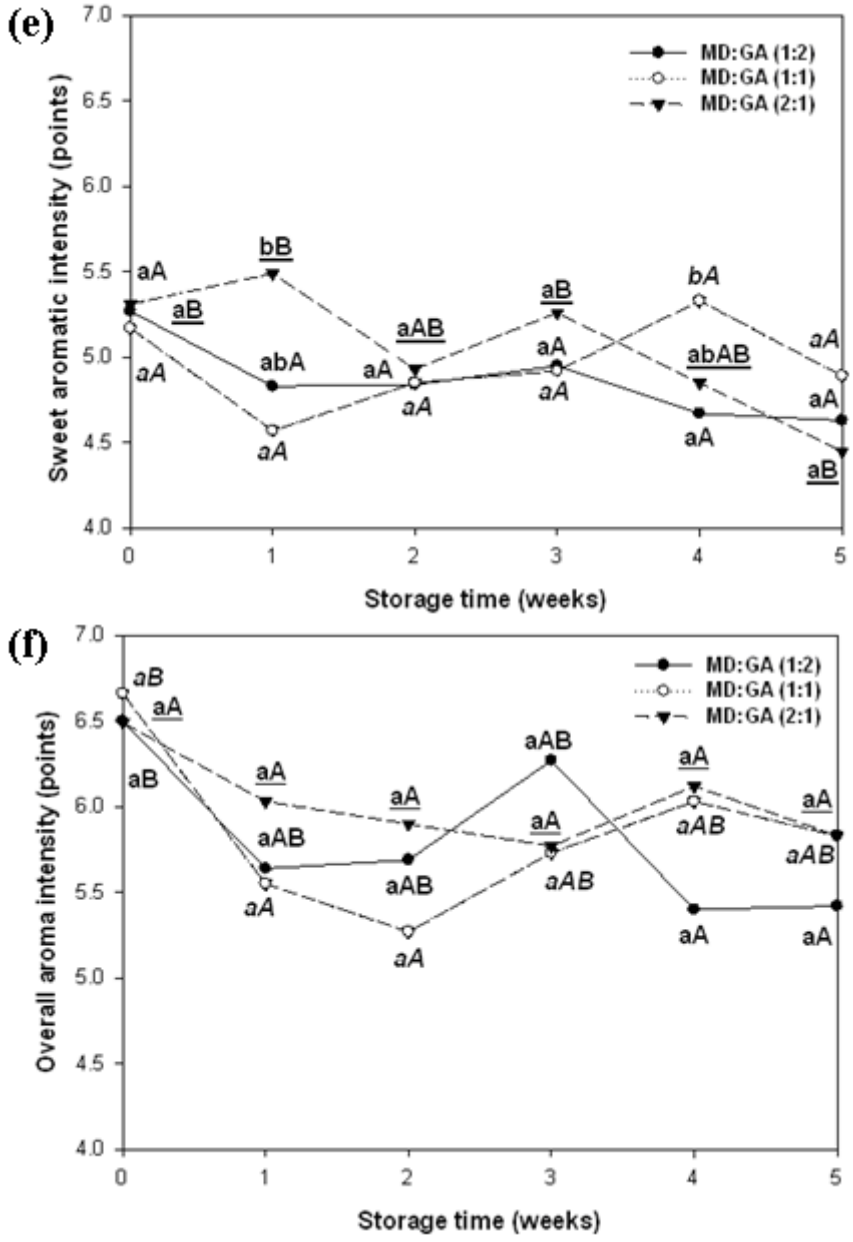

Figure 13 (Continued)

CONCLUSION AND RECOMMENDATION

On the basis of their high FD factor of 25 in dynamic headspace dilution analysis (DHDA) in all treatments, sulfur containing compounds (nos.**3**, **6**, **17**, **21**, **24**, **31**, **36**, **37**, **41**, **50**, and **61**) may play important roles in the formation of Thai chili paste flavor. The odor activity values (OAV) revealed dimethyl trisulfide (no.**31**), allyl mercaptan (no.**6**), diallyl disulfide (no.**41**), and dimethyl sulfide (no.**3**) as the most important aroma-active compounds with high OAVs. Additionally, allyl mercaptan having a meaty and bologna-like odor is the highly volatile that had a highest FD factor in DHDA but is of low FD factor (log_3FD factor < 2) in AEDA. On the basis of their high relative abundance and high FD factors, allyl methyl disulfide (no.**24**), allyl methyl trisulfide (no.**50**), and 3-vinyl-4*H*-1,2-dithiin (no.**61**) were predominant in Thai chili paste. Other potent aroma-active compounds included 2/3-methylbutanal (nos.**7** and **8**), 2,3-butanedione (no.**10**), 1-octen-3-one (no.**26**), (*Z*)-1,5-octadien-3-one (no.**25**), methional (no.**36**) and *β*-damascenone (no.**69**). The thermal induced volatiles detected only by DHDA included methanethiol (no.**1**), 2-methylpropanal (no.**4**), 1-propanethiol (no.**5**), thiophene (no.**11**), 2-methyl-3-furanthiol (no.**19**), octanal (no.**25**), and (*E*,*Z*)-2,4-nonadien-1-ol (no.**64**).

The results of AEDA method indicated that 2-vinyl-4*H*-1,3-dithiin (no.**70**) had the highest log_3FD factor in all treatments. In neutral fraction, the following compounds contributed the highest log_3FD factor in H25-CP: methyl propyl disulfide (no. **21**), dipropyl disulfide (no.**30**), benzaldehyde (no.**47**), dipropyl trisulfide (no.**59**), 3-vinyl-4*H*-1,2-dithiin (no **61**), (*E*,*E*)-2,4-decadienal (no. **67**), *β*-damascenone (no.**69**), and 2-vinyl-4*H*-1,3-dithiin (no.**70**). Moreover, 3-ethyl-2,4-dimethylpyrazine (no.**33**), 2,3-diethyl-5-methylpyrazine (no.**42**), and *o*-aminoacetophenone (no.**80**) were the potent aroma-active compounds in H25-CP with the highest FD factor in basic fraction. Additionally, the quantitative data showed that dimethyl disulfide (no.**14**), diallyl disulfide (no.**41**), allyl methyl trisulfide (no.**50**), diallyl trisulfide (no.**66**), butanoic acid (no.**53**) and 3-methylbutanoic acid (no.**56**) were the potent odorant with the highest concentration in H25-CP. Furthermore, It was found that 4-hydroxy-2,5-

dimethyl-3-(2*H*)-furanone was formed during heating that perceived in higher concentration in the excessive heating of Thai chili paste (H50-CP).

Sensory evaluation revealed that the intensity of garlic note was the highest in UH-CP and the intensity of shallot note was similar in all samples. On the other hand, the intensities of dried shrimp, roasted chili, salt, and sweet aromatic increased when heating the sample but there were not significantly different of dried shrimp, roasted chili notes and sweet aromatic between H25-CP and H50-CP. Therefore, to further clarify that the predominant compounds in H25-CP are related to the characteristic overall aroma of Thai chili paste, it would be necessary to investigate in detail of selected standard flavor recombination experiments.

The total oil and the encapsulation efficiency of microencapsulated spray-dried CPO powders showed the highest contents when the mass ratio of MD to GA increased to 1:2. Among three different samples, the MD/GA (2:1) sample showed the higher surface oil content and lower encapsulation efficiency. This implied that adding MD to the wall composition resulted in the least capacity to encapsulate the CPO. The results of water activity at 25 °C of each sample were similar. Moreover, the study showed that the observed Tg of all samples (82.87-96.97 °C) were higher than storage condition.

The release of selected aroma-active compounds (butanoic acid, 3-methylbutanoic acid, diallyl disulfide, benzaldehyde, and phenol) of spray-dried CPO powders by using gum arabic and maltodextrin DE 18.5 as wall materials during purge time for 60 min at 30 °C was determined by DHA-GC/FID. The highest release rates of selected volatiles were observed during the first 5 min of purging period. Among the volatiles detected, benzaldehyde which partially water soluble compounds was released at the highest rate. Furthermore, there were no significant differences ($P>0.05$) in the release rates of benzaldehyde, 3-methylbutanoic acid and diallyl disulfide among the samples during purging.

On the basis of the content of emitted volatiles in the headspace of spray-dried CPO powders, the MD/GA (1:2) sample showed lower amounts of the released volatiles than other samples. Additionally, the MD/GA (2:1) sample provided higher release of butanoic acid, water soluble compound, during purging than the MD/GA ratios of 1:1 and 1:2 samples.

The amount of 17 selected volatile compounds retained in each samples was determined by using GC-MS during storage at 50 °C and 68% RH for 5 weeks. Among the observed volatiles, only hexanal increased during storage. Dimethyl disulfide, phenylacetaldehyde and benzaldehyde could not be retained in the capsules throughout storage time. These might be caused by the highly volatile and water soluble properties of these compounds.

The amount of aroma components retained in microcapsules varied depending upon the carrier solids used (maltodextrin and gum arabic). The sample containing higher proportions of gum arabic provided better flavor retention. The MD/GA (1:2) sample showed excellent flavor retention at the initial time of storage for diallyl trisulfide (222.31 ng/g), benzaldehyde (87 ng/g), 2-vinyl-4*H*-1,3-dithiin (68.4 ng/g), 1,3-dithiane (5.03 ng/g), allyl methyl disulfide (2.90 ng/g) and diallyl disulfide (32.85 ng/g). On the other hand, the MD/GA (2:1) sample presented poor flavor retention for 1,3-dithiane (1.92 ng/g), allyl methyl disulfide (2.23 ng/g), dimethyl trisulfide (10.52 ng/g), 3-vinyl-4*H*-1,2-dithiin (7.23 ng/g) and 2-vinyl-4*H*-1,3-dithiin (45.23 ng/g).

According to the percentage of flavor retentions of detected volatiles at the end of storage, the MD/GA (1:2) sample showed higher flavor retention in dimethyl trisulfide (43.08%), 3-methylbutanoic acid (73.19%), 1,3-dithiane (22.65%), 3-vinyl-4*H*-1,2-dithiin (31.40%) and 2-vinyl-4*H*-1,3-dithiin (17.06%) than other samples. On the contrary, the MD/GA (2:1) sample showed low flavor retention of 1,3-dithiane (14.17%), allyl methyl disulfide (23.90%), dimethyl trisulfide (15.52%), and 3-vinyl-4*H*-1,2-dithiin (17.54%).

On the basis of total content of flavor retention of selected volatiles at the initial and at the end of storage time, the MD/GA (1:2) sample showed higher contents (550.94 and 136.82 ng/g) than the 1:1 (423.92 and 109.07 ng/g) and 2:1 (415.19 and 130.50 ng/g) ratios of MD/GA samples.

The results from QDA showed that the panelists could not detect the variation in the intensities of chili note, salt aromatic, sweet aromatic, and overall aroma intensity among all samples after week 5 of storage at 50 °C and 68% RH. Moreover, there were slight changes in the shrimpy note and salt aromatic in the MD/GA (2:1) sample, garlic note and overall aroma intensity of a MD/GA (1:2) sample. Therefore, it can be concluded that spray-dried CPO powders could retain the most aroma intensities during storage time for 5 weeks.

Concerning further research, it can be suggested that more detailed studies with the glass transition temperature as a critical determinant in the encapsulated powder matrix going from a glassy amorphous state to a rubbery amorphous state as a powder storage at various water activities should be determined. A better understanding of the effect of storage relative humidity on the properties of capsule matrixes would be useful in the quality control and applications of these powders. Moreover, the oxidation of encapsulated flavor is also used as an index of the encapsulated flavor stability. Therefore, it is recommended that the further study on the effect of relative humidity on oxidation of microencapsulated CPO during storage should be also investigated.

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APPENDICES

Appendix A

Calculation of Retention Index

Retention index is a measure of relative retention using normal alkanes as a standard of reference. Retention index can be calculated from data obtained by programmed GC using the formula derived by Van den Dool and Kratz (1963).

$$RI = 100N + 100n \frac{t_{Ra} - t_{RN}}{t_{R(N+n)} - t_{RN}}$$

Where N = carbon number of the lower alkane

n = the difference in carbon number of the two n-alkanes that bracket the compound

t_{Ra} = the retention time of unknown (or test) compound

t_{RN} = the retention time of the lower alkane

$t_{R(N+n)}$ = the retention time of the upper alkane (Cadwallader, 2004).

Appendix Table A1 Semiquantitive concentrations of volatile compounds in Thai chili paste identified by dynamic headspace analysis/gas chromatography-mass spectrometry (DHA/GC-MS).

No.[a]	RI[b]		Compound	Concentration (ng/g)[c]		
	FFAP	RTX5		UH-CP	H25-CP	H50-CP
1	696	na[d]	methanethiol	212.00	792.00	908.00
2	760	<500	dimethyl sulfide	15.90	137.02	489.88
4	779	555	2-methylpropanal	397.00	1019.00	1327.00
5	na	610	1-propanethiol	2384.00	1058.00	262.00
6	906	604	allyl mercaptan	66.31	127.69	366.00
7/8	929	672	2/3-methylbutanal	196.18	2376.67	3643.39
9	946	704	allyl methyl sulfide	149.13	830.81	1197.06
14	1107	753	dimethyl disulfide	217.56	947.99	2123.79
15	1089	804	hexanal	268.50	134.85	320.46
17	1188	867	3,3'-thiobis-1-propene	170.94	621.20	898.05
18	1198	873	2,4-dimethylthiophene	40.41	44.55	51.73
21	1239	na	methyl propyl disulfide	87.45	156.58	220.92
22	1259	886	3,4-dimethylthiophene	77.82	154.54	298.10
23	1271	952	1,3-dithiane	2393.93	4337.38	4842.25
24	1288	929	allyl methyl disulfide	2759.00	6552.00	8182.00
29	1344	na	unknown	7.93	6.55	9.75
30	1383	1121	dipropyl disulfide	43.40	131.07	123.02
31	1389	988	dimethyl trisulfide	1406.20	4193.53	4061.90
37	1450	na	(*E*)-2-propenyl propyl disulfide	66.77	126.14	129.51
39	1461	na	unknown	13.32	43.11	61.35
41	1473	1096	diallyl disulfide	6673.59	11242.99	10873.56
47	1533	na	benzaldehyde	34.47	50.57	52.10
50	1606	1164	allyl methyl trisulfide	1706.00	1835.00	1604.00
61	1721	1219	3-vinyl-4*H*-1,2-dithiin	1108.00	1319.00	1461.00
63	1744	na	2-vinyl-1,3-dithiine	13.93	85.42	111.19
66	1805	1327	diallyl trisulfide	343.00	959.00	887.00
70	1847	1243	2-vinyl-4*H*-1,3-dithiin	259.00	226.00	223.00

Appendix Table A1 (Continued)

No.[a]	RI[b]		Compound	Concentration (ng/g)[c]		
	FFAP	RTX5		UH-CP	H25-CP	H50-CP
74	1974	na	phenol	18.95	24.45	20.91
86	1034	na	trichloromethane	38.68	41.77	57.53
87	1047	na	1-propanol	224.18	491.11	214.88
88	1102	na	(*E*)-2-methyl-2-butenal	2734.11	525.14	633.03
89	1115	na	2-methylene-4-pentenal	nd[e]	72.87	92.03
90	na	878	*p*-xylene	nd	nd	111.60
92	1166	na	2-ethyl-*trans*-2-butenal	646.87	553.10	643.01
98	1234	995	2-pentylfuran	nd	34.69	31.20
104	1453	648	acetic acid	55.40	70.47	54.81
105	1467	842	furfural	28.05	28.58	20.78
128	na	745	3-methyl-1-butanol	140.59	154.15	109.02
130	na	765	(*E*)-2-pentenal	138.25	31.98	nd
134	na	839	2-methyl-2-pentenal	3291.48	2671.82	2214.80
135	na	849	2-ethenyl-2-butenal	106.49	216.92	47.28
136	na	860	(*E*)-2-hexenal	145.74	27.78	14.56
137	na	895	1,1'-thiobis-propane	992.37	503.95	237.71
138	na	906	heptanal	233.11	286.87	223.51
140	na	1001	1,2-dithiolane	549.30	394.02	222.23
142	na	1007	octanal	251.30	742.58	422.90
143	na	1210	decanal	1410.69	1878.20	881.69
145	na	1362	decanoic acid	221.11	336.48	113.58
146	na	1264	nonanoic acid	240.01	428.07	133.86
147	<600	na	unknown	4288.44	6335.46	nd
148	<600	na	unknown	nd	1815.83	2185.75
150	993	na	unknown	60.69	91.85	175.26
153	1018	na	unknown	nd	26.62	33.58
155	1067	na	unknown	nd	34.97	37.81
156	1125	na	unknown	125.52	182.39	19.48
160	1192	na	unknown	24.10	66.25	58.85

Appendix Table A1 (Continued)

No.[a]	RI[b]		Compound	Concentration (ng/g)[c]		
	FFAP	RTX5		UH-CP	H25-CP	H50-CP
162	1206	na	unknown	76.86	73.39	63.63
163	1224	na	unknown	47.56	15.66	20.96
165	1246	na	unknown	17.25	26.25	15.35
167	1265	na	unknown	18.22	27.54	33.33
169	1327	na	unknown	23.19	33.48	10.98
171	1338	na	unknown	14.93	16.80	23.80
172	1360	na	unknown	17.44	10.92	8.11
174	1374	na	unknown	8.54	13.15	14.55
177	1419	na	unknown	9.87	45.16	46.89
178	1435	na	unknown	86.73	218.63	259.25
179	1539	na	unknown	698.31	1638.55	1455.69
181	1579	na	unknown	nd	18.49	20.53
184	1657	na	unknown	8.36	25.76	37.85
186	1784	na	unknown	90.92	27.03	30.36
189	1927	na	unknown	16.57	25.68	18.67
191	na	<500	unknown	1630.56	2432.48	1876.67
192	na	<500	unknown	4742.16	7876.98	5025.00
195	na	584	unknown	536.30	457.74	702.22
196	na	594	unknown	1236.86	487.50	nd
197	na	626	unknown	499.46	50.90	290.64
198	na	688	unknown	nd	489.95	76.27
199	na	717	unknown	nd	692.64	148.46
202	na	736	unknown	nd	22.60	18.46
203	na	740	unknown	49.32	65.07	8.61
204	na	773	unknown	98.59	98.91	112.95
206	na	784	unknown	329.53	24.97	21.76
208	na	792	unknown	86.81	226.94	154.89
212	na	815	unknown	nd	40.59	44.28
217	na	852	unknown	nd	33.64	33.39

