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Volatile compounds and physicochemical characteristics of Thai roasted chilli seasoning

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Abstract

Thai roasted chilli seasoning is a condiment used to enhance the taste and aroma of foods, especially noodles. The characteristics of aroma-active compounds were analysed by using SPME, followed by gas chromatography-mass spectrometry (GC/MS) combined with descriptive sensory analysis to describe the aroma attributes. The roasted chilli seasoning was produced by using the optimum mixing ratio and roasting conditions (91°C for 23 mins). Firstly, the result revealed very low aflatoxin content (0.36 µg/kg) along with undetected aerobic plate count and yeasts and moulds. The predominant odorants in Thai roasted chilli seasoning obtained from GC-MS were mainly acid, Strecker aldehydes, Maillard reaction products, and sulfur-containing compounds. Acetic acid with a pungent aroma note showed the highest intensity, followed by prop-2-en-1-ol and diallyl disulfide contributing to garlic and spicy note respectively. Based on sensory evaluation, roasted, burnt, fatty, garlic-like, onion-like, pungent, spicy and sweet were found as distinctive notes for Thai roasted chilli seasoning. Maillard reaction products such as 2-methyl-butanal, 3-methyl-butanal and furfural contribute to roasted notes, whereas garlic and onion odour notes are from prop-2-en-1-ol and allyl methyl trisulfide. Acetic acid together with (E)-2-heptenal and 1-(2-furanyl)-Ethanone contributed to pungent note in the sample, whereas diallyl disulfide (sulfur-containing volatiles) mainly responsible for spicy odour.

1. Introduction

Roasted chilli seasoning is a condiment or flavouring used to enhance the taste and aroma of foods, especially noodles. This type of seasoning has long been a gastronomic culture, and is not only popular in Thailand but is also popularly consumed in East and Southeast Asia. (Hye-Ryun *et al.,* 2016).

Thai roasted chilli seasoning is typically made of dry chilli, garlic, shallot and oil, but different processes and materials are used in its preparation (Schnuggy, 2012). The different sources of product caused the different colours, odours, and tastes. Although this kind of seasoning is widely consumed, but still lacking any relevant research. Thai per capita consumption of all chilli types and its products was 13.7 g (71% as fresh, 7% in dry and powder forms, and the remaining 22% is processed forms) (National Bureau of Agricultural Commodity and Food Standards, 2016). However, the majority of consumers are still concerned about quality and safety. Most manufacturers lack the knowledge to produce a safe and standardized food product to prevent contamination of foreign matter, mould and toxins. (Hiraga *et al.*, 2004). The application of sodium metabisulfite ($Na_2S_2O_5$) and citric acid in soaking solutions after the blanching process could enhance the quality of dried chilli by preventing the colour changes and intensifying the residue of bioactive compounds (Chaethong and Pongsawatmanit, 2015). In the roasting process, heat transfer and the applied temperature are the most critical parameters. The formation of volatile compounds which gives a special aroma is due to chemical conversions such as hydrolytic reactions, amino acid conversion, oxidative degradation reactions of lipids (fatty acids and carotenoids), Maillard, and caramelization browning (Cremer and Eichner, 2000; Schenker *et al.,* 2002; Cuicui and Lixia, 2018).

The volatile composition of chilli seasonings depends on many factors such as chilli variety, ingredients and process. Several studies on the identification of volatile compounds in this product and ingredient have been reported. However, the key aroma-active compounds in Thai roasted chilli seasoning are still unclear. Volatiles in fresh chilli, dried, and the