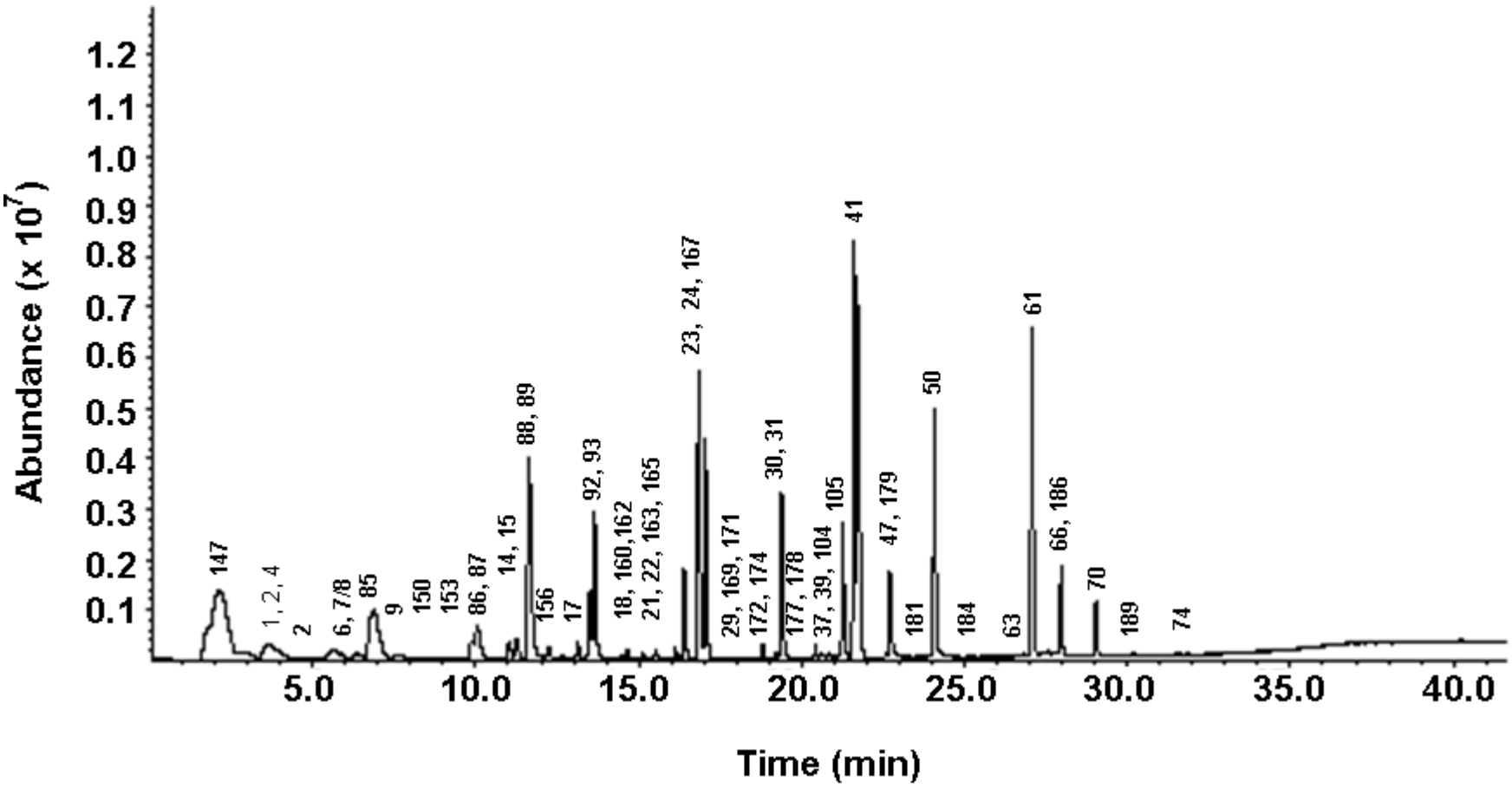
Appendix Table A1 (Continued)

No.[a]	RI[b]		Compound	Concentration (ng/g)[c]		
	FFAP	RTX5		UH-CP	H25-CP	H50-CP
219	na	862	unknown	25.89	56.93	44.22
221	na	880	unknown	7504.46	7148.07	422.93
223	na	892	unknown	114.56	566.18	157.48
226	na	900	unknown	127.80	157.79	119.97
227	na	903	unknown	163.28	383.83	203.92
228	na	922	unknown	nd	74.29	47.32
230	na	958	unknown	29.10	31.32	21.89
231	na	963	unknown	47.21	69.50	50.09
233	na	974	unknown	1887.78	5336.86	3279.71
235	na	982	unknown	43.80	158.80	71.04
238	na	1013	unknown	15.44	37.10	44.96
239	na	1018	unknown	12.23	12.38	15.31
240	na	1022	unknown	30.19	82.36	106.70
242	na	1031	unknown	88.94	77.71	128.36
243	na	1040	unknown	101.14	232.24	137.88
246	na	1065	unknown	39.08	58.85	66.99
247	na	1073	unknown	78.85	55.11	64.54
248	na	1077	unknown	53.53	61.55	23.10
249	na	1081	unknown	120.24	145.03	200.87
252	na	1124	unknown	nd	719.44	619.86
254	na	1130	unknown	140.17	378.82	243.06
255	na	1140	unknown	104.12	489.71	122.97
256	na	1149	unknown	17.78	37.09	32.73
260	na	1175	unknown	206.68	1001.15	833.40
263	na	1188	unknown	265.26	1655.50	1335.57
264	na	1195	unknown	97.57	52.10	52.79
265	na	1248	unknown	149.01	264.29	202.17
268	na	1284	unknown	43.22	50.48	29.76
269	na	1302	unknown	84.14	54.02	35.44

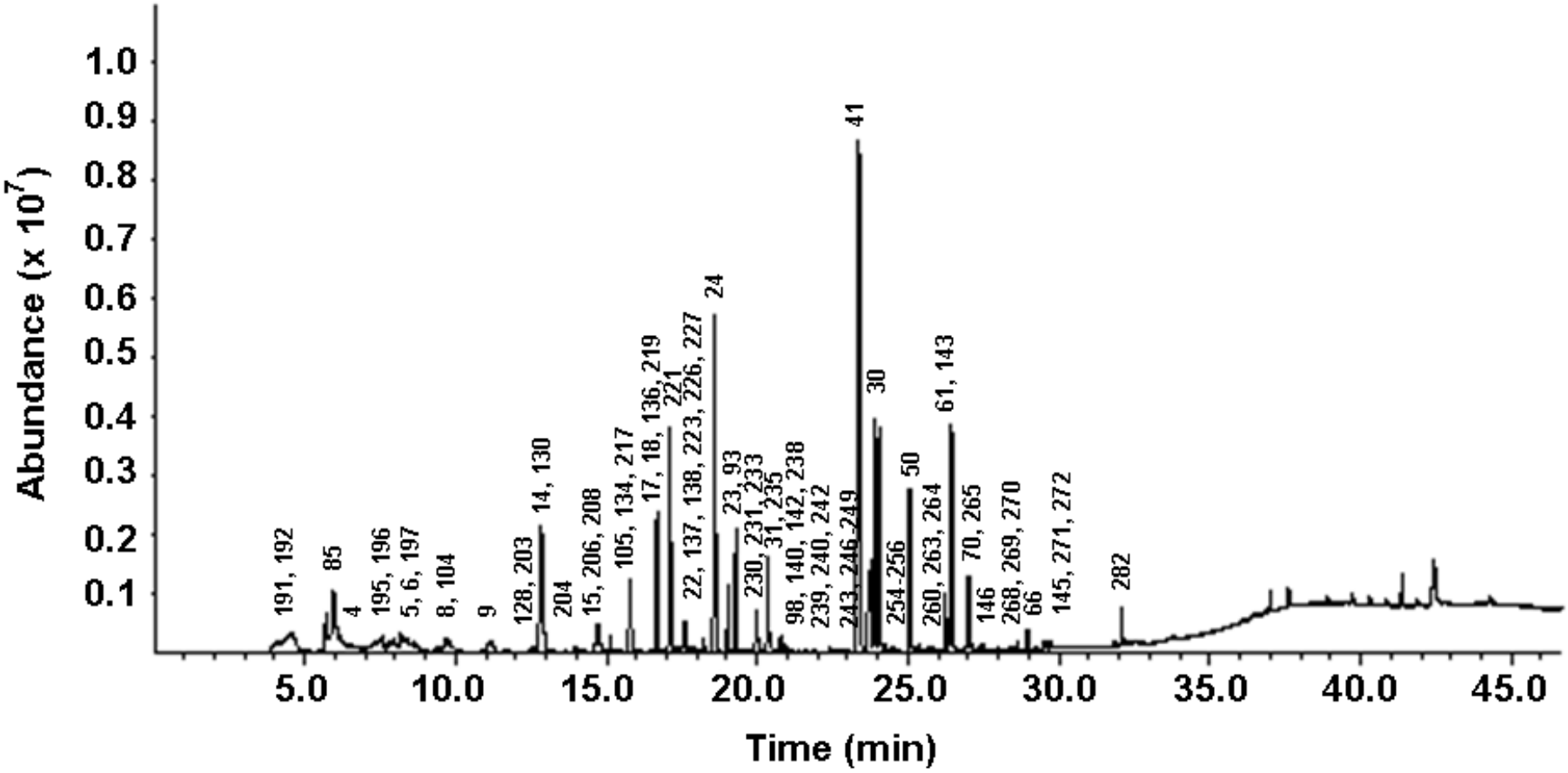
Appendix Table A1 (Continued)

No.[a]	RI[b]		Compound	Concentration (ng/g)[c]		
	FFAP	RTX5		UH-CP	H25-CP	H50-CP
270	na	1312	unknown	196.88	251.70	130.16
271	na	1340	unknown	52.78	280.56	268.14
272	na	1353	unknown	322.88	833.73	674.78
282	na	1473	unknown	1044.61	96.73	nd

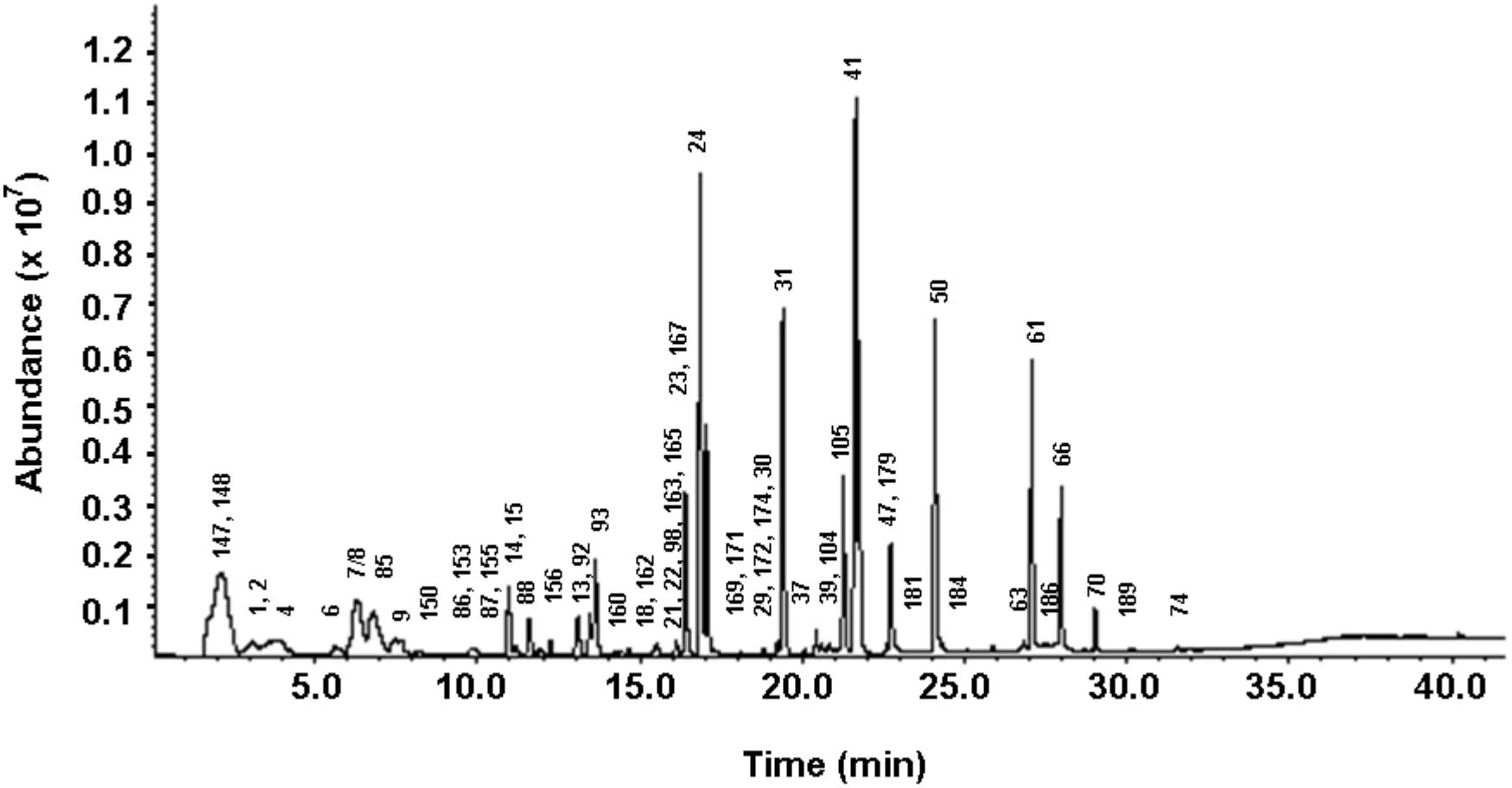
[a] Numbers correspond to those in Tables 7-9, and Appendix Table A2. [b] Retention indices (RI) calculated from DHA/GC-MS results on FFAP (Stabilwax®-DA) or RTX5 (RTX®-5SILMS) column. [c] Semiquantitive concentration = concentration of internal standard x peak area of compound/peak area of the internal standard on Stabilwax-DA column, except compounds no. 5, 90, 128, 130, 134-138, 140-142, 143, 145, 146, 191, 192, 195-199, 202-204, 206, 208, 212, 217, 219 221, 223, 226-228, 230, 233, 235, 238-240, 242, 243, 246-249, 252, 254-256, 260, 263, 265, 268-272 and 282; UH-CP = unheated CP, H25-CP = CP heated at 100°C for 25 min, and H50-CP = CP heated at 100 °C for 50 min. [d] na = not available. [e] nd = not detected.



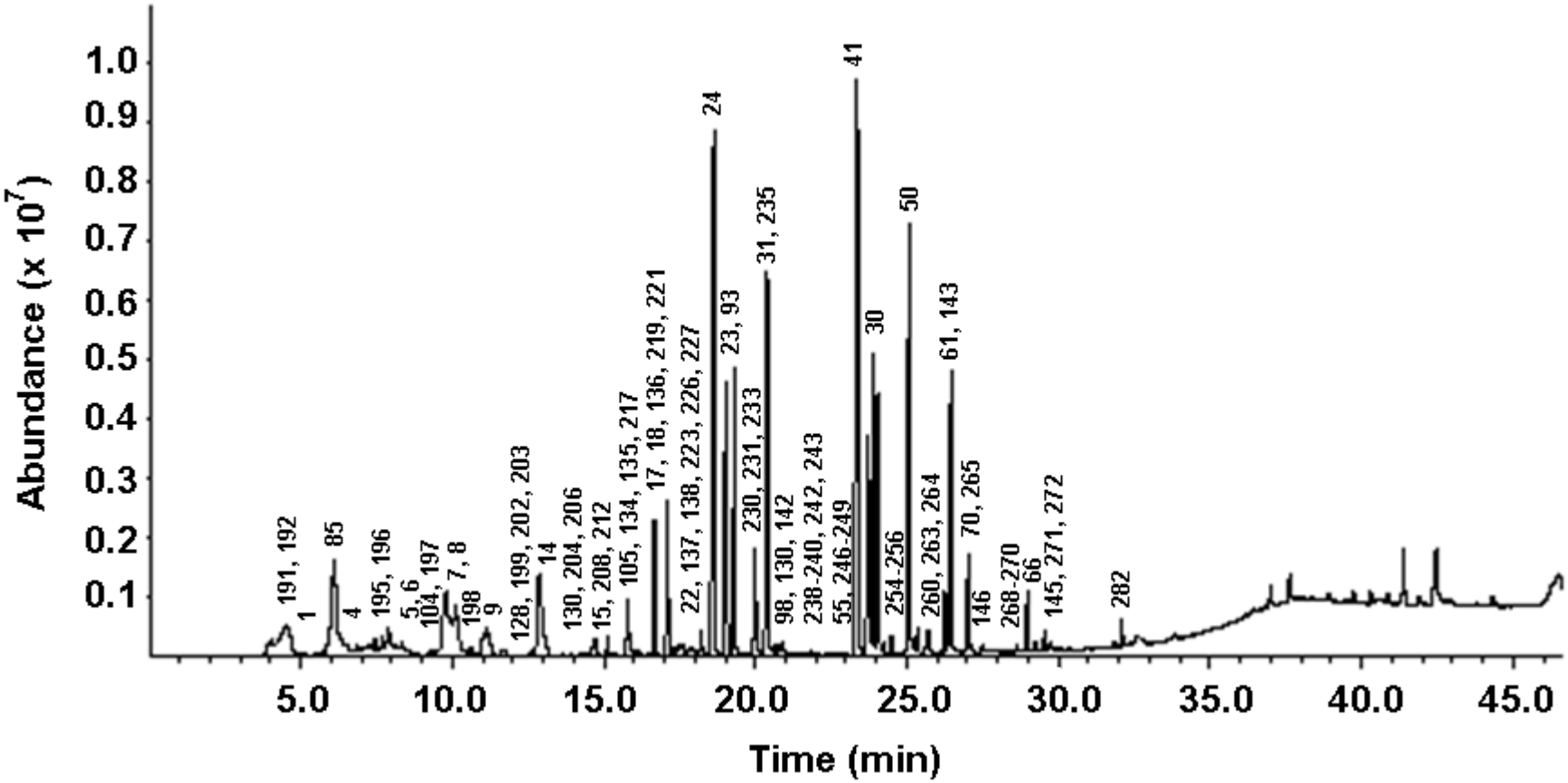
Appendix Figure A1 Total ion current gas chromatogram of an unheated CP sample determined by using DHA/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



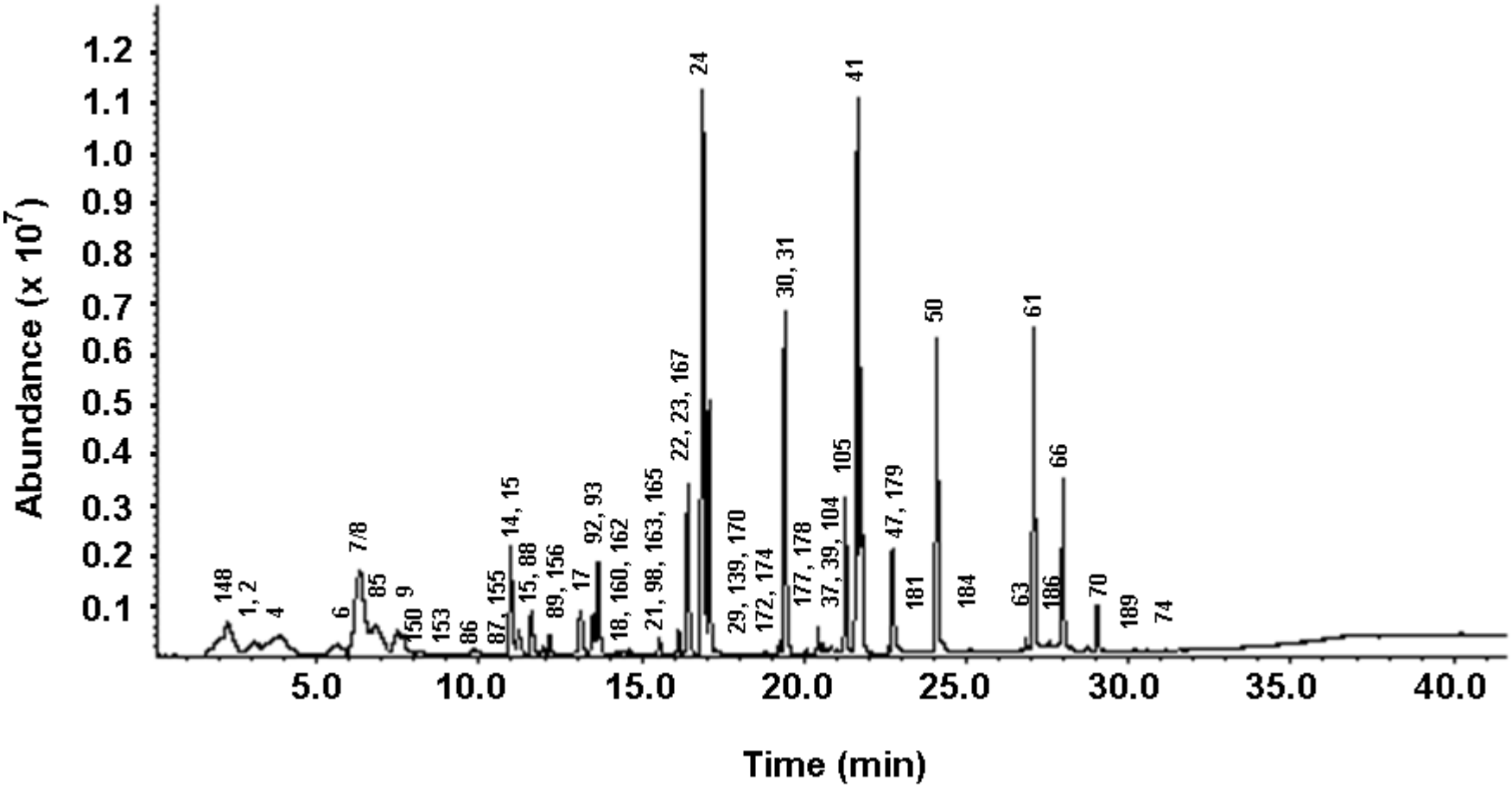
Appendix Figure A2 Total ion current gas chromatogram of an unheated CP sample determined by using DHA/GC-MS on RTX-5MS column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



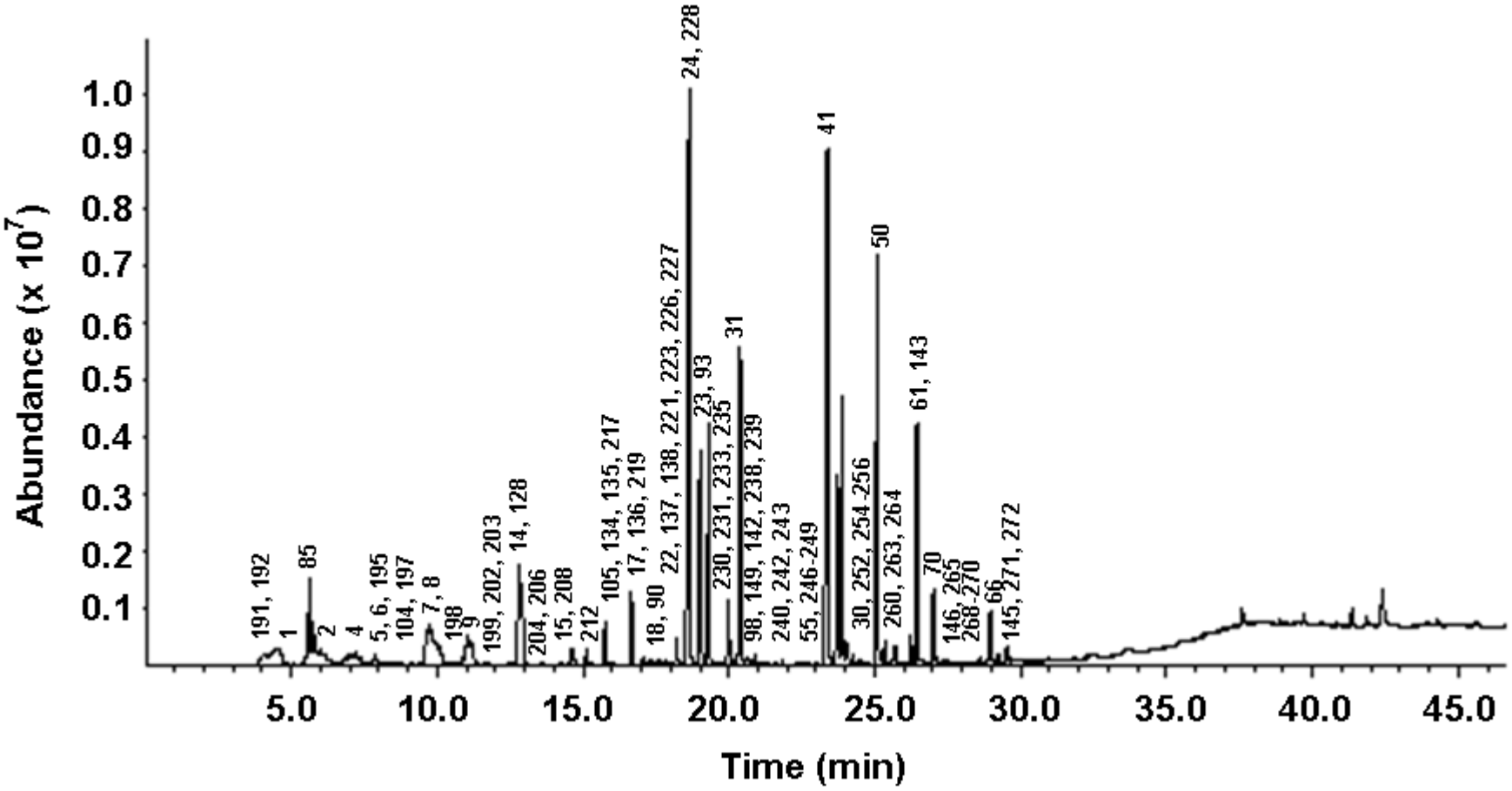
Appendix Figure A3 Total ion current gas chromatogram of a CP sample heated at 100°C for 25 min determined by using DHA/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



Appendix Figure A4 Total ion current gas chromatogram of a CP sample heated at 100°C for 25 min determined by using DHA/GC-MS on RTX-5MS column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



Appendix Figure A5 Total ion current gas chromatogram of a CP sample heated at 100°C for 50 min determined by using DHA/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9 and Appendix Tables A1and A2.



Appendix Figure A6 Total ion current gas chromatogram of a CP sample heated at 100°C for 50 min determined by using DHA/GC-MS on RTX-5MS column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.

Appendix Table A2 Semiquantitive concentrations (ng/g) of volatile compounds in Thai chili paste extracts identified by cool on-column gas chromatography-mass spectrometry (COC/GC-MS).

No.[a]	RI[b]		Compound	Fraction[c]	Concentration (ng/g)[c]		
	FFAP	DB5			UH-CP	H25-CP	H50-CP
7	931	<700	2-methylbutanal	NF	0.14	2.66	0.13
8	940	<700	3-methylbutanal	NF	0.30	6.78	0.29
9	974	<700	allyl methyl sulfide	NF	0.40	2.19	0.39
10	982	na[e]	2,3-butanoic acid	AF	nd[f]	nd	0.82
14	1090	757	dimethyl disulfide	NF	7.10	23.30	21.88
15	1099	802	hexanal	NF	1.55	1.19	1.50
17	1165	872	3,3'-thiobis-1-propene	NF	nd	2.57	nd
21	1250	na	methyl propyl disulfide	NF	1.68	1.48	1.64
22	1270	890	3,4-dimethylthiophene	NF	nd	1.38	nd
23	1283	924	1,3-dithiane	NF	65.01	45.78	62.52
24	1298	937	allyl methyl disulfide	NF	80.62	63.55	78.19
30	1389	1121	dipropyl disulfide	NF	0.87	1.92	0.90
31	1394	970	dimethyl trisulfide	NF	2.77	168.50	2.55
33	1450	1083	3-ethyl-2,5-dimethylpyrazine	BF	0.52	1.18	0.62
36	1456	910	methional	NF	1.22	2.24	1.05
41	1498	1094	diallyl disulfide	NF	190.75	265.74	116.91
47	1537	990	benzaldehyde	NF	0.81	1.02	0.80
53	1630	na	butanoic acid	AF	17.55	68.66	26.39
55	1659	1058	phenylacetaldehyde	NF	4.23	4.44	2.29
56	1671	na	3-methylbutanoic acid	AF	71.98	150.84	86.15
59	1683	1356	dipropyl trisulfide	NF	0.30	0.35	0.27
61	1760	1213	3-vinyl-4*H*-1,2-dithiin	NF	186.18	179.52	185.86
63	1783	1202	2-vinyl-1,3-dithiane	NF	2.73	11.31	2.75
66	1813	1324	diallyl trisulfide	NF	5.44	275.75	4.87
70	1881	1253	2-vinyl-4*H*-1,3-dithiin	NF	210.05	137.97	211.39
71	1850	na	hexanoic acid	AF	4.61	4.16	5.31
74	2013	na	phenol	NF	21.60	1.02	1.58

Appendix Table A2 (Continued)

No.[a]	RI[b]		Compound	Fraction[c]	Concentration (ng/g)[c]		
	FFAP	DB5			UH-CP	H25-CP	H50-CP
77	2035	na	4-hydroxy-2,5-dimethyl-3-(2H)-furanone (HDMF)	AF	0.62	1.86	1.91
82	2568	na	phenylacetic acid	AF	3.66	4.03	2.96
84	910	<700	ethyl acetate	NF	1.56	4.55	1.52
85	959	na	ethyl alcohol	NF	11.10	0.30	10.45
86	1042	<700	trichloromethane	NF	2.80	1.41	2.69
87	1063	na	1-propanol	NF	4.63	2.41	4.19
90	1158	879	*p*-xylene	NF	nd	0.27	nd
91	1158	na	1-butanol	NF	6.38	2.31	6.27
94	1176	na	2-ethyl-*trans*-2-butenal	NF	6.46	6.38	6.46
96	1213	1034	D-limonene	NF	0.15	0.66	0.13
97	1222	na	3-methyl-1-butanol	NF	nd	1.46	2.19
98	na	995	2-pentylfuran	NF	1.20	0.92	2.21
99	1281	na	methylpyrazine	BF	1.07	0.88	2.70
100	1333	915	2,5-dimethylpyrazine	BF	0.75	0.78	2.07
101	1339	916	2,6-dimethylpyrazine	BF	2.82	3.05	8.38
103	1409	1006	trimethylpyrazine	BF	1.03	1.15	2.84
104	1459	na	acetic acid	AF	7.28	159.39	8.36
105	na	849	furfural	NF	1.61	1.52	2.09
106	1479	1091	tetramethylpyrazine	BF	4.12	2.42	5.77
107	1544	na	propanoic acid	AF	3.71	34.46	6.05
108	1572	na	2-methylpropanoic acid	AF	11.12	42.92	16.60
109	1663	na	2-furanmethanol	NF	1.08	5.92	1.34
110	1741	na	pentanoic acid	AF	1.58	1.97	1.82
112	1807	na	4-methylpentanoic acid	AF	5.16	4.09	4.63
113	1922	1526	butylated hydroxytoluene (BHT)	NF	33.82	19.98	34.27
114	1952	na	2-ethylhexanoic acid	AF	9.10	17.06	26.62
115	1979	na	2-acetylpyrrole	BF	1.13	1.08	3.54

Appendix Table A2 (Continued)

No.[a]	RI[b]		Compound	Fraction[c]	Concentration (ng/g)[c]		
	FFAP	DB5			UH-CP	H25-CP	H50-CP
116	2063	na	octanoic acid	AF	1.05	nd	1.05
117	2157	na	sorbic acid	AF	118.27	122.54	126.82
118	2451	na	benzenecarboxylic acid	AF	1.01	1.32	0.99
119	2505	na	5-(hydroxymethyl)-2-furancarboxaldehyde	AF	2.16	nd	0.53
120	na	<700	3-methylpentane	NF	18.44	15.30	14.17
121	na	<700	2-methyl-1-pentene	NF	4.57	3.07	2.52
122	na	<700	3-methylene-pentane	NF	0.27	0.19	0.17
123	na	<700	(*E*)-4-methyl-2-pentene	NF	0.46	0.34	0.32
124	na	<700	(*Z*)-3-methyl-2-pentene	NF	0.18	0.13	0.12
125	na	<700	tetrahydrofuran	NF	0.04	2.72	4.18
126	na	<700	1,2-dichloroethane	NF	0.41	0.15	nd
127	na	715	2-ethylfuran	NF	0.02	0.58	0.79
129	na	763	3,5-dimethyl-2-hexene	NF	0.31	0.26	1.26
131	na	768	toluene	NF	0.48	0.26	0.15
132	na	816	1,1-diethoxy-propane	NF	0.60	0.31	0.18
133	na	836	2-methyl-2-pentenal	NF	8.51	8.18	7.31
139	na	941	1,4-dithiane	NF	12.47	12.47	31.20
141	na	1003	2-ethyl-5-methylpyrazine	BF	1.28	16.34	5.12
144	na	1258	dimethyl tetrasulfide	NF	nd	8.99	7.89
149	985	na	unknown	NF	0.64	0.62	0.64
151	1005	na	unknown	NF	0.20	0.26	0.19
152	1014	na	unknown	NF	0.27	0.19	0.27
154	1030	na	unknown	BF	0.81	3.93	8.01
157	1129	na	unknown	NF	0.98	0.23	0.86
158	1139	na	unknown	NF	0.37	0.47	0.37
159	1146	na	unknown	NF	0.13	1.43	0.13
161	1203	na	unknown	NF	0.08	0.48	0.09
164	1235	na	unknown	NF	0.27	0.14	0.26
166	1260	na	unknown	NF	0.34	0.14	0.33

Appendix Table A2 (Continued)

No.[a]	RI[b]		Compound	Fraction[c]	Concentration (ng/g)[c]		
	FFAP	DB5			UH-CP	H25-CP	H50-CP
168	1304	na	unknown	BF	0.98	0.78	1.52
170	1330	na	unknown	NF	0.26	0.19	0.27
173	1367	na	unknown	BF	0.16	0.19	0.35
175	1391	na	unknown	BF	0.24	0.37	1.41
176	1397	na	unknown	BF	0.40	0.43	0.84
180	1541	na	unknown	NF	3.59	8.96	3.42
182	1611	na	unknown	NF	0.85	46.77	1.01
183	1633	na	unknown	NF	0.67	0.92	nd
185	1674	na	unknown	NF	0.70	3.61	0.60
187	1797	na	unknown	BF	1.21	0.50	2.02
188	1868	na	unknown	AF	1.16	4.81	1.84
190	2098	na	unknown	BF	nd	1.15	2.80
193	na	<700	unknown	BF	3.32	55.63	23.49
194	na	<700	unknown	NF	1.00	0.22	0.26
200	na	728	unknown	NF	0.29	0.03	0.16
201	na	732	unknown	BF	1.45	11.36	4.18
205	na	782	unknown	NF	nd	0.05	0.10
207	na	790	unknown	NF	nd	0.39	0.12
209	na	793	unknown	NF	nd	0.24	0.02
210	na	810	unknown	BF	0.86	4.75	1.51
211	na	813	unknown	NF	0.23	0.25	nd
213	na	817	unknown	NF	0.12	0.20	0.35
214	na	819	unknown	BF	0.62	5.07	9.23
215	na	822	unknown	BF	0.28	4.64	2.10
216	na	832	unknown	NF	0.24	0.07	0.25
218	na	858	unknown	NF	0.32	0.94	0.24
219	na	862	unknown	NF	0.22	3.28	0.11
220	na	875	unknown	NF	0.38	0.31	1.96
222	na	884	unknown	BF	3.40	16.28	6.32
224	na	893	unknown	NF	0.22	0.43	0.57

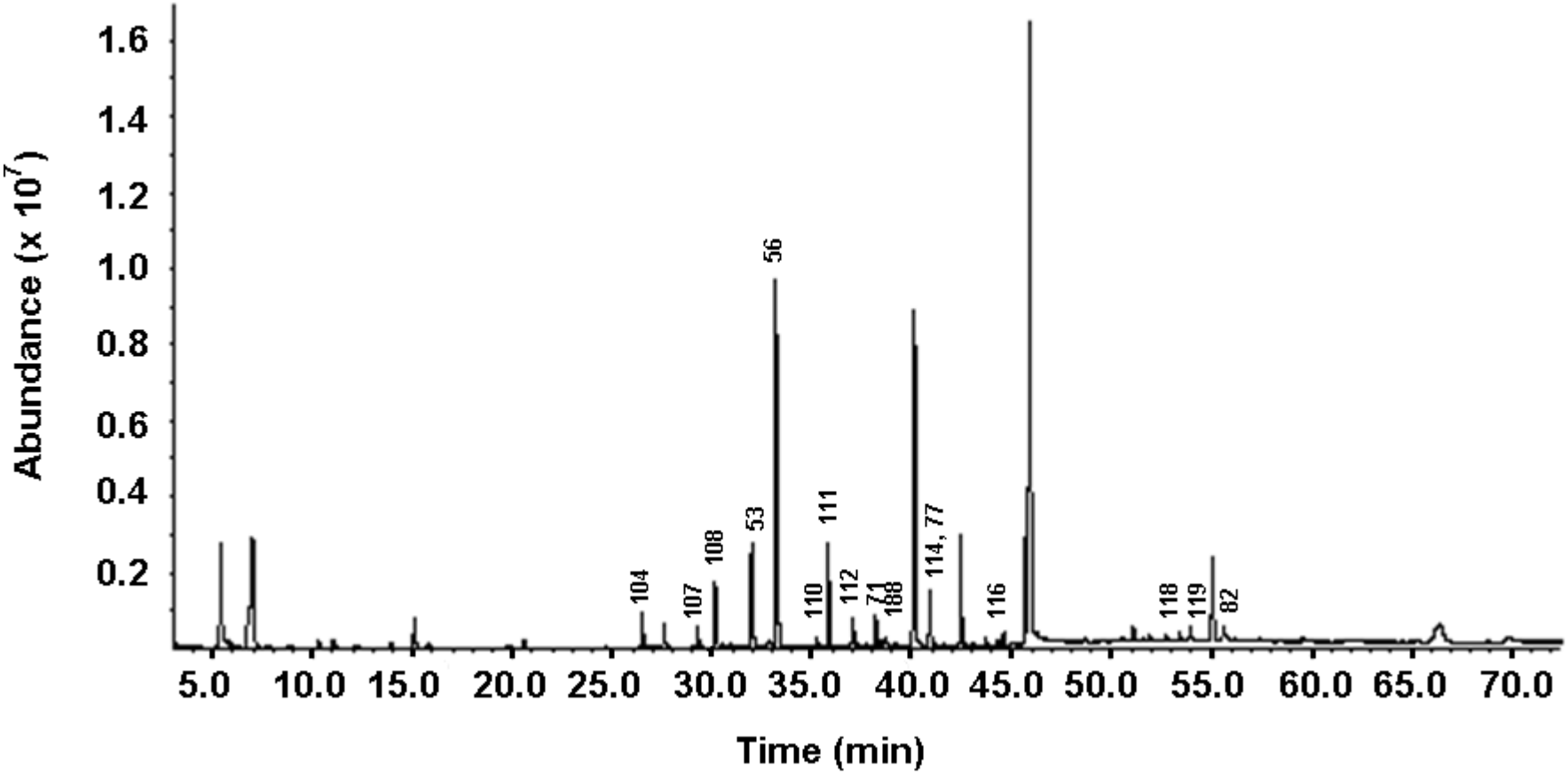
Appendix Table A2 (Continued)