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effects of the drying method on the volatile profile have been determined (Jun *et al*., 2005; Toontom *et al*., 2012). Diverse volatile components including aldehydes, ketones, acids, esters, and sulfur compounds were found in dried samples than in fresh ones. Strecker aldehydes such as acetaldehyde, 2-methylpropanal, 2-methylbutanal, and 3-methylbutanal were only detected in dried red peppers (Cremer and Eichner, 2000; Jun *et al*., 2005). The formation of these aldehydes indicates that the Maillard reaction that occurred during drying correlated to the concentrations of free amino acids present in the samples. Muangkote *et al*. (2018) identified about 25 compounds in the extract of roasted dried chilli cv. Jinda. They reported that N-decyl acetamide and Maillard reaction products were produced during roasting which increases the total phenolic content value and the antioxidant activities. Toontom *et al.* (2012) detected 11 groups of volatile compounds in dried Chee fah chilli (*Capsicum annuum* Linn. var. *Acuminatum Fingarh*.), among which dominated in the dried chilli volatile flavour attributes detected by the panellists including acids, ketones, pyrroles, furans, and aldehydes. The addition of dried chilli pepper (*Capsicum annum*) in chilli pepper aromatized olive oil caused an increase in hexanal, related to oxidation processes (Caporaso *et al*., 2013). Rotsatchakul *et al*. (2008) identified about 80 aroma components in Thai fried chilli paste (nam phrik phao), found predominant odorants sulfur-containing compounds, which from garlic and shallot, in heated and unheated chilli paste. They reported that the predominant odorants in chilli paste heated at 100°C for 25 mins were 3-vinyl-4*H*-1,2-dithiin, allyl methyl disulfide, and allyl methyl trisulfide.

The purpose of the present study was to identify characteristic aroma active compounds of Thai roasted chilli seasoning using SPME, followed by gas chromatography-mass spectrometry (GC-MS). Descriptive sensory analysis of roasted chilli seasonings was also evaluated to describe the aroma attributes. The physicochemical characteristics and microbiological properties were also investigated to confirm the product quality. Our results will help in the understanding of key flavour characteristics of Thai roasted chilli seasoning (ground chilli with oil) and could be used to create ingredient formulations or processing to give the desired flavouring characteristics.

2. Materials and methods

2.1 Materials and process description

The main ingredient is red pods of fresh chilli (*Capsicum annuum* L.) of the Jinda cultivar, red shallot (*Allium ascalonicum* L.), and Chinese garlic (*Allium sativum* L.) obtained from a wholesale market in Pathumthani province, Thailand was selected, kept at 4±2°C. Sodium metabisulphite and citric acid were food grade. All reagents used were of analytical grade.

The process to obtain the high quality of Thai roasted chilli seasoning begins with the dried chilli pod preparation (Chaethong and Pongarlicgsawatmanit 2015). The sound and red pods of fresh chilli were washed, drained, and blanched (10 kg/batch) at 100°C for 3 mins using a ratio of chilli and water of 1:10 (w/w). Drained and immediately soaked in the solutions with combinations of 0.25% w/w $Na_2S_2O_5$ and 1.0% w/w citric acid using a ratio of blanched chilli to the solution of 1:3 for 30 mins at room temperature (T = 30±2°C) prior to drying in a conventional tray dryer at 65°C for 14 hrs to obtain dried chilli with a final moisture content of approximately 8% wb. Fried shallot and garlic were prepared by peeling, washing, slicing to 0.1 cm, mixing with 10% w/w of soybean oil, and drying at 65°C for 7 hrs. All dried ingredients, dried chilli without stem, fried shallot, and fried garlic, were coarse ground and mixed with the ratio 5:1:1 (w/w), and then mixed between ground dried ingredients and soybean oil was 2:1 (w/w). Mixed seasonings were then roasted under roasting temperature at 91°C for 23 min using a hot air oven (Yamato DV61, Tokyo, Japan) to maintain the desired temperature. Two hundred grams of mixed ingredients with a thin layer were roasted on the heated stainless steel tray in the oven. Figure 1 reports the flow chart for the production of this seasoning.

2.2 Physicochemical characterization

The roasted chilli seasoning (3 g) was weighed into aluminium cans for determination of moisture content in a hot air oven at 105°C for 6 hrs or until a constant weight was obtained (adapted from AOAC, 2000). The water activity of each sample was measured at 25°C using a water activity instrument (Series 3, AquaLab, Washington, USA). The measurements were taken in triplicate, and the mean and standard deviation are reported. The tridimensional $L^*a^*b^*$ colour space was measured by using a Color reader (MINOLTA CR-10, Osaka, Japan). Each sample was placed in a plastic Petri dish (30 mm in diameter) for measurement of reflectance in the CIE $L^*a^*b^*$ colour space.

Total aflatoxin was analysed using a Total Aflatoxin ELISA kit (MycoJudge, Tokyo, Japan). Briefly, this assay is based on indirect-competitive ELISA. The antibody binding to AFB1-BSA is competitively inhibited by AFB1 in the samples. A total of 10 g of roasted chilli seasoning was extracted with 100 mL of 70% methanol, centrifuge at 3,000 rpm for 3 mins and the clear supernatant was filtered through a nylon syringe filter of 0.45 micron. Thirty microlitres of standard and

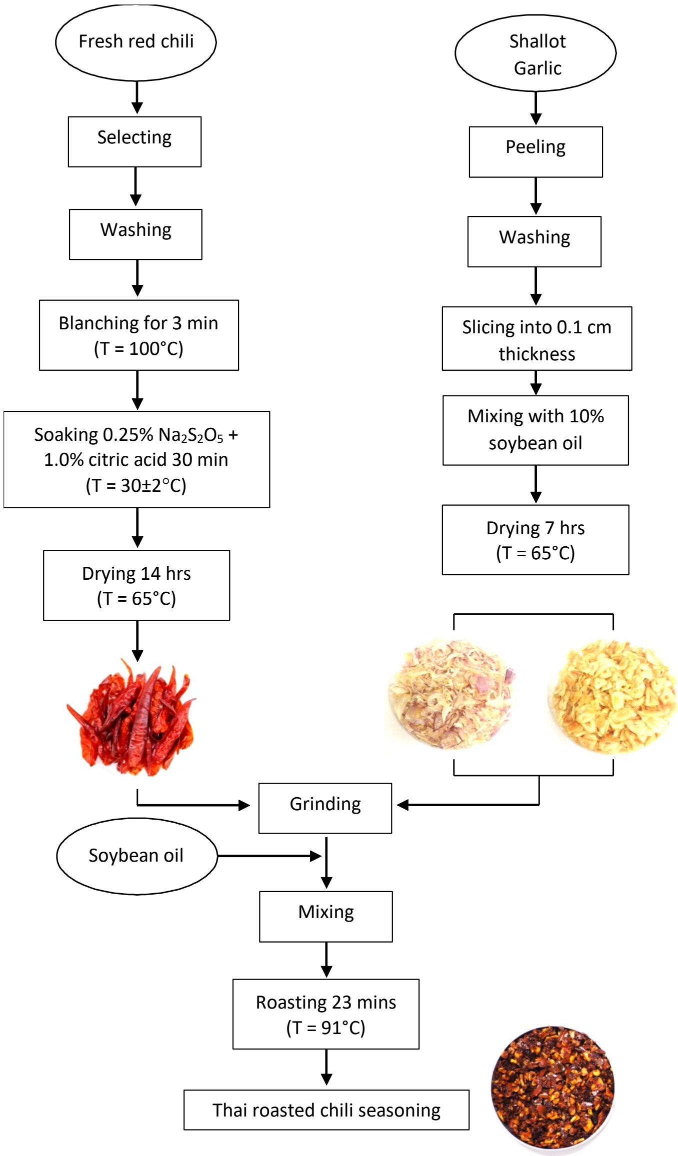

Figure 1. Flow chart of Thai roasted chili seasoning production

extracted samples were added to a 96-well microtiter plate. The ELISA was subsequently developed by the addition of a TMB (3,30,5,50-tetramethylbenzidine) substrate solution. Colour development was in inverse proportion to the amount of AFB1 in the samples. The concentration of AFB1 was quantified by measuring the absorbance at 450 nm by comparison with a reference standard.

Thiobarbituric acid reactive substances (TBARS) were analysed according to the methods of TBARS assay for food and beverages (Oxford Biomedical Research, Inc., Toyko, Japan). One gram of sample was homogenized with 15 mL of deionized water before being adjusted to 20 mL volume. The homogenized mixture was centrifuged at 10,000 rpm for 10 mins and filtered through a nylon syringe filter of 0.45 micron. The filtrate (1 mL) and 2-thiobarbituric acid were mixed and incubated at room temperature for 1 hr. The absorbance of the samples was measured at 530 nm. The amount of TBARS was calculated and expressed as mg of malondialdehyde (MDA) per 1 kg of sample.

Total bacteria counts were determined using a plate count agar and incubated at 35°C for 48 hrs (BAM, 2001a). Yeasts and moulds were enumerated by plating on potato dextrose agar after incubation at 25°C for 5 days (BAM, 2001b).