No.[a]	RI[b]		Compound	Fraction[c]	Concentration (ng/g)[c]		
	FFAP	DB5			UH-CP	H25-CP	H50-CP
225	na	896	unknown	NF	0.12	0.07	0.29
227	na	905	unknown	NF	0.24	0.26	0.38
229	na	948	unknown	NF	43.63	30.94	nd
232	na	965	unknown	NF	2.28	5.44	0.59
234	na	980	unknown	NF	3.61	65.36	5.15
236	na	991	unknown	BF	0.77	7.17	0.92
237	na	1008	unknown	NF	0.65	0.21	nd
238	na	1013	unknown	NF	0.22	0.45	0.44
241	na	1025	unknown	NF	nd	0.07	0.12
244	na	1052	unknown	NF	0.63	0.44	nd
245	na	1065	unknown	NF	0.30	0.44	nd
250	na	1099	unknown	BF	1.85	5.00	nd
251	na	1107	unknown	BF	4.86	7.49	1.37
253	na	1127	unknown	NF	3.19	3.87	1.09
256	na	1149	unknown	BF	2.07	9.48	3.95
257	na	1161	unknown	NF	nd	304.64	0.59
258	na	1166	unknown	BF	0.99	5.03	nd
259	na	1167	unknown	NF	0.58	14.25	0.28
261	na	1179	unknown	NF	1.86	19.16	293.39
262	na	1181	unknown	NF	nd	24.60	3.13
266	na	1273	unknown	NF	nd	0.22	0.34
267	na	1281	unknown	NF	nd	0.88	1.02
272	na	1353	unknown	NF	1.81	78.72	0.09
273	na	1358	unknown	NF	3.33	0.43	67.35
274	na	1360	unknown	NF	1.15	3.03	0.34
275	na	1365	unknown	NF	1.51	1.07	3.43
276	na	1377	unknown	NF	0.87	0.12	2.25
277	na	1395	unknown	NF	0.27	5.31	0.93
278	na	1399	unknown	NF	0.25	0.35	nd
279	na	1406	unknown	NF	1.04	11.09	0.17

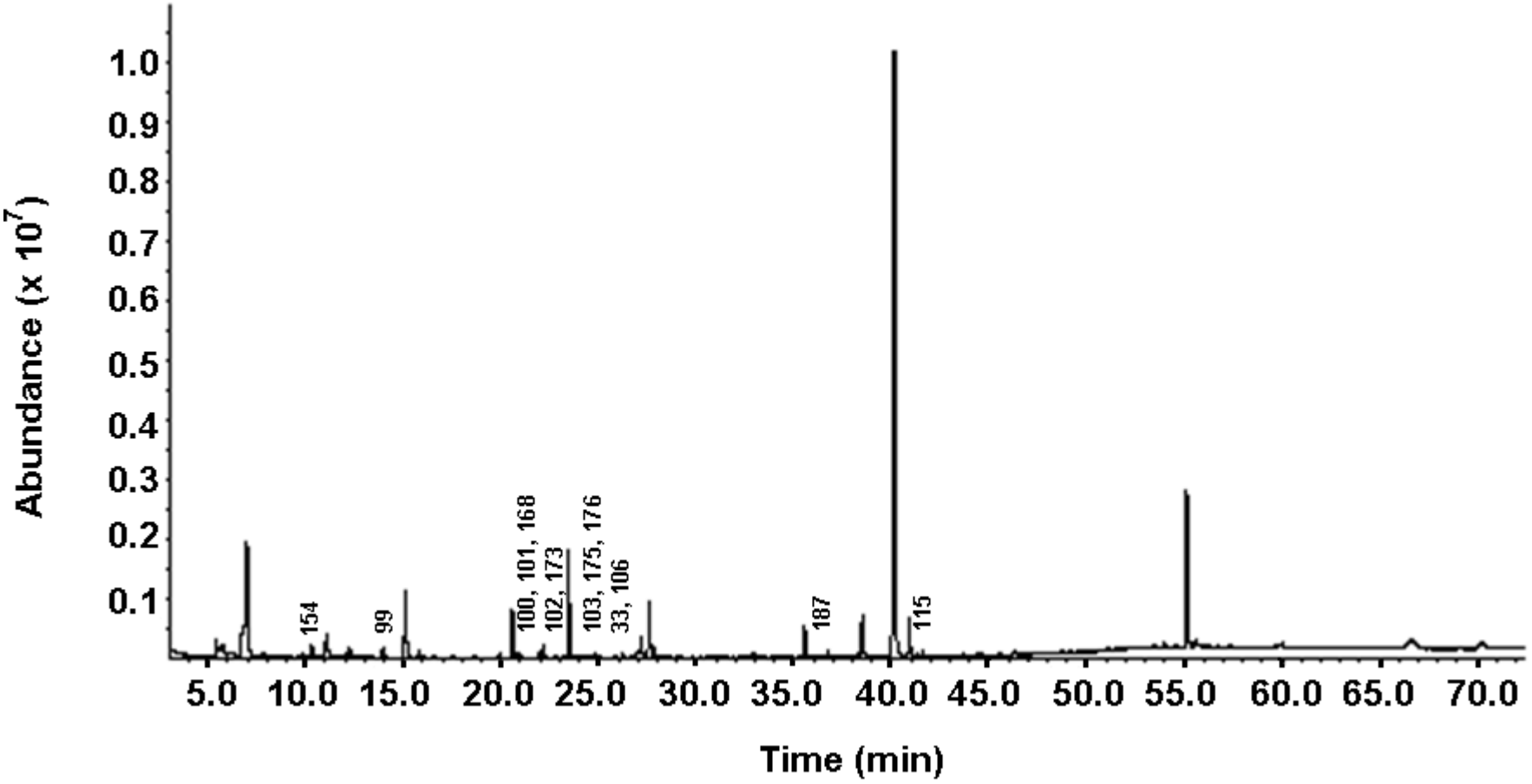
Appendix Table A2 (Continued)

No.[a]	RI[b]		Compound	Fraction[c]	Concentration (ng/g)[c]		
	FFAP	DB5			UH-CP	H25-CP	H50-CP
280	na	1417	unknown	NF	0.47	0.19	10.98
281	na	1423	unknown	NF	nd	0.38	9.89
283	na	1502	unknown	NF	1.49	1.18	0.60
284	na	1505	unknown	NF	nd	0.19	0.16

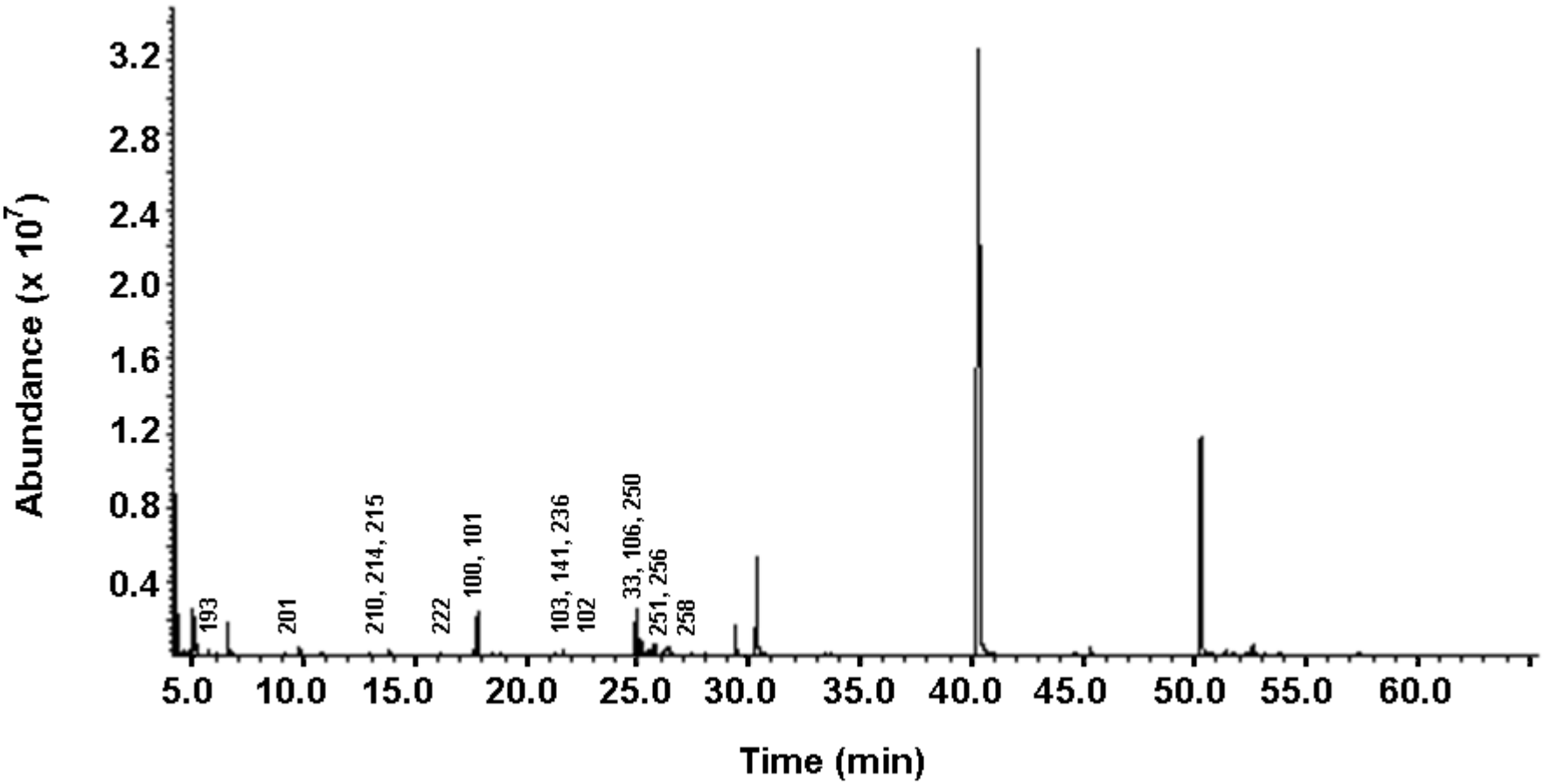
[a] Numbers correspond to those in Tables 7-9, and Appendix Table A1. [b] Retention indices (RI) calculated from COC/GC-MS results on FFAP (Stabilwax®-DA) or DB-5MS column. [c] Fraction containing odorant; AF = acidic fraction, BF = basic fraction and NF = neutral fraction. [d] Semiquantitive concentration = concentration of internal standard x peak area of compound/peak area of the internal standard on Stabilwax-DA column, except compounds no. 98, 105, 120-127, 129, 131-133, 139, 141, 144, 193, 194, 200, 201, 205, 207, 209-211, 213-216, 218-220, 222, 224, 225, 227, 229, 232, 236-238, 241, 244, 245, 250, 251, 253, 256-259, 261, 262, 266, 267, 272-281, 283, and 284; UH-CP = unheated CP, H25-CP = CP heated at 100°C for 25 min, and H50-CP = CP heated at 100 °C for 50 min. [e] na = not available. [f] nd = not detected.



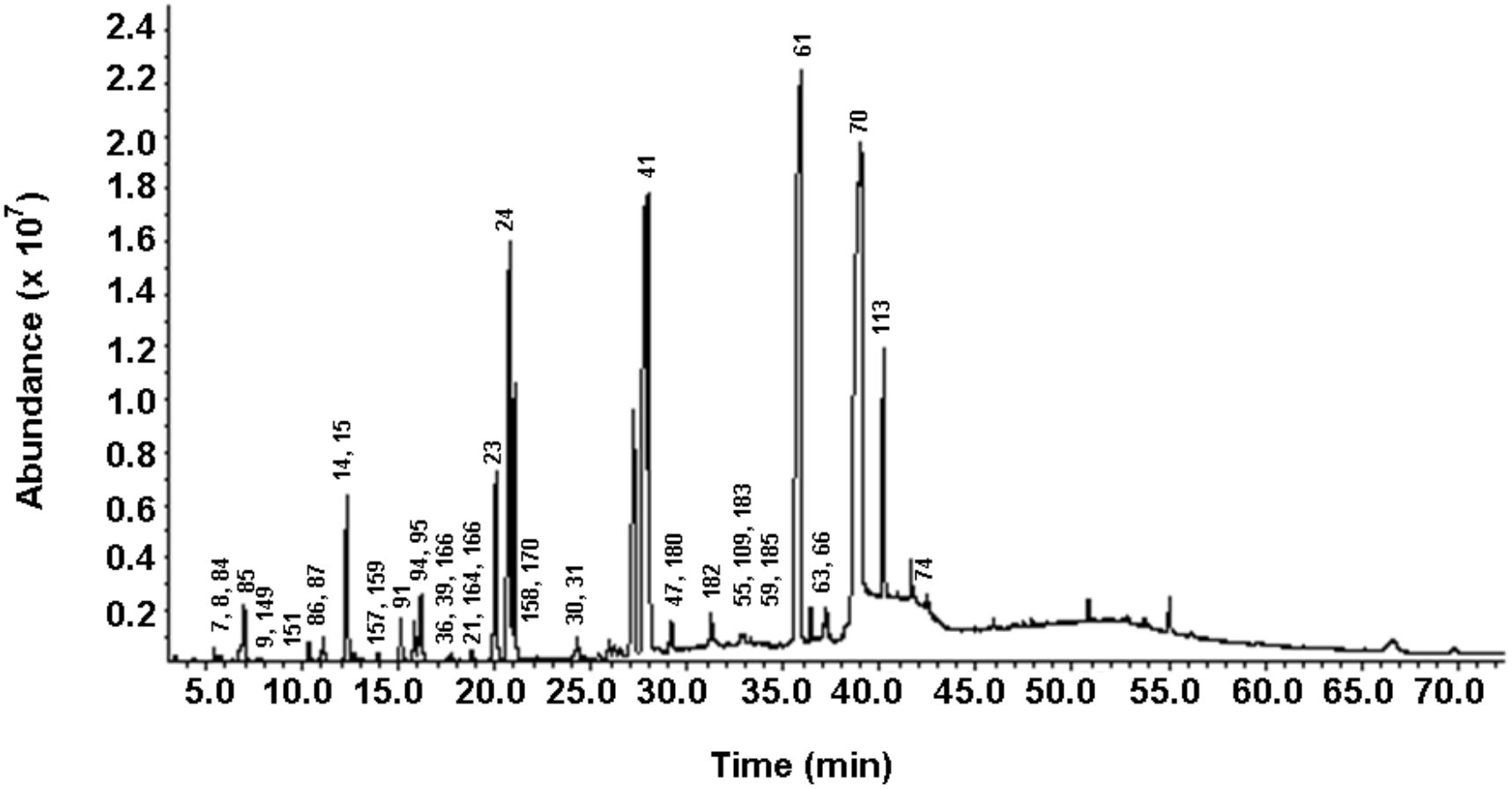
Appendix Figure A7 Total ion current gas chromatogram of an acidic fraction of unheated CP extract by using COC/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



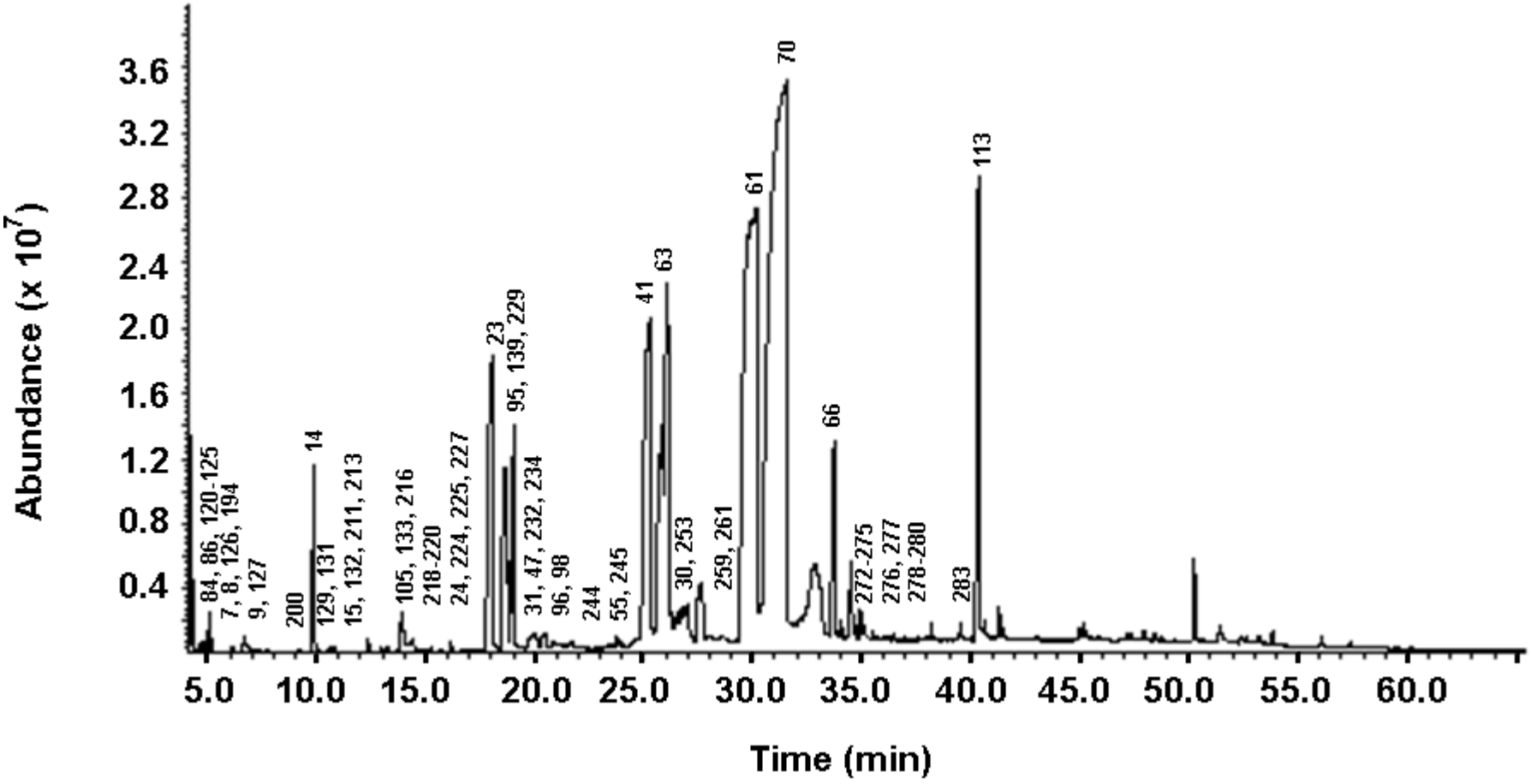
Appendix Figure A8 Total ion current gas chromatogram of a basic fraction of unheated CP extract by using COC/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



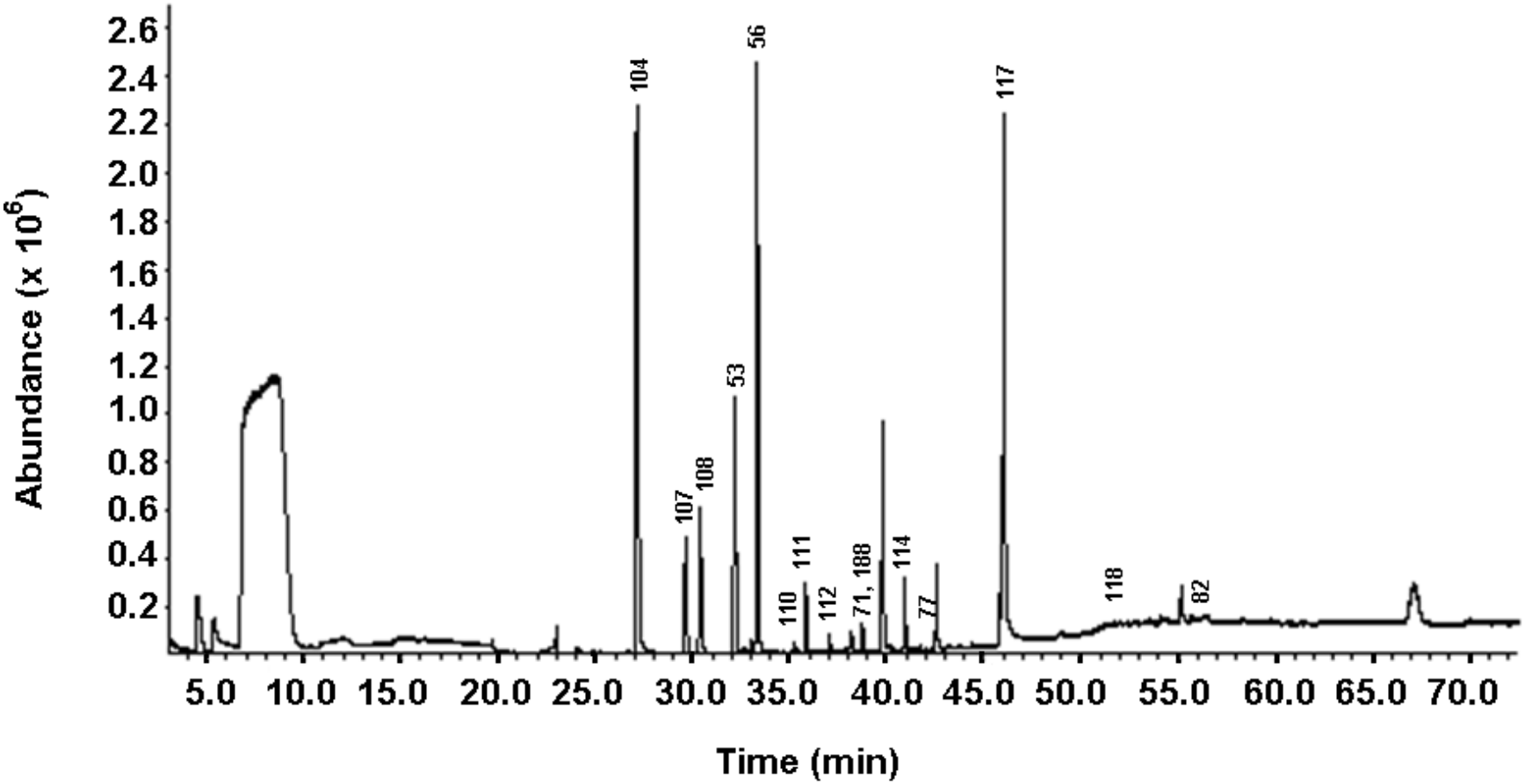
Appendix Figure A9 Total ion current gas chromatogram of a basic fraction of unheated CP extract by using COC/GC-MS on DB-5MS column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



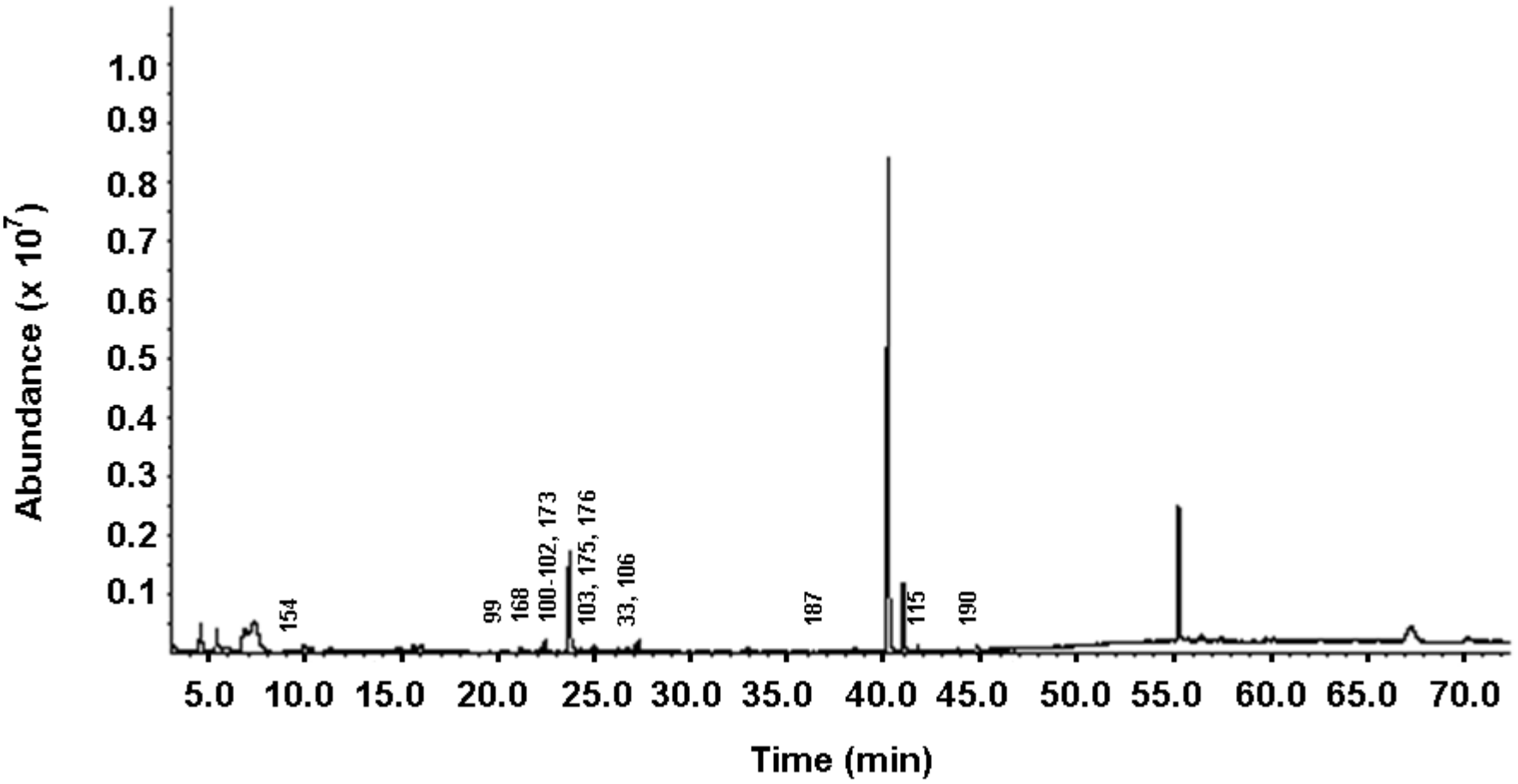
Appendix Figure A10 Total ion current gas chromatogram of a neutral fraction of unheated CP extract by using COC/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



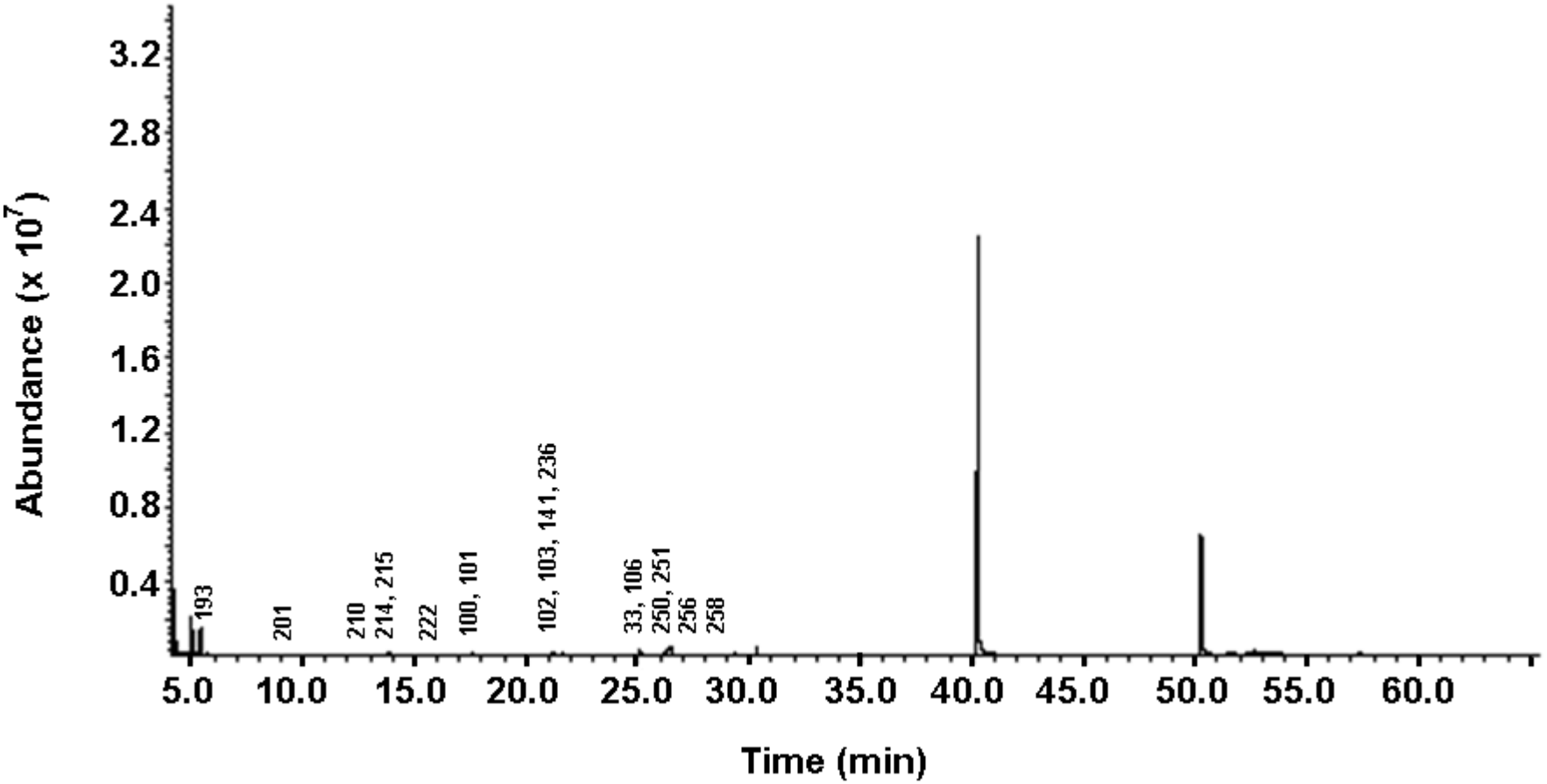
Appendix Figure A11 Total ion current gas chromatogram of a neutral fraction of unheated CP extract by using COC/GC-MS on DB-5MS column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



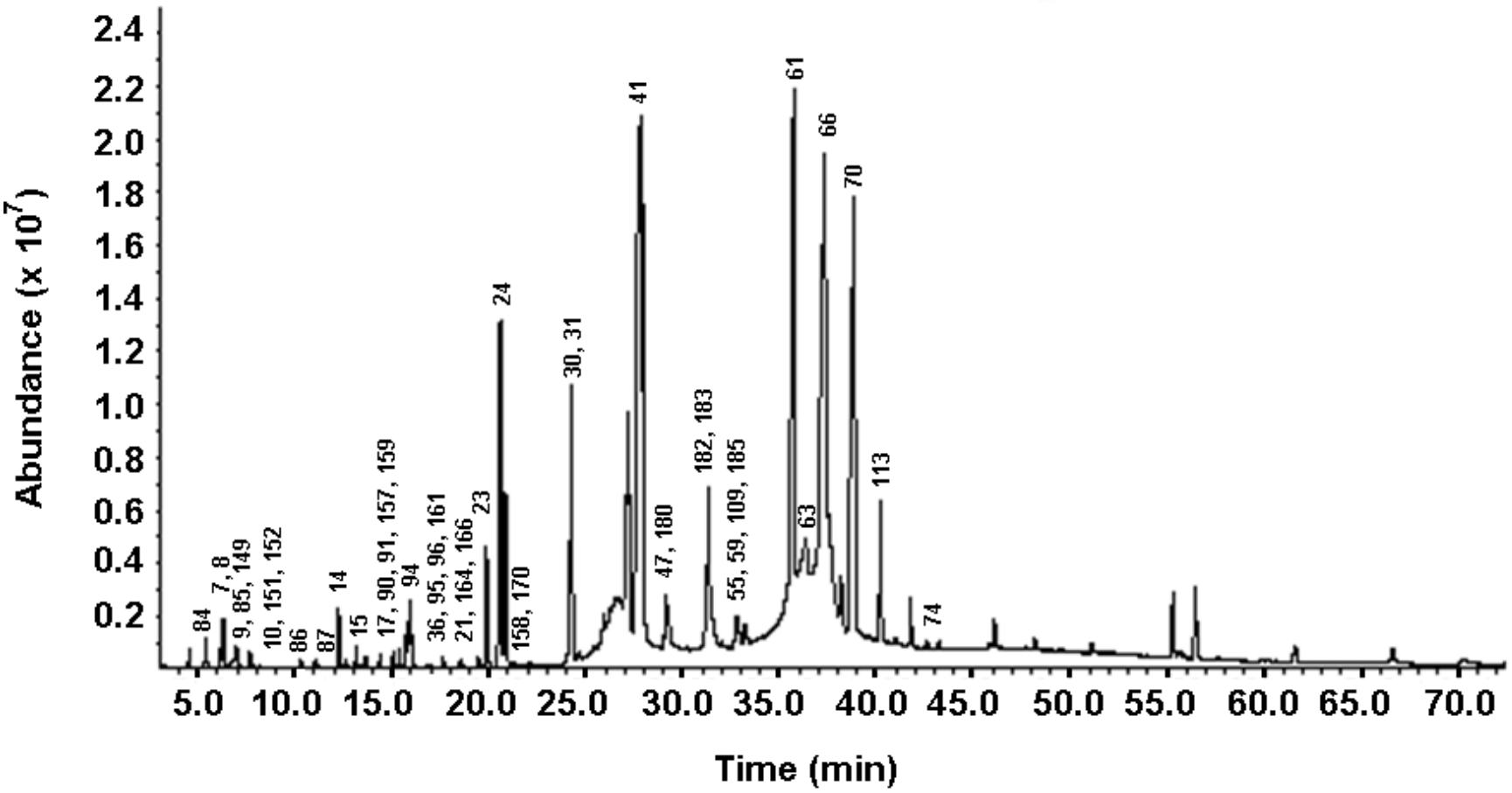
Appendix Figure A12 Total ion current gas chromatogram of an acidic fraction of the extract from CP sample heated at 100°C for 25 min by using COC/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



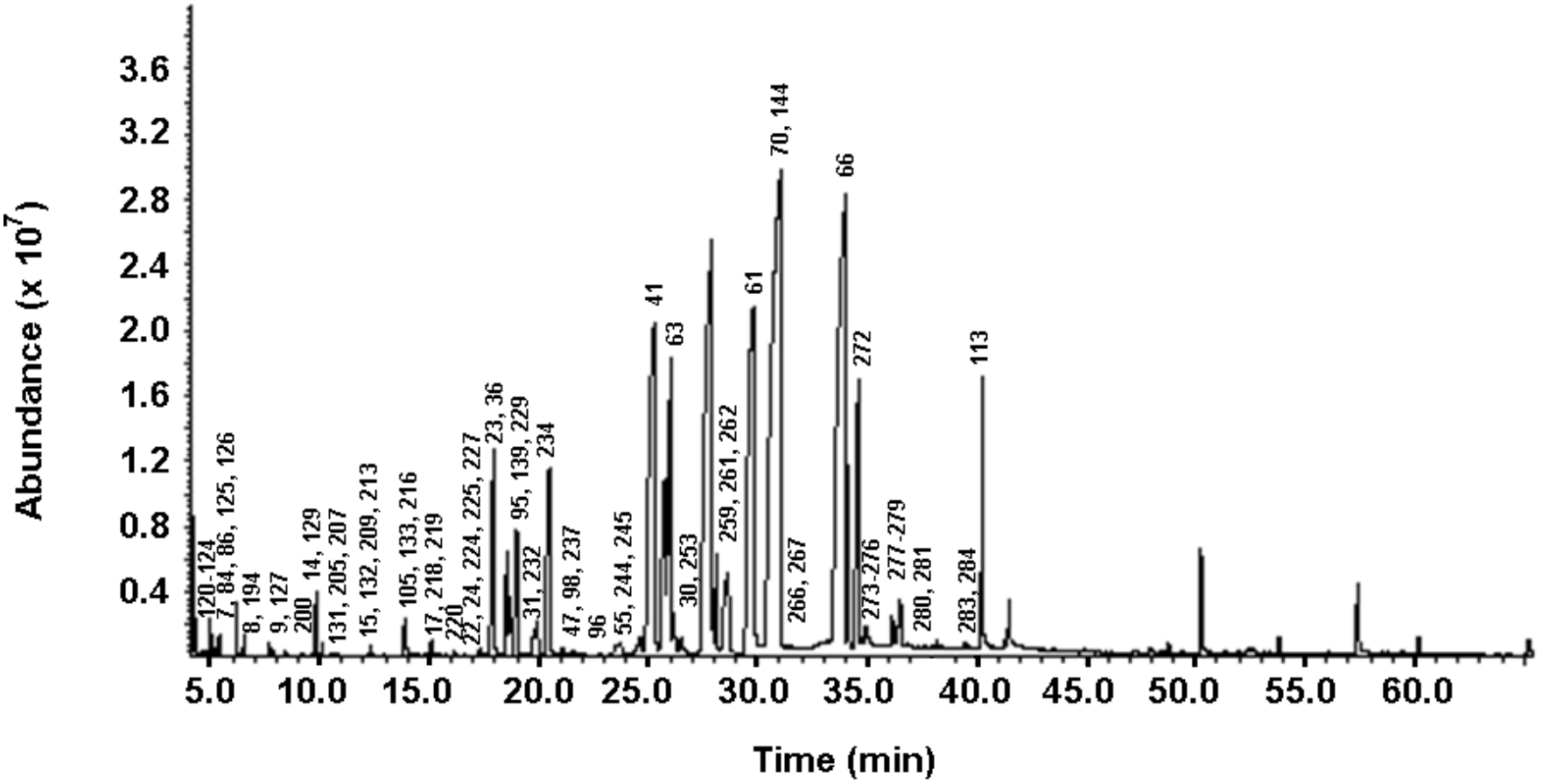
Appendix Figure A13 Total ion current gas chromatogram of a basic fraction of the extract from CP sample heated at 100°C for 25 min by using COC/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