2.3 Determination of volatile compounds

One gram of seasoning was placed in a 22 mL headspace vial and screwed with an aluminum cap. The sample vial was extracted using headspace solid-phase microextraction (SPME). The sample was equilibrated at 40°C with an agitator speed of 250 rpm for 10 min. An SPME fibre coated with (30/50 µm DVB/Carboxen^{TM}/PDMS StablFlex^{TM}, Supelco: 57348-U) was adsorbed for 10 min and transferred immediately to an injection port of gas chromatography (GC) and desorbed at 230°C for 5 mins. Chromatographic analyses were performed on an Agilent 7890B GC system coupled with Agilent 5977B mass spectrometric detection (Agilent Technologies, Santa Clara, CA). Volatile compounds were separated on a DB-WAX UI column (60 m × 0.25 mm, film thickness 0.25 µm). The initial oven temperature was 40°C, held for 4 mins, ramped at 4°C/mins to 240°C, and held for 3 mins with a total run time of 56 mins. The Helium carrier gas was used at a flow rate of 1 mL/min. Qualitative ion fragments in the scan mode with the mass range 30−300 amu acquired from MS were analysed with mass spectral database NIST 08 (NIST Library Search program), and hypothetical compounds with a match score >80% were selected as the possible component of volatile compounds.

2.4 Sensory analysis

The descriptive sensory analysis of roasted chilli seasonings was evaluated by 12 trained panels who regularly consumed Thai roasted chilli seasoning and were randomly recruited from Rangsit University at Central Location Test (CLT). Freshly prepared seasonings (2 g) were placed into the sniff plastic cup (125 mL). Two roasted chilli seasonings available on the market were selected and subjected to assess differences in sensory quality. The panellists were asked to list aroma attributes that were representative of the seasonings for descriptive analysis. Eight attribute descriptors including roasted, burnt, fatty, garlic-like, onion-like, pungent, spicy and sweet were in agreement with the panellists to use for evaluation on the aroma profile. Each sample was evaluated in triplicate, and the sensory scores were averaged and presented in the form of a spider diagram for all aroma notes.

2.5 Statistical analysis

All measurements were performed using three independently prepared samples. The results were reported as the mean value ± standard deviation. The data were subjected to analysis of variance (ANOVA) using the SPSS V.12 statistical software package (SPSS (Thailand) Co., Ltd., Bangkok). Duncan's multiple range test at 95% probability was applied to determine significant differences among the means of treatment parameters.

3. Results and discussion

3.1 Physicochemical characteristics

The results of the characteristic analysis of high-quality Thai roasted chilli seasoning are reported in Table 1. The moisture content and a_w are very important indicators for the dried products because they are strongly correlated with storage stability (Labuza *et al.,* 1971). The water activity of roasted chilli seasoning was 0.329, which was much lower than the critical a_w value (0.650) that all microbial growth is inhibited (Park, 2008). This is due to only dry ingredients mixed in the formulation and a_w value was subsequently reduced by the roasting process. According to previous results, the optimum mixing ratio between dried ingredients and oil for chilli seasoning was 2:1 (w/w) because high oil content was not preferred (Surasereewong *et al*., 2021). In addition, lipid oxidation can lead to significant changes in the sensory properties of oily products that can be detected by consumers (Fennema, 1996). Formulas that use less oil content may prevent rancidity and provide a longer shelf life. In this study, the TBARS value of the freshly prepared samples was about 2.01 mg MDA/kg. The TBARS value is the most common indicator used to measure the degree of lipid oxidation, consumers can detect foreign smell at TBARS >3.0 and the value >7.0 means off-odours and deteriorates (Samdaeng *et al*., 2015). Colour is generally recognized as a quality factor in the buying decision of chilli products (Klieber, 2000). In studying the colour characteristics of the seasoning were 21.46 for $L*$ (lightness), 15.35 for $a*$ (redness), 7.11 for $b*$ (yellowness). The product colour is similar to fried chilli paste (nam phrik phao) is about 25.20 for L *, 8.50 for a *, 2.90 for b *, which is the natural colour characteristic of the ingredients used and the yellowness made product was brown from roasting (Rattanathawornkiti, 2010). For microbiological properties including aerobic plate count, yeasts and moulds were not detected in the seasoning (Table 1). It might be due to the preparation step of dried ingredients and the heating process reducing the initial microbial load. The results were confirmed by very low aflatoxin content (0.36 μg/kg), which is regulated by Thai Community Product Standard (2013) (321: nam prik pad), in which the maximum aflatoxin content was set at 20 μg/kg.