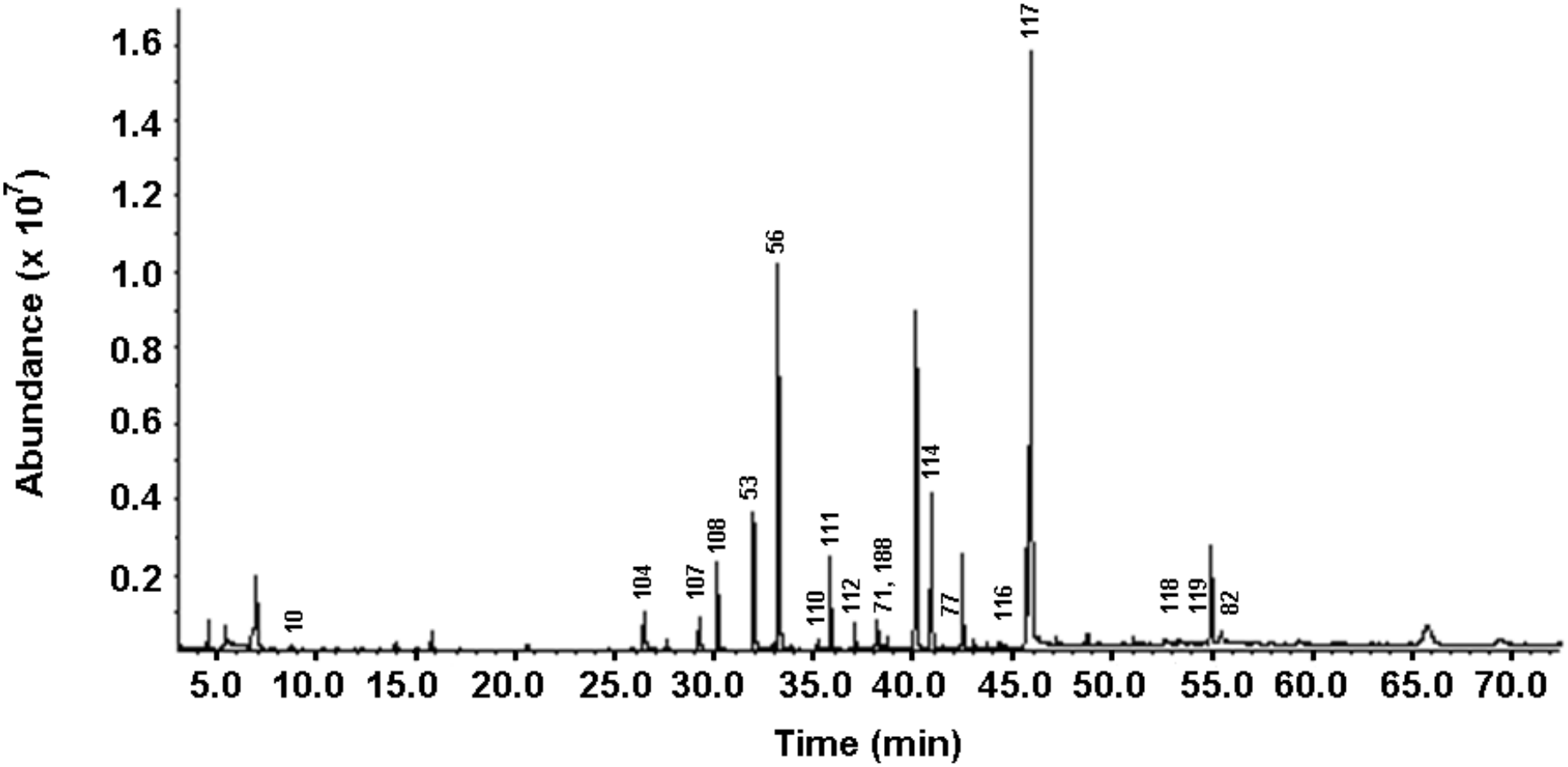
Appendix Figure A14 Total ion current gas chromatogram of a basic fraction of the extract from CP sample heated at 100°C for 25 min by using COC/GC-MS on DB-5MS column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



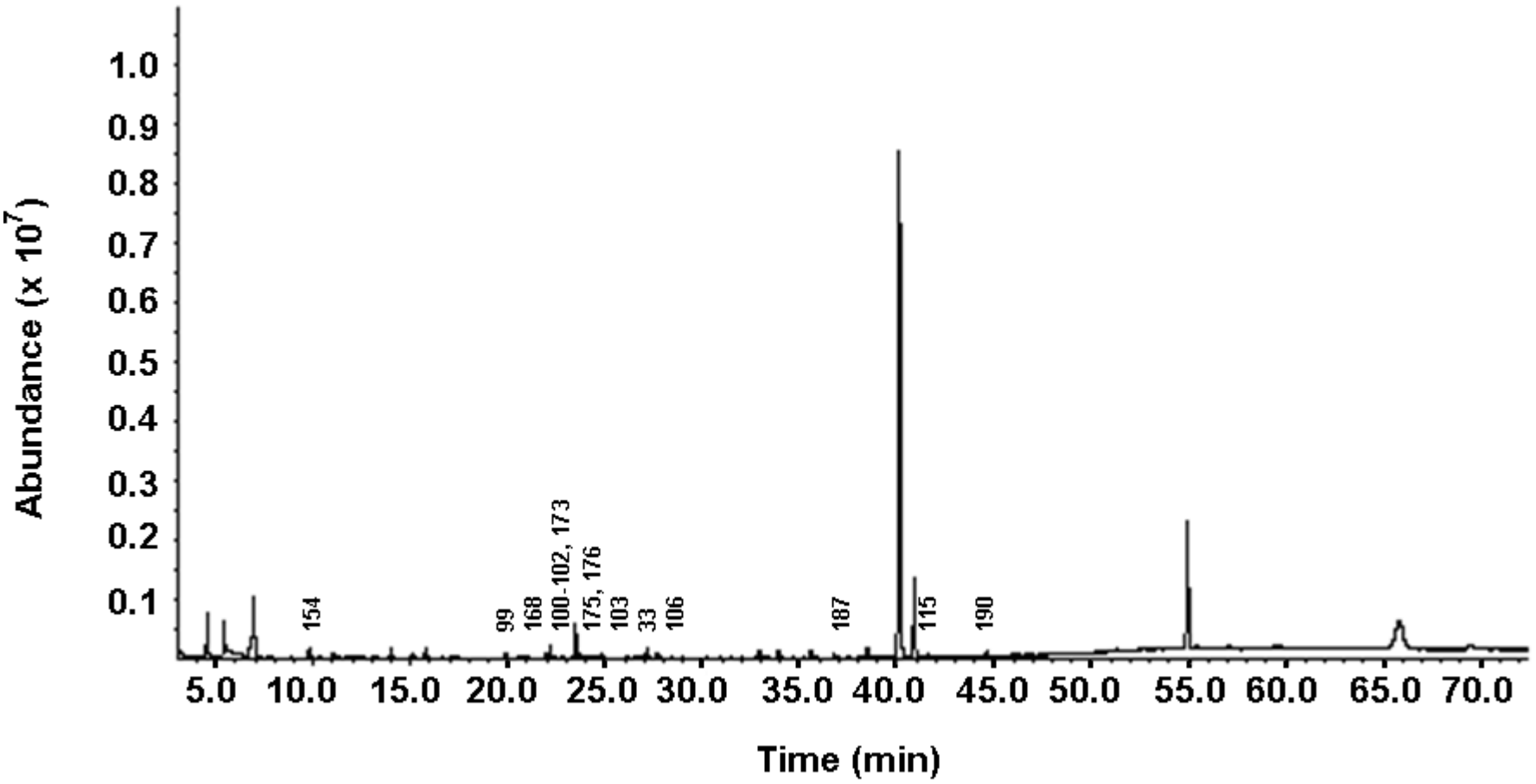
Appendix Figure A15 Total ion current gas chromatogram of a neutral fraction of the extract from CP sample heated at 100°C for 25 min by using COC/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



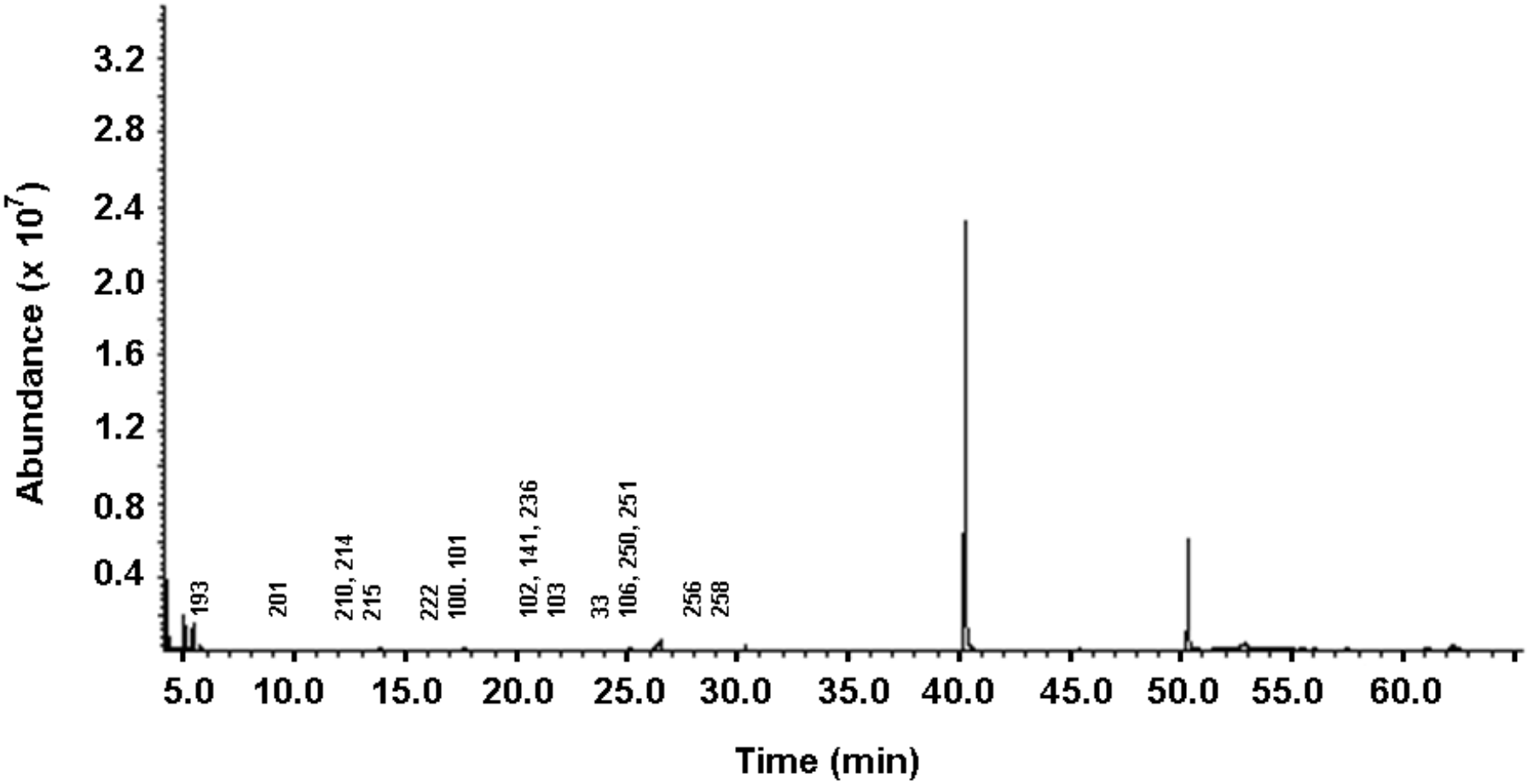
Appendix Figure A16 Total ion current gas chromatogram of a neutral fraction of the extract from CP sample heated at 100°C for 25 min by using COC/GC-MS on DB-5MS column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



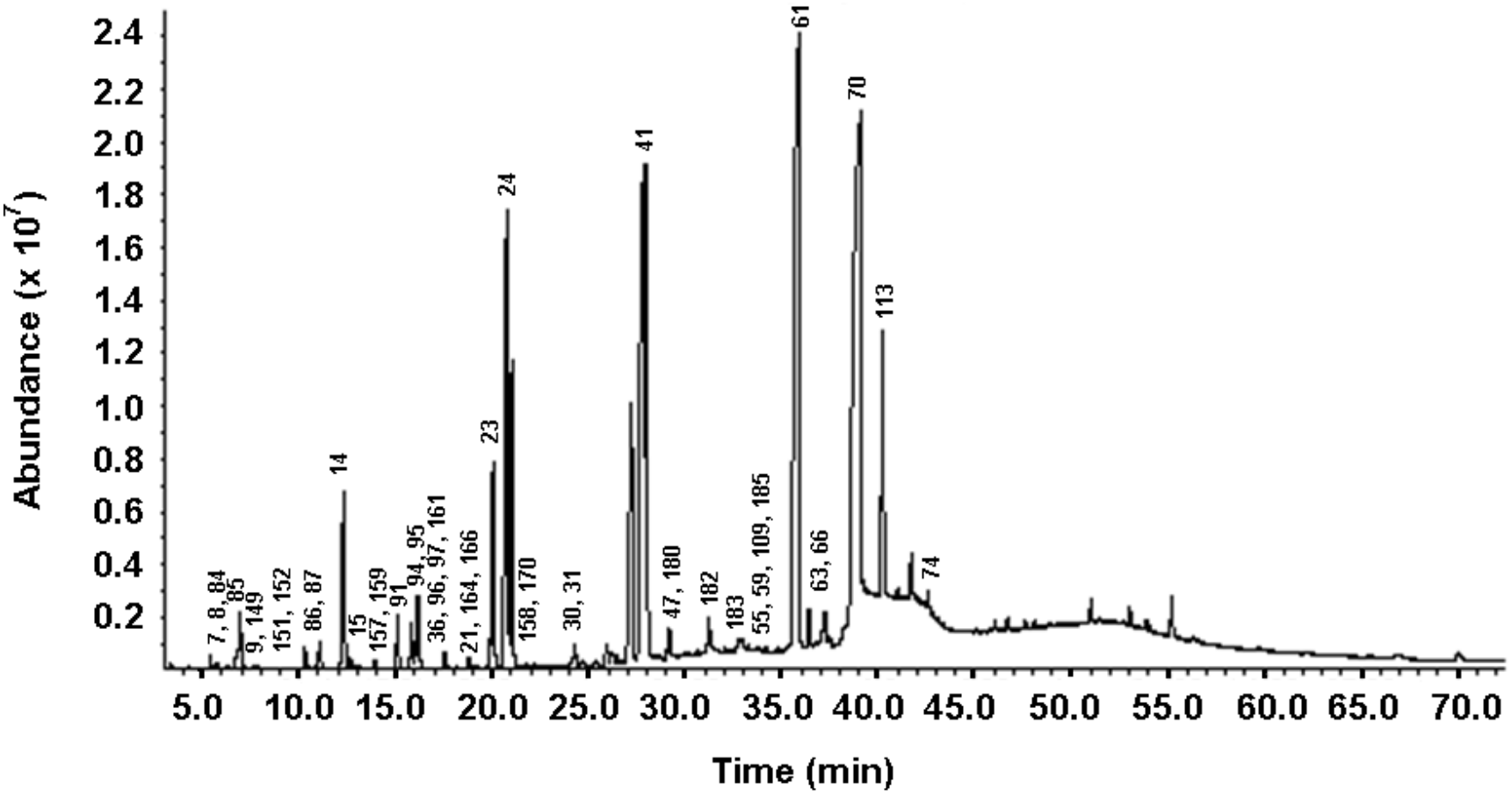
Appendix Figure A17 Total ion current gas chromatogram of an acidic fraction of the extract from CP sample heated at 100°C for 50 min by using COC/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



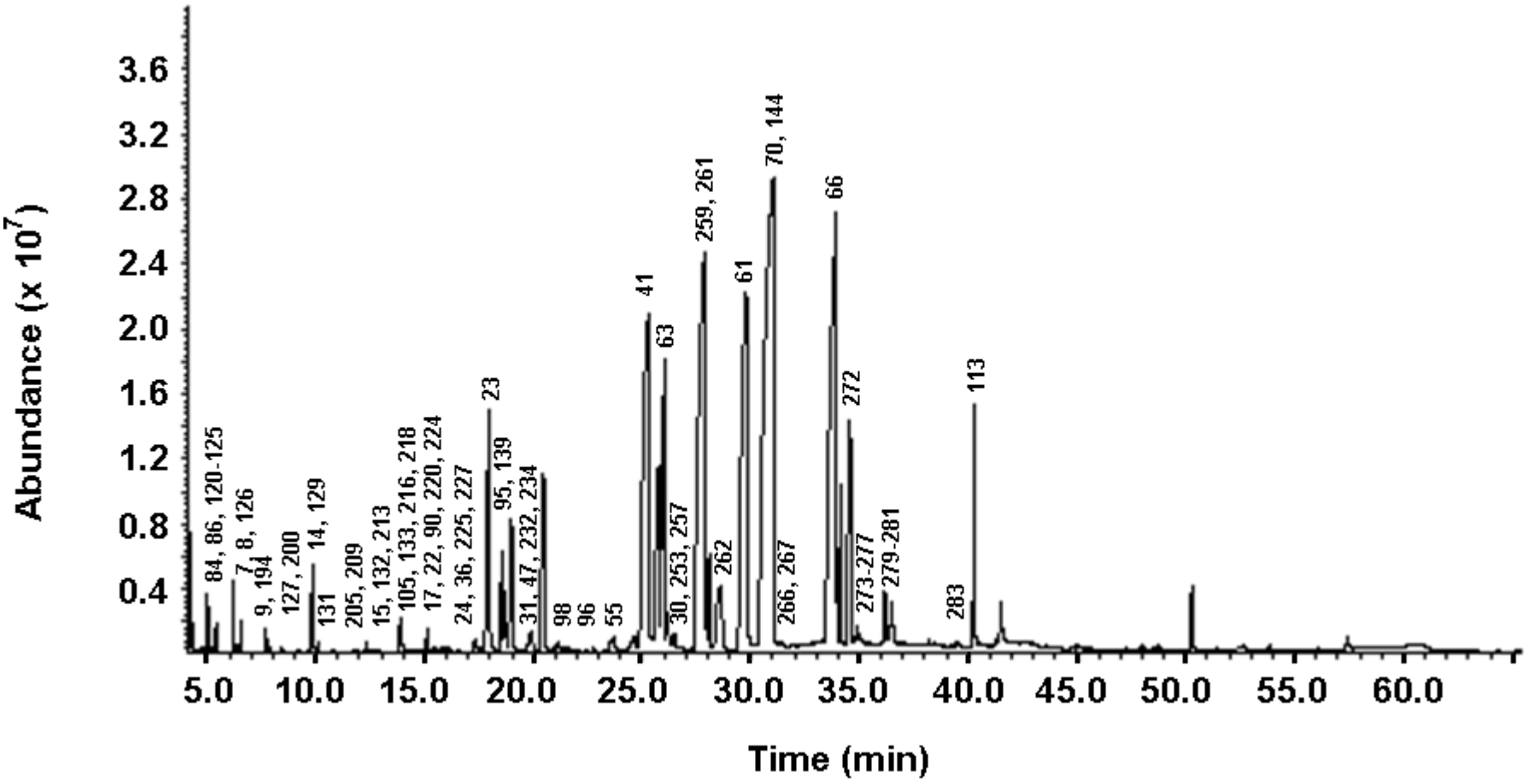
Appendix Figure A18 Total ion current gas chromatogram of a basic fraction of the extract from CP sample heated at 100°C for 50 min by using COC/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2. .