Table 1. Physicochemical and microbiological properties of roasted chili seasoning.

Properties	Mean±SD (n = 3)
Physicochemical properties	
Moisture (%, wb)	5.11±0.01
a_W	0.329±0.000
$L*$	21.46±0.53
$a*$	15.35±0.65
$b*$	7.11±0.30
TBARS (mg MDA /kg)	2.01±0.20
Total aflatoxin (μg/kg)	0.36±0.00
Microbiological properties	
Aerobic Plate Count (CFU/g)	ND
Yeasts and moulds (CFU/g)	ND

ND: not detected.

3.2 Volatile compounds

Volatile compounds of Thai roasted chilli seasoning were analysed by SPME followed by GC-MS as shown in Figure 2. A total of 49 volatile compounds were detected. The volatile compounds were primarily composed of alcohols, aldehydes and ketones, acids, esters, alkanes, terpene hydrocarbon, sulfur-containing compounds, and miscellaneous compounds (Table 2). Acetic acid (34) (pungent sour) was the most intense aroma compound, followed by prop-2-en-1-ol (19) (garlic), heptane (2), acetonitrile (15), and diallyl disulfide (37) (spicy). A similar result was reported by Toontom *et al.* (2012), acetic acid was mainly present in dried chilli and increased after drying. Acetic acid (pungent sour), and alcohols played important roles in the aroma quality of Korean fermented red pepper paste (*gochujang*) (Kyung and Hyung, 2014). Acetic acid was the main volatile acid in peppers, which was produced by carbohydrate catabolism (Mateo *et al*., 1997). Prop-2-en-1-ol (allyl alcohol) is reportedly present in garlic oil and had garlic liked odour (Chung *et al.,* 2007). This component was an anti-yeast and fungistatic activity in garlic following prolonged heating, which is generated from alliin (Choi and Kyung, 2005). Diallyl disulfide (spicy) is sulfur-containing volatiles from garlic that is responsible for medicinal properties and also presented the most abundant flavour in garlic oil (Calvo-Gómez *et al.,* 2004). Breakdown of thiosulfinate plays a major role in the formation of di- and trisulfide found as components of garlic. Upon mild heating, or even at room temperature, the thiosulfinates undergo nonenzymatic rearrangement leading to the formation of symmetrical and mixed mono-, di-, and trisulfides and these are the main components of commercial garlic oils (Brodnitz *et al*., 1971).