Appendix Figure A19 Total ion current gas chromatogram of a basic fraction of the extract from CP sample heated at 100°C for 50 min by using COC/GC-MS on DB-5MS column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



Appendix Figure A20 Total ion current gas chromatogram of a neutral fraction of the extract from CP sample heated at 100°C for 50 min by using COC/GC-MS on Stabilwax-DA column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.



Appendix Figure A21 Total ion current gas chromatogram of a neutral fraction of the extract from CP sample heated at 100°C for 50 min by using COC/GC-MS on DB-5MS column. For peak numbers see Tables 7-9, and Appendix Tables A1and A2.

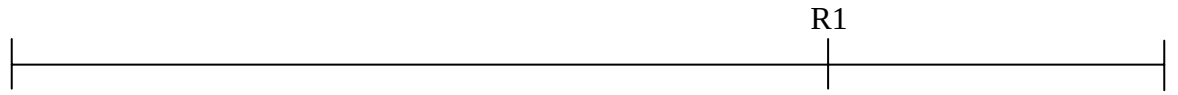
Sensory Evaluation Form

Product Sample: Thai chili paste

Panelist's Name ---------- Sex ---------- Date ----------

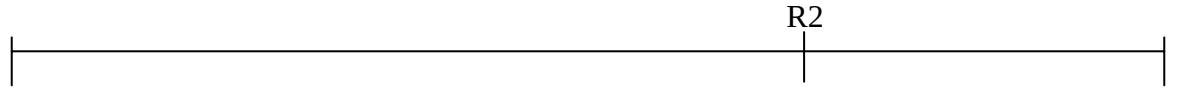
Please describe intensity of aroma characteristics of Thai fried chili paste by placing the mark (I) on the scale to locate the intensity of each sensory attribute

1. Dried shrimp



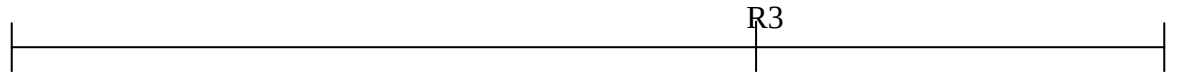
None Very strong

2. Roasted chili



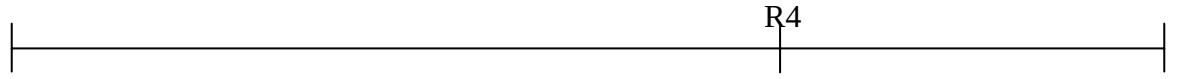
None Very strong

3. Garlic



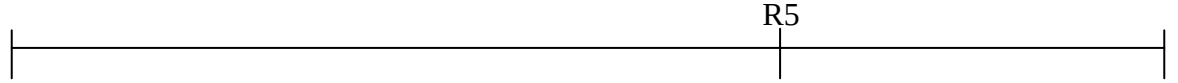
None Very strong

4. Salt aromatic



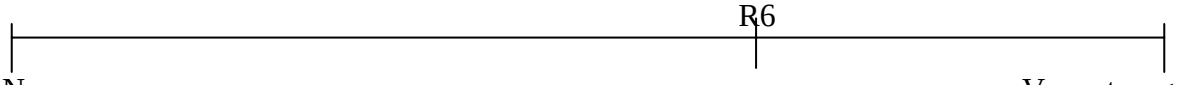
None Very strong

5. Shallot



None Very strong

6. Sweet aromatic



None Very strong

Comment……..

"Thank you"

Appendix Table A3 Change in the attribute of Thai chili paste aroma during heat processing.

Attribute	Sample[a]		
	UH-CP	H25-CP	H50-CP
dried shrimp	6.07 ± 1.11 a	8.93 ± 1.06 b	8.65 ± 0.77 b
roasted chili	5.11 ± 1.06 a	6.93 ± 0.94 b	6.29 ± 1.08 b
garlic	9.89 ± 1.04 a	7.97 ± 0.84 b	7.51 ± 0.76 b
salt aromatic	5.58 ± 0.90 a	7.40 ± 0.83 b	8.10 ± 0.74 c
shallot	7.37 ± 1.09 a	7.69 ± 0.99 a	7.43 ± 0.81 a
sweet aromatic	4.48 ± 1.06 a	7.43 ± 0.81 b	7.46 ± 0.94 b

[a]Average scores obtained by 10 trained panelists ± standard deviation. Means in a row followed by different letters are significantly different (P<0.05); UH-CP = unheated CP, H25-CP = CP heated at 100°C for 25 min, and H50-CP = CP heated at 100 °C for 50 min.

Appendix B

Appendix Table B1 Molecular weight, odor thresholds, boiling point, and solubility of major volatile compounds identified in microencapsulated chili paste oil.

No.[a]	Compound	Molecular weight	Odor threshold (mg/kg)	Boiling point (°C)	Solubility[k]
8	3-methylbutanal	86.132[b]	0.00015[d]	93[i]	VSL
10	dimethyl disulfide	94.201[b]	0.012[d]	109[i]	VSL
15	hexanal	100.159[b]	0.0045[d]	131[i]	SL
22	3,4-dimethylthiophene	112.194[b]	na[g]	na	na
23	1,3-dithiane	120.238[b]	na	na	na
24	allyl methyl disulfide	120.238[b]	na	83[i]	SL
31	dimethyl trisulfide	126.267[b]	0.00001[d]	165[i]	SL
41	diallyl disulfide	146.276[b]	0.03[e]	136[i]	In
47	benzaldehyde	106.122[b]	0.35[d]	178[i]	SL
53	butanoic acid	88.11[c]	0.24[d]	163.5[j]	SL
55	phenylacetaldehyde	120.149[b]	0.004[d]	195[i]	VSL
56	3-methylbutanoic acid	102.13[c]	0.25[d]	176[h]	SL
61	3-vinyl-4*H*- 1,2-dithiin	144.26[c]	na	na	na
66	diallyl trisulfide	178.342[b]	na	112 °C at 16 mmHg[i]	In
70	2-vinyl-4*H*-1,3-dithiin	144.26[c]	na	na	na

Appendix Table B1 (Continued)

No.[a]	Compound	Molecular weight	Odor threshold (mg/kg)	Boiling point (°C)	Solubility [k]
71	hexanoic acid	116.16 [c]	0.24 [d]	202 [h]	In
74	phenol	94.111 [b]	5 [f]	182 [i]	S

[a] Numbers correspond to those in Tables 7-9 and Appendix Table A1 and A2. Molecular weight reported by [b] Leffingwell (2004) and [c] National Institute of Standards and Technology (NIST) Chemical WebBook (2008). Odor threshold in water reported by [d] Buttery and Ling (1998); [e] Teleky-Vamossy and Petro-Turza (1986) and [f] Dietrich *et al.* (1999). [g] n.a. = not available. Boiling point reported by [h] Reineccius (1994), [i] Burdock (2004), and [j] ChemExper Chemical Directory (2008). [k] Solubility in water reported by Reineccius (1994); In = insoluble; SL = slightly soluble; S = soluble; VSL = very slightly soluble and VS = very soluble.

Appendix Table B2 The recovery concentration ± standard deviation (ng/g) of selected volatile compounds that released from each of the spray-dried powders during purging for 60 min.[a, b]

Compound	MD:GA[c]	Purging time (min)				
		5	10	15	20	25
butanoic acid						
	1:2	11.10 ± 0.85 aA	19.45 ± 3.27 aAB	23.16 ± 2.70 aBC	26.29 ± 2.78 aBCD	30.02 ± 2.15 aCD
	1:1	9.43 ± 1.46 aA	13.44 ± 1.28 aAB	16.20 ± 1.29 aBC	18.56 ± 1.40 aBCD	21.25 ± 1.87 aCDE
	2:1	17.84 ± 0.93 bA	33.84 ± 5.19 bAB	43.91 ± 8.25 bABC	54.46 ± 11.31 bABCD	65.46 ± 14.85 bABCDE

Appendix Table B2 (Continued)

Compound	MD:GA[c]	Purging time (min)			
		30	40	50	60
butanoic acid					
	1:2	35.46 ± 3.44 abDE	41.33 ± 5.64 abEF	47.32 ± 5.17 abFG	53.85 ± 6.98 abG
	1:1	23.70 ± 2.06 aDE	27.90 ± 3.96 aEF	31.98 ± 4.03 aFG	35.55 ± 5.01 aG
	2:1	79.08 ± 23.44 bBCDE	93.73 ± 30.03 bCDE	108.60 ± 38.20 bDE	122.80 ± 39.68 bE

Appendix Table B2 (Continued)

Compound	MD:GA[c]	Purging time (min)				
		5	10	15	20	25
3-methylbutanoic acid						
	1:2	6.20 ± 2.04 aA	10.14 ± 1.60 aB	13.76 ± 2.40 aC	17.11 ± 1.44 aD	20.31 ± 1.21 aD
	1:1	7.30 ± 0.01 aA	12.57 ± 3.28 aAB	16.65 ± 4.47 aBC	20.17 ± 3.80 aBCD	23.08 ± 3.39 aCDE
	2:1	9.24 ± 1.57 aA	14.06 ± 3.49 aAB	18.93 ± 4.51 aABC	23.51 ± 4.63 aABCD	27.51 ± 5.72 aBCD

Appendix Table B2 (Continued)

Compound	MD:GA[c]	Purging time (min)			
		30	40	50	60
3-methylbutanoic acid					
	1:2	23.89 ± 1.56 aE	29.52 ±0.51 aF	34.07 ± 0.15 aG	40.01 ± 0.44 aH
	1:1	26.22 ± 3.28 aDE	30.87 ±2.43 aEF	35.33 ± 2.99 aFG	40.25 ± 4.49 aG
	2:1	33.00 ± 7.40 aCDE	39.13 ±8.12 aDEF	44.91 ± 9.25 aEF	51.71 ± 9.74 aF

Appendix Table B2 (Continued)

Compound	MD:GA[c]	Purging time (min)				
		5	10	15	20	25
diallyl disulfide						
	1:2	11.07 ± 1.42 aA	17.04 ± 1.39 aAB	22.06 ±2.18 aABC	27.84 ± 2.26 aBCD	32.58 ± 1.25 aCDE
	1:1	11.56 ± 4.05 aA	20.89 ± 4.42 aAB	25.24 ±5.86 aAB	29.72 ± 6.96 aABC	34.59 ± 7.35 aABCD
	2:1	8.95 ± 4.02 aA	14.99 ± 5.69 aAB	20.09 ±7.85 aABC	26.17 ± 9.03 aBCD	32.47 ± 10.58 aCD

Appendix Table B2 (Continued)

Compound	MD:GA[c]	Purging time (min)			
		30	40	50	60
diallyl disulfide					
	1:2	35.73 ± 8.30 aDE	41.61 ±6.27 aEF	49.28 ± 6.60 aFG	56.56 ± 8.84 aG
	1:1	37.71 ± 8.45 aBCD	43.45 ±11.76 aBCD	49.29 ± 14.24 aCD	54.67 ± 16.17 aD
	2:1	39.30 ± 0.07 aDE	47.68 ±1.24 aEF	54.82 ± 0.82 aFG	61.76 ± 0.31 aG

Appendix Table B2 (Continued)

Compound	MD:GA[c]	Purging time (min)				
		5	10	15	20	25
benzaldehyde						
	1:2	19.36 ± 2.60 aA	37.75 ± 6.93 aAB	60.93 ± 17.82 aBC	84.46 ± 11.78 aCD	107.75 ± 3.16 aDE
	1:1	21.27 ± 0.80 aA	45.20 ± 10.47 aAB	72.06 ± 6.90 aBC	100.13 ± 16.04 aCD	128.20 ± 26.24 aDE
	2:1	17.49 ± 5.42 aA	34.68 ± 11.83 aA	58.80 ± 14.52 aAB	89.23 ± 18.99 aABC	114.95 ± 34.50 aBCD

Appendix Table B2 (Continued)

Compound	MD:GA[c]	Purging time (min)			
		30	40	50	60
benzaldehyde					
	1:2	130.82 ± 9.15 aEF	156.70 ± 2.64 aFG	184.95 ± 23.48 aGH	213.92 ± 18.28 aH
	1:1	156.67 ± 29.55 aEF	199.31 ± 15.04 aF	232.16 ± 16.01 aFG	261.67 ± 19.95 aG
	2:1	140.57 ± 36.72 aCDF	174.91 ± 48.89 aDF	210.53 ± 43.35 aFG	251.85 ± 36.40 aG

Appendix Table B2 (Continued)

Compound	MD:GA[c]	Purging time (min)				
		5	10	15	20	25
phenol						
	1:2	24.06 ± 7.02 aA	40.83 ± 1.68 aB	54.98 ± 1.83 aC	70.84 ± 2.89 bD	87.72 ± 2.29 bE
	1:1	18.30 ± 7.11 aA	35.06 ± 3.01 aB	48.38 ± 0.49 aC	61.08 ± 0.74 aD	74.12 ± 2.11 aE
	2:1	25.58 ± 9.94 aA	41.00 ± 7.38 aB	52.02 ± 5.44 aBC	66.74 ± 0.86 abCD	81.14 ± 3.27 abDE

Appendix Table B2 (Continued)

Compound	MD:GA[c]	Purging time (min)			
		30	40	50	60
phenol					
	1:2	104.48 ± 1.71 bF	121.88 ±3.90 bG	139.92 ± 9.56 aH	158.88 ± 10.82 bI
	1:1	86.40 ± 6.47 aF	101.55 ±2.66 aG	117.21 ± 2.11 aH	132.42 ± 2.40 aI
	2:1	94.90 ± 4.59 abE	111.46 ±7.11 abF	131.93 ± 10.05 aG	152.71 ± 4.37 abH

[a] a and b means within a column with different letters are significantly different (P<0.05); [b] A-I means within a row with different letters are significantly different (P<0.05). [c] MD:GA = the proportion of maltodextrin and gum arabic.

Appendix Table B3 Concentration (ng/g) of selected volatile compounds retained in each of the powder samples during storage for 5 weeks. [a, b]

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
nd[f]	<700	3-methylbutanal				
			1:2	1.42 ± 0.73 aB (100)	0.86 ± 0.30 aAB (60.33)	0.47 ± 0.07 aA (33.06)
			1:1	1.38 ± 0.32 aC (97.02)	1.10 ± 0.73 aBC (77.74)	0.52 ± 0.34 aABC (36.72)
			2:1	0.83 ± 0.15 aC (58.79)	0.46 ± 0.20 aB (32.31)	0.36 ± 0.13 aB (25.29)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
nd	<700	3-methylbutanal				
			1:2	0.34 ± 0.07 aA (23.99)	0.23 ± 0.12 bA (16.00)	0.07 ± 0.02 A (5.24)
			1:1	0.25 ± 0.02 aAB (17.62)	0.15 ± 0.01 abA (10.70)	
			2:1	0.26 ± 0.06 aAB (18.35)		

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1030	741	dimethyl disulfide				
			1:2	0.69 ± 0.30 aB (100)	0.17 ± 0.04 aAB (25.04)	0.12 ± 0.02 aA (17.05)
			1:1	0.55 ± 0.12 aA (78.85)	0.12 ± 0.00 aB (17.80)	0.09 ± 0.02 aB (12.47)
			2:1	0.38 ± 0.12 aA (55.24)	0.25 ± 0.12 aA (35.59)	0.12 ± 0.03 aA (17.45)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1041	800	hexanal				
			1:2	0.26 ± 0.02 abA (54.05)	0.21 ± 0.18 aA (44.20)	0.71 ± 0.11 aAB (149.22)
			1:1	0.47 ± 0.14 bA (100)	0.61 ± 0.09 bA (128.81)	1.15 ± 0.57 aA (242.26)
			2:1	0.21 ± 0.04 aA (44.41)	0.47 ± 0.01 abA (98.32)	1.75 ± 0.31 aAB (367.61)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
1041	800	hexanal				
			1:2	2.04 ± 0.22 aB (429.41)	3.75 ± 0.39 aC (789.88)	11.62 ± 1.39 aD (2446.24)
			1:1	3.98 ± 0.30 bB (838.28)	9.73 ± 0.73 bC (2048.44)	10.87 ± 2.48 aC (2288.48)
			2:1	3.67 ± 0.19 bB (771.87)	12.75 ± 1.66 bC (2685.36)	16.33 ± 1.74 aD (3437.68)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1230	929	1,3-dithiane				
			1:2	5.03 ± 1.79 aB (100)	3.78 ± 1.57 aAB (75.06)	2.25 ± 0.62 aAB (44.65)
			1:1	3.76 ± 0.37 aC (74.65)	2.61 ± 1.18 aBC (51.79)	1.80 ± 0.17 aAB (35.69)
			2:1	1.92 ± 0.18 aC (38.07)	1.48 ± 0.15 aBC (29.38)	1.21 ± 0.51 aAB (24.15)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]											
FFAP	HP-5MS			3				4				5			
1230	929	1,3-dithiane													
			1:2	1.45	± 0.80	aA	(28.77)	1.42	± 0.84	aA	(28.29)	1.14	± 0.39	aA	(22.65)
			1:1	1.63	± 0.65	aAB	(32.42)	1.24	± 0.82	aAB	(24.58)	0.82	± 0.31	aA	(16.34)
			2:1	0.75	± 0.08	aA	(14.83)	0.81	± 0.07	aA	(16.08)	0.71	± 0.06	aA	(14.17)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]											
FFAP	HP-5MS			1				2				3			
1223	914	allyl methyl disulfide													
			1:2	2.90	± 0.07	aA	(100)	2.53	± 1.31	aA	(87.13)	2.06	± 0.53	aA	(71.03)
			1:1	2.82	± 0.98	aC	(97.08)	2.49	± 0.33	aAB	(85.81)	2.35	± 0.43	aAB	(80.98)
			2:1	2.23	± 0.43	aC	(76.85)	1.54	± 0.10	aB	(52.97)	1.40	± 0.05	aB	(48.13)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]											
FFAP	HP-5MS			3				4				5			
1223	914	allyl methyl disulfide													
			1:2	1.89	± 0.63	aA	(65.11)	1.87	± 0.56	aA	(64.39)	1.40	± 0.41	aA	(48.23)
			1:1	2.21	± 0.26	aAB	(76.09)	1.75	± 0.67	aAB	(60.27)	1.17	± 0.32	aA	(40.17)
			2:1	1.08	± 0.04	aAB	(37.32)	1.06	± 0.19	aAB	(36.39)	0.69	± 0.04	aA	(23.90)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
nd	902	3,4-dimethylthiophene				
			1:2	1.56 ± 0.88 aC (74.91)	1.29 ± 0.39 aBC (61.94)	0.47 ± 0.22 aAB (22.78)
			1:1	2.08 ± 0.04 aD (100)	1.07 ± 0.08 aC (51.65)	0.47 ± 0.22 aB (22.40)
			2:1	1.32 ± 0.60 aC (63.36)	0.73 ± 0.10 aBC (34.93)	0.37 ± 0.10 aAB (18.04)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
nd	902	3,4-dimethylthiophene				
			1:2	0.26 ± 0.07 aAB (12.70)	0.17 ± 0.08 aA (7.99)	0.12 ± 0.07 A (5.60)
			1:1	0.23 ± 0.04 aAB (11.10)	0.22 ± 0.08 aAB (10.57)	
			2:1	0.16 ± 0.05 aAB (7.75)	0.18 ± 0.02 aAB (8.90)	