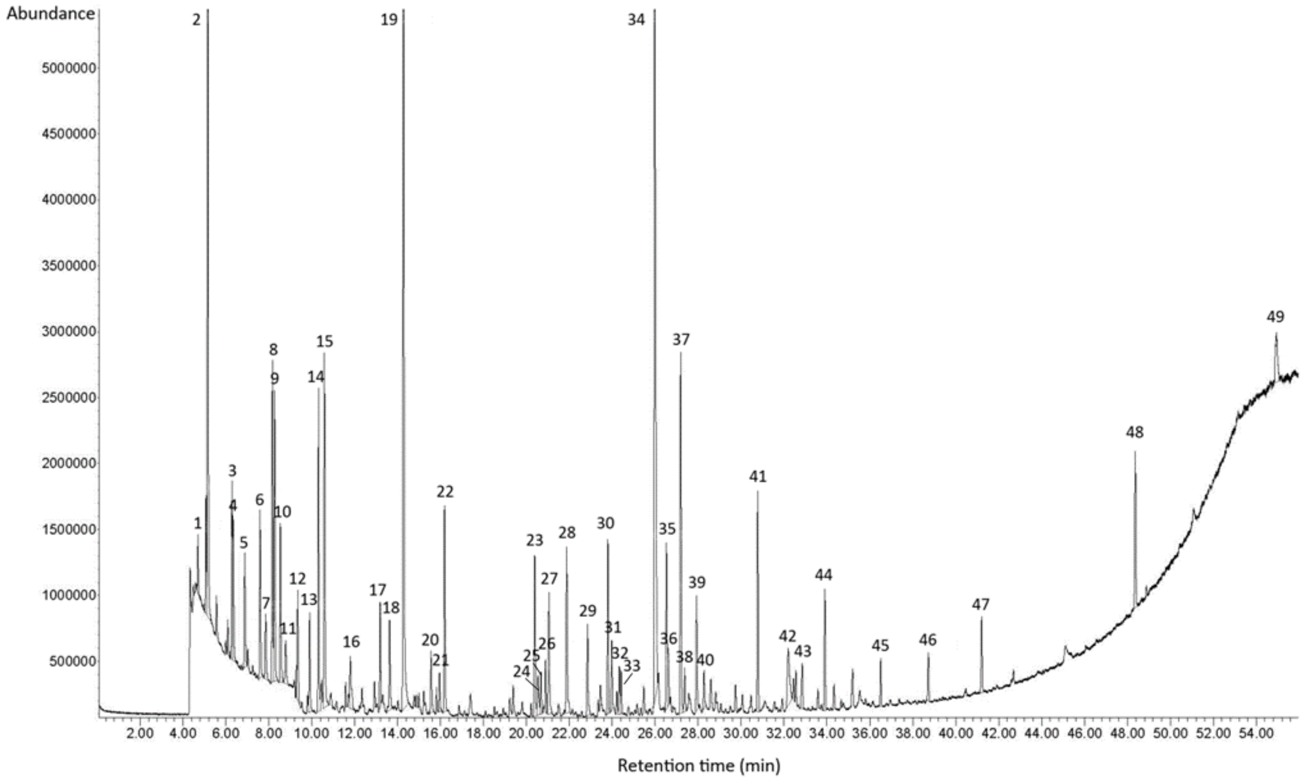

Figure 2. SPME-GC/MS chromatogram of Thai roasted chili seasoning obtained from roasting at 91°C for 23 mins.

Table 2. Volatile compounds of Thai roasted chili seasoning roasting at 91°C for 23 mins.

Peak No.*	Compounds name	Peak Area (%)	Quality (%)	Flavor description
	Alcohols			
11	Ethanol	0.60	86	Sweet
19	Prop-2-en-1-ol	12.07	80	Garlic liked odor
46	Benzyl alcohol	0.59	97	Sweet, Flower
	Aldehydes and Ketones			
3	2-methyl- Propanal	1.65	91	Wine
4	2-Propanone	1.54	80	Fruity flavor
8	2-methyl-Butanal	3.41	91	Roasted, Musty, Chocolate, Nutty, Fermented
9	3-methyl-Butanal	3.20	95	Malty, Roasted flavor
13	Pentanal	1.20	91	Fermented, Bready, Fruity, Nutty,Berry
17	Hexanal	1.10	95	Fruity, Hey-liked, Freshy cut grass, Leaf odor, Grassy
18	2-methyl-(E)-2-Butenal	1.19	94	Green, Fruity
21	2-methyl-2-Pentenal	0.45	96	Spicy, Fruity, Green, Pulpy
24	3-hydroxy 2-Butanone	0.65	80	Sweet buttery, fatty
27	1-hydroxy-2-Propanone	1.70	49	Sweet flavor
28	(E)-2-heptenal	2.13	98	Fatty, Green, Pungent
35	Furfural	2.13	95	Roasted flavor, Caramel, Woody
36	2-furan-carboxaldehyde	0.75	38	Smoke
39	1-(2-furanyl)-Ethanone	1.37	91	Diffusive, Pungent, Green, Ethereal
42	Benzeneacetaldehyde	1.52	95	Roasted, Sweet, Flora
47	1-(1H-pyrrol-2-yl)-Ethanone	0.92	94	Musty, Nutty-liked with a coumarin nuance
48	2,3-dihydro-3,5-dihydroxy-6-methyl-4H-Pyran-4-one	1.90	90	Caramel odor
	Acids			
34	Acetic acid	12.74	91	Sharp vinegar, Pungent, Sour
43	2-methyl-Butanoic acid	0.63	80	Cheesy

* Peak nos. refer to peaks in Figure 2.

Table 2 (Cont.). Volatile compounds of Thai roasted chili seasoning roasting at 91°C for 23 mins.

Peak No.*	Compounds name	Peak Area (%)	Quality (%)	Flavor description
	Esters			
6	Acetic acid ethyl ester	1.82	91	Fruity
7	Methyl-carbamic acid ethyl ester	0.85	9	-
22	Isobutyl 3-methylbut-3-enyl carbonate	2.31	50	Fruity
26	2-methyl-Propanoic acid 4-methylpentyl ester	0.50	90	Sweet, Floral, Fruity,
30	4-Methylpentyl 2-methylbutanoate	1.78	91	Fruity (tabasco, chili pepper)
33	4-Methylpentyl 3-methylbutanoate	0.45	90	Fruity,Waxy,Soapy
40	4-Methylpentyl 4-methylpentanoate	0.49	45	Weak fruit, Peach
	Alkanes			
1	Hexane	0.49	91	Alkane
2	Heptane	7.86	91	Alkane
10	dichloromethane	1.81	97	-
12	2,2,4,6,6-pentamethyl-Heptane	1.34	83	-
29	2-methyl-Tridecane	1.08	94	-
49	1,4,7,10,13,16- Hexaoxacyclooctadecane	1.32	86	-
	Terpene hydrocarbon			
44	gamma-himachalene	1.36	99	-
	Sulfur-containing compounds			
20	Diallyl sulfide	0.85	99	Green, Floral
23	Methyl 2-propenyl Disulfide	1.90	96	Spicy
25	(E)-1-Methyl-2-(prop-1-en-1-yl)disulfane	0.51	97	-
31	Dimethyl trisulfide	0.83	97	Alliaceous, Onion, Fresh, Savory, Green
37	Diallyl disulfide	4.08	95	Spicy
38	(E)-Allyl-2-(prop-1-en-1-yl)disulfane	0.56	95	Sulfurous alliaceous
41	Methyl 2-propenyl trisulfide	2.41	98	Alliaceous creamy garlic onion
45	Di-2-propenyl trisulfide	0.51	98	Alliaceous, green, onion
	Miscellaneous compounds			
5	Hexamethyl-cyclotrisiloxane	1.25	90	-
14	Octamethyl-cyclotetrasiloxane	3.14	91	-
15	Acetonitrile	4.55	53	Ether-liked, Sweet, Burnt, Faint, Distinct
16	Toluene	0.90	91	Paint, Sweet, Pungent, Benzene-liked
32	4,5-dihydro-2,4-dimethyl-Oxazole	0.57	64	Boiled beef

* Peak nos. refer to peaks in Figure 2.

Aldehydes and ketones are the major groups of volatile compounds presented in Thai roasted chilli seasoning. The formation of aldehydes indicates that the Maillard reaction occurred during the heating of these peppers (Cremer and Eichner, 2000). 2-Methyl-butanal (8), 3-methyl-butanal (9) showed the highest abundance, which provided characteristic roasted garlic (Molina *et al.,* 2017). This volatiles also have been identified in dried red peppers were in the group of Strecker aldehydes derived from isoleucine and leucine, respectively (Cremer and Eichner, 2000; Muriel *et al*., 2004; Jun *et al*., 2005). The formation of these aldehydes indicates that the Maillard reaction that occurred during roasting correlated to the concentrations of free amino acids present in the samples. The concentration of these aldehydes of Thai fried chilli pastes increased with increasing heating time at 100°C (Rotsatchakul *et al.* (2008). Strecker degradation is caused by oxidation of the amino acids that involves the interaction of dicarbonyls such as diacetyl, pyruvaldehyde, hydroxyacetone and hydroxydiacetyl to degrade amino acids to aldehydes (Bailey, 1994). Furfural (35) was the major volatile component in this group, is usually an indicator of thermal damage during roasting (Vanderhaegen *et al*., 2003). (E)-2-heptenal (28) was reported by Zhang *et al.* (2019) which found in onion, generally give green and pungent odours. Caporaso *et al.* (2013) reported that (E)-2-heptenal originating from linoleic acid auto-oxidation. The next identified component in the seasoning is benzeneacetaldehyde, an aromatic compound that has been previously identified by Toontom *et al.* (2012) in dried chilli and which have burnt sugar, sweet odour in the finished product. 2-methyl-(E)-2-butenal (18), hexanal (17), 1-hydroxy-2-

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Propanone (27) were found in fresh chilli pepper, dried chilli and their products which represented a fruity and grassy flavour (Jun *et al*., 2005; Toontom *et al.,* 2012; Apichartsrangkoon *et al.,* 2013; Hye-Ryun *et al.,* 2016; Molina *et al.,* 2017; Morales *et al.,* 2018). Cremer and Eichner (2000) indicated that hexanal is known as an oxidation product of enzymatic as well as autoxidative linoleic acid oxidation, increased strongly with heating. Thus, it could be used as a marker compound for the heat treatment of paprika. 2,3-Dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one (48) was detected in the chilli seasoning, is mainly a flavouring compound in dried chilli which have a caramel flavour (Toontom *et al.,* 2012). 2-Propanone (4), 1-(2-furanyl)-ethanone (39) were present in the sample representing a pungent odour. This compound has been reported in fresh garlic, black garlic, and chilli pepper (Molina *et al.,* 2017; Morales *et al.,* 2018). 3-hydroxy 2-butanone (24) has been identified in *gochujang* and fried onion which provide sweet buttery, fatty odour (Kyung and Hyung, 2014; Zhang *et al.,* 2019).