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1313	967	dimethyl trisulfide				
			1:2	11.39 ± 2.03 aB (97.04)	10.27 ± 0.69 bAB (87.47)	8.77 ± 0.96 bAB (74.68)
			1:1	11.74 ± 2.79 aC (100)	7.95 ± 1.74 abAB (67.73)	7.44 ± 1.89 abAB (63.38)
			2:1	10.52 ± 0.92 aD (89.64)	5.25 ± 1.34 aC (44.74)	3.82 ± 0.51 aBC (32.55)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
1313	967	dimethyl trisulfide				
			1:2	7.84 ± 3.49 aAB (66.80)	6.40 ± 3.71 aAB (54.51)	5.06 ± 1.68 aA (43.08)
			1:1	7.39 ± 2.94 aAB (62.95)	5.32 ± 3.20 aAB (45.32)	4.25 ± 1.86 aA (36.19)
			2:1	3.35 ± 0.39 aABC (28.52)	1.94 ± 0.58 aAB (16.51)	1.82 ± 0.15 aA (15.52)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1417	1078	diallyl disulfide				
			1:2	32.85 ± 3.77 bC (100)	27.80 ± 0.35 bC (84.63)	21.40 ± 2.75 aB (65.15)
			1:1	17.27 ± 2.34 aA (52.58)	17.02 ± 2.74 aA (51.82)	15.64 ± 0.93 aA (47.63)
			2:1	23.40 ± 3.32 abB (71.25)	23.29 ± 2.66 abB (70.90)	19.95 ± 2.30 aAB (60.72)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
1417	1078	diallyl disulfide				
			1:2	19.22 ± 1.02 aAB (58.50)	15.97 ± 3.24 aAB (48.62)	14.46 ± 1.80 aA (44.03)
			1:1	15.58 ± 0.92 aA (47.42)	13.41 ± 0.61 aA (40.83)	13.38 ± 0.77 aA (40.74)
			2:1	19.74 ± 2.20 aAB (60.10)	16.57 ± 0.20 aA (50.45)	16.11 ± 1.35 aA (49.04)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1461	nd	benzaldehyde				
			1:2	87.06 ± 6.08 bC (100)	81.43 ± 4.87 bC (93.53)	52.62 ± 4.67 bB (60.44)
			1:1	62.32 ± 10.34 abB (71.58)	45.94 ± 8.22 aB (52.77)	48.29 ± 6.91 abB (55.47)
			2:1	52.44 ± 7.82 aC (60.23)	31.55 ± 5.31 aB (36.24)	30.64 ± 5.00 aB (35.19)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
1461	nd	benzaldehyde				
			1:2	24.12 ± 5.84 aA (27.71)	21.97 ± 3.96 aA (25.24)	
			1:1	24.04 ± 0.16 aA (27.61)	18.94 ± 1.13 aA (21.76)	
			2:1	17.78 ± 2.38 aA (20.42)		

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1571	810	butanoic acid				
			1:2	43.59 ± 4.61 aB (80.37)	39.73 ± 2.05 bB (73.25)	25.25 ± 3.51 aA (46.56)
			1:1	43.72 ± 3.07 aD (80.61)	34.45 ± 4.29 abC (63.53)	23.10 ± 1.15 aB (42.59)
			2:1	54.23 ± 3.34 aC (100)	25.72 ± 5.17 aB (47.42)	20.28 ± 6.69 aAB (37.39)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
1571	810	butanoic acid				
			1:2	22.34 ± 2.68 aA (41.20)	19.30 ± 2.46 aA (35.59)	18.89 ± 0.40 aA (34.83)
			1:1	17.20 ± 4.19 aAB (31.72)	16.66 ± 2.21 aAB (30.71)	14.34 ± 2.92 aA (26.43)
			2:1	16.99 ± 0.96 aAB (31.32)	14.62 ± 0.11 aA (26.95)	12.86 ± 1.85 aA (23.71)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1581	1044	phenylacetaldehyde				
			1:2	2.37 ± 0.17 a		
			1:1	5.06 ± 1.66 a		
			2:1	2.19 ± 0.36 a		

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1611	nd	3-methylbutanoic acid				
			1:2	22.27 ± 0.36 aB (100)	19.34 ± 2.11 aAB (86.85)	18.91 ± 2.24 aAB (84.93)
			1:1	21.74 ± 2.90 aC (97.61)	20.80 ± 1.97 aBC (93.38)	17.61 ± 1.58 aABC (79.07)
			2:1	20.37 ± 2.56 aC (91.47)	17.90 ± 1.36 aBC (80.37)	16.53 ± 0.92 aAB (74.22)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
1611	nd	3-methylbutanoic acid				
			1:2	17.22 ± 0.65 aA (77.32)	16.72 ± 0.86 aA (75.08)	16.30 ± 0.13 cA (73.19)
			1:1	17.49 ± 1.44 aABC (78.52)	16.34 ± 1.52 aAB (73.36)	15.14 ± 0.27 bA (67.98)
			2:1	15.70 ± 0.52 aAB (70.48)	14.03 ± 1.45 aA (62.98)	13.53 ± 0.34 aA (60.77)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1663	1189	3-vinyl-4*H*-1,2-dithiin				
			1:2	14.26 ± 4.00 abA (82.35)	11.37 ± 6.07 aA (65.66)	8.60 ± 2.45 aA (49.66)
			1:1	17.32 ± 0.48 bB (100)	14.86 ± 1.99 aB (85.81)	8.35 ± 5.01 aA (48.23)
			2:1	7.23 ± 2.57 aB (41.76)	7.04 ± 0.07 aAB (40.66)	6.93 ± 2.21 aAB (40.02)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
1663	1189	3-vinyl-4*H*-1,2-dithiin				
			1:2	6.93 ± 3.41 aA (40.00)	6.05 ± 1.89 aA (34.91)	5.44 ± 1.13 aA (31.40)
			1:1	8.07 ± 2.85 aA (46.59)	6.96 ± 1.07 aA (40.20)	4.67 ± 1.74 aA (26.99)
			2:1	5.93 ± 0.75 aAB (34.24)	4.66 ± 1.66 aAB (26.91)	3.02 ± 0.39 aA (17.45)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1721	1304	diallyl trisulfide				
			1:2	222.31 ± 9.82 bE (100)	153.02 ± 13.59 bD (68.83)	131.12 ± 2.86 cC (58.98)
			1:1	142.21 ± 31.23 aC (63.97)	95.51 ± 3.98 aB (42.96)	72.39 ± 6.09 aB (32.56)
			2:1	153.44 ± 5.89 aD (69.02)	143.48 ± 14.50 bD (64.54)	106.12 ± 3.99 bC (47.74)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
1721	1304	diallyl trisulfide				
			1:2	107.66 ±5.05 cB (48.43)	102.16 ± 7.41 bB (45.95)	46.34 ± 2.82 bA (20.84)
			1:1	64.60 ±1.31 aAB (29.06)	65.34 ± 7.03 aAB (29.39)	35.98 ± 2.35 aA (16.18)
			2:1	74.85 ±1.33 bB (33.67)	99.28 ± 2.87 bC (44.66)	57.07 ± 0.85 cA (25.67)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1773	1215	2-vinyl-4*H*-1,3-dithiin				
			1:2	68.40 ± 1.07 cE (100)	41.48 ± 1.06 aD (60.64)	39.40 ± 2.17 bD (57.61)
			1:1	52.86 ± 2.59 bC (77.29)	50.74 ± 3.10 bC (74.18)	21.66 ± 0.56 aB (31.67)
			2:1	45.23 ± 2.14 aD (66.12)	43.79 ± 2.41 abD (64.03)	32.99 ± 4.16 bC (48.24)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
1773	1215	2-vinyl-4*H*-1,3-dithiin				
			1:2	23.34 ± 1.50 bC (34.12)	18.23 ± 2.51 aB (26.65)	11.67 ± 1.91 bA (17.06)
			1:1	9.68 ± 3.72 aA (14.15)	9.78 ± 1.23 aA (14.30)	3.61 ± 2.34 aA (5.28)
			2:1	19.67 ± 5.38 abB (28.75)	15.33 ± 4.68 aAB (22.41)	9.25 ± 1.58 abA (13.53)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
1787	855	hexanoic acid				
			1:2	18.99 ± 2.44 aE (84.05)	12.07 ± 0.12 aD (53.41)	10.40 ± 1.69 aCD (46.05)
			1:1	21.86 ± 3.34 aC (96.76)	14.27 ± 3.54 aB (63.17)	7.83 ± 1.42 aA (34.64)
			2:1	22.59 ± 0.80 aC (100)	11.97 ± 0.85 aB (53.00)	10.87 ± 2.06 aB (48.11)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
1787	855	hexanoic acid				
			1:2	7.35 ± 2.28 aBC (32.52)	3.81 ± 0.28 aAB (16.85)	3.07 ± 0.18 aA (13.57)
			1:1	6.02 ± 0.50 aA (26.66)	3.67 ± 0.12 aA (16.26)	2.89 ± 0.03 aA (12.77)
			2:1	5.44 ± 0.62 aA (24.06)	4.70 ± 0.77 aA (20.81)	3.04 ± 0.28 aA (13.46)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			0	1	2
948	nd	phenol				
			1:2	15.85 ± 0.89 aC (91.95)	15.87 ± 0.23 aC (92.06)	14.07 ± 0.41 aB (81.66)
			1:1	17.23 ± 1.70 aC (100)	16.11 ± 0.96 aBC (93.50)	15.49 ± 1.80 aABC (89.89)
			2:1	16.87 ± 1.38 aC (97.88)	16.97 ± 0.50 aC (98.49)	14.45 ± 0.12 aB (83.86)

Appendix Table B3 (Continued)

RI[c]		Compound	MD:GA[d]	Storage time (weeks) (flavor retention %)[e]		
FFAP	HP-5MS			3	4	5
948	nd	phenol				
			1:2	13.89 ± 0.40 aAB (80.61)	13.31 ± 0.22 abAB (77.22)	12.86 ± 0.16 aA (74.60)
			1:1	14.10 ± 1.07 aAB (81.84)	14.15 ± 0.43 bAB (82.08)	12.82 ± 0.06 aA (74.40)
			2:1	13.37 ± 0.33 aAB (77.59)	12.79 ± 0.23 aA (74.20)	12.40 ± 0.30 aA (71.98)

[a] a-c means within a column with different letters are significantly different (P<0.05); [b] A-E means within a row with different letters are significantly different (P<0.05). [c] Retention indices (RI) calculated from GCO results on DB-FFAP or HP-5MS column. [d] MD:GA = the proportion of maltodextrin and gum arabic. [e] flavor retention expressed as percentage of the flavor content in the powder during storage based on the highest original volatiles found in the capsules at the initial time of storage. [f] nd = not detected.

Appendix Table B4 Mean sensory scores and standard deviation of the aroma attributes quantified in each of the samples during storage for 5 weeks (intensity scale of 0-15 points).[a, b]

Attribute	MD:GA[c]	Storage time (weeks)					
		0	1	2	3	4	5
Chili							
	1:2	6.25 ± 0.88 aA	6.17 ± 0.68 bA	5.99 ± 0.94 aA	6.07 ± 0.92 bA	5.53 ± 0.95 aA	5.38 ± 0.87 aA
	1:1	6.27 ± 0.69 aB	5.31 ± 0.72 aA	5.27 ± 1.41 aA	5.24 ± 0.82 aA	5.61 ± 0.54 aAB	5.73 ± 0.59 aAB
	2:1	5.83 ± 0.54 aA	5.81 ± 1.00 abA	5.88 ± 1.03 aA	5.85 ± 0.72 abA	5.58 ± 0.90 aA	5.65 ± 0.81 aA
Shrimpy							
	1:2	5.45 ± 0.85 aA	5.30 ± 0.77 aA	5.45 ± 0.54 aA	5.54 ± 0.79 aA	4.91 ± 1.12 aA	5.06 ± 0.68 abA
	1:1	5.65 ± 0.74 aA	5.20 ± 0.96 aA	4.99 ± 1.09 aA	4.80 ± 1.14 aA	5.12 ± 0.64 aA	5.61 ± 0.60 bA
	2:1	5.80 ± 0.48 aB	5.56 ± 0.69 aB	5.61 ± 0.66 aB	5.27 ± 0.47 aAB	5.59 ± 0.76 aB	4.78 ± 0.69 aA
Garlic							
	1:2	5.85 ± 0.71 aAB	6.34 ± 1.10 abB	5.52 ± 0.71 aAB	5.75 ± 1.22 aAB	5.96 ± 1.01 aAB	5.32 ± 1.12 aA
	1:1	6.08 ± 0.89 aA	5.66 ± 0.85 aA	6.10 ± 1.52 aA	5.34 ± 0.98 aA	5.91 ± 1.11 aA	6.41 ± 1.00 bA
	2:1	6.51 ± 0.65 aAB	6.75 ± 1.02 bB	5.93 ± 0.79 aAB	5.84 ± 1.31 aAB	5.57 ± 0.64 aA	5.81 ± 1.20 abAB

Appendix Table B4 (Continued)

Attribute	MD:GA[c]	Storage time (weeks)					
		0	1	2	3	4	5
Salt aromatic							
	1:2	6.00 ± 0.67 aB	5.96 ± 0.86 aB	5.20 ± 0.85 aAB	5.87 ± 0.63 aB	4.54 ± 1.31 aA	5.33 ± 1.25 aAB
	1:1	5.67 ± 0.60 aA	5.51 ± 1.00 aA	5.13 ± 1.04 aA	5.24 ± 1.37 aA	5.50 ± 0.64 bA	4.99 ± 1.25 aA
	2:1	6.17 ± 0.57 aB	6.05 ± 0.81 aB	5.38 ± 0.67 aAB	5.46 ± 0.95 aAB	4.84 ± 0.91 abA	4.71 ± 1.07 aA
Sweet aromatic							
	1:2	5.27 ± 0.51 aA	4.83 ± 0.63 abA	4.84 ± 0.62 aA	4.95 ± 0.68 aA	4.67 ± 0.65 aA	4.63 ± 1.03 aA
	1:1	5.17 ± 0.87 aA	4.57 ± 0.93 aA	4.85 ± 0.56 aA	4.92 ± 0.83 aA	5.33 ± 0.51 bA	4.89 ± 1.11 aA
	2:1	5.31 ± 0.42 aB	5.49 ± 0.58 bB	4.93 ± 0.57 aAB	5.26 ± 0.99 aB	4.85 ± 0.52 abAB	4.45 ± 0.97 aB
Overall aroma intensity							
	1:2	6.50 ± 0.90 aB	5.64 ± 0.80 aAB	5.69 ± 0.68 aAB	6.27 ± 0.84 aAB	5.40 ± 0.92 aA	5.42 ± 1.29 aA
	1:1	6.66 ± 0.67 aB	5.55 ± 1.34 aA	5.27 ± 0.85 aA	5.73 ± 0.94 aAB	6.03 ± 0.89 aAB	5.83 ± 1.04 aAB
	2:1	6.49 ± 0.34 aA	6.03 ± 0.82 aA	5.90 ± 1.14 aA	5.77 ± 0.60 aA	6.12 ± 0.81 aA	5.83 ± 1.03 aA

[a] a and b means within a column with different letters are significantly different (P<0.05); [b] A and B means within a row with different letters are significantly different (P<0.05). [c] MD:GA = the proportion of maltodextrin and gum arabic.

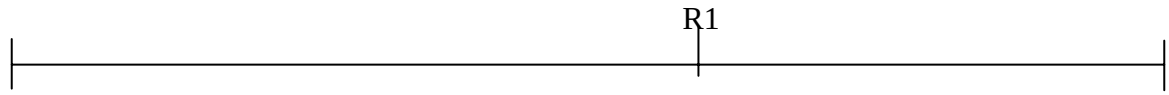
Sensory Evaluation Form

Product Sample: Thai chili paste oil (CPO) powder solution

Panelist's Name _____ Sex _____ Date _____

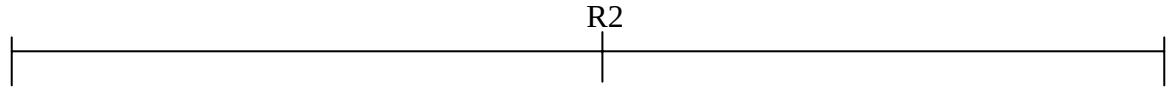
Please describe intensity of aroma characteristics of CPO powder solution by placing the mark (I) on the scale to locate the intensity of each sensory attribute

1. Chili



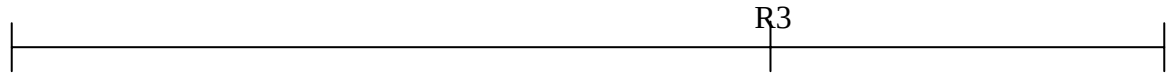
None Very strong

2. Shrimpy



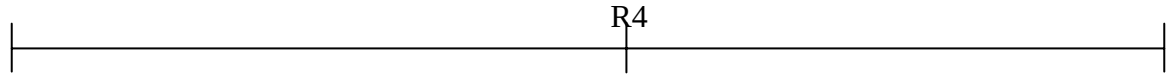
None Very strong

3. Garlic



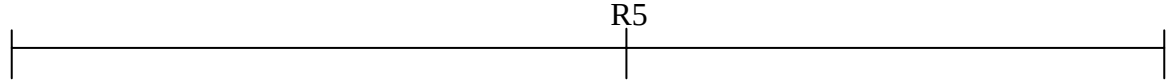
None Very strong

4. Salt aromatic (opened a cap and stood for 3 min before sniffing)



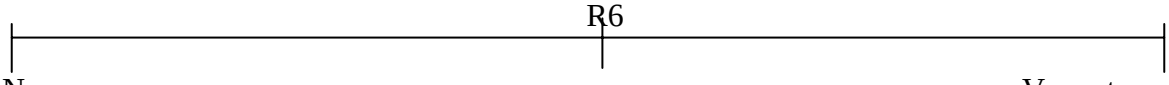
None Very strong

5. Sweet aromatic



None Very strong

6. Overall CP aromatic intensity



None Very strong

Comment……..

……..

"Thank you"

CIRRICULUM VITAE

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