Another group is an alkane that was found in high amounts in Thai roasted chilli seasoning, heptane (2) showed the highest abundance, followed by dichloromethane (10), 2,2,4,6,6-pentamethyl-heptane (12), 1,4,7,10,13,16-hexaoxacyclooctadecane (49). Heptane compound has been previously identified by SPME-GC/MS in olive oil and not found in dried chilli pepper Caporaso *et al.* (2013). Gamma-himachalene, terpene hydrocarbon was also detected in Thai green chilli paste, fermented garlic, and sun-dried chilli (Toontom *et al.,* 2012; Apichartsrangkoon *et al.,* 2013).

In the group of sulfur-containing compounds, except for the most abundant diallyl disulfide (37), there are methyl 2-propenyl trisulfide (41), methyl 2-propenyl disulfide (23), diallyl sulfide (20), dimethyl trisulfide (31). This group is the key volatile compound in garlic especially allyl methyl sulfide, diallyl disulfide, diallyl trisulfide, and di-2-propenyl trisulfide (Singh *et al.*, 2014). Kyung *et al.* (2002) report that diallyl trisulfide was generated from alliin in garlic by heating to 121°C for 15 mins. Sulfur compounds are decomposition products of thiosulfinates and are derived from amino acid flavor precursors (alliin) of the *Allium* family including garlic and shallot (Wu *et al*., 1982; Block *et al*., 1986). Sulfur-containing compounds such as 3-vinyl-4*H*-1,2-dithiin (pungent, garlic), allyl methyl disulfide (pungent, fresh garlic), and allyl methyl trisulfide (eggy, meaty, garlic) were founded as predominant odorants, which from garlic and shallot, in Thai fried chilli paste (nam phrik phao) heated at 100°C for 25 mins.

Seven ester compounds were identified from Thai roasted chilli seasoning. Isobutyl 3-methylbut-3-enyl carbonate (22) showed the highest peak abundant followed by 4-methylpentyl 2-methylbutanoate (30) and this compound has been identified in fermented garlic extract with a high concentration under high pressure and temperature process Yusuf and Bewaji (2011). They presented fruity and sweet odour and could enrich the flavour composition of chilli. Generally, esters were produced by oxidation or pyrolysis of unsaturated fatty acids in peppers under high temperature, especially pericarp and seeds which could explain the increases of esters during the initial drying process (Ge *et al*., 2020). Although heating promotes the formation of esters, high drying temperature and long drying time would lead to the loss of esters compound.

3.3 Sensory attributes of Thai roasted chilli seasoning

In this study, roasted, burnt, fatty, garlic-like, onion-like, pungent, spicy and sweet were the main aroma attributes detected in all roasted chilli seasonings after training sessions. The data that were obtained were plotted in a spider diagram (Figure 3). The results showed that the intensity of roasted odour was not different in all samples, while the higher intensity of burnt odour was detected for 2 commercial samples. The combination of several aldehydes such as 2-methyl-Butanal, 3-methyl-Butanal and furfural from the Maillard reaction contributed to the roasted note. This could be interpreted as extreme values in roasted odour could be detected from the volatile flavour compounds obtained from severe roasting conditions. The developed seasoning exhibited the maximum intensity of the garlic and onion-like odour in comparison with commercial samples depending on the type and quantity of ingredients used. Prop-2-en-1-ol (allyl alcohol) has been proposed to be responsible for the garlic note (Chung *et al*., 2007). Allyl methyl trisulfide has been reported to contribute to garlic and onion odour in Thai fried chilli paste (nam phrik phao) (Rotsatchakul *et al*., 2008). In addition, pungent, spicy, sweet and fatty notes were found as distinctive notes for Thai roasted chilli seasoning. Acetic acid, mainly present in dried chilli,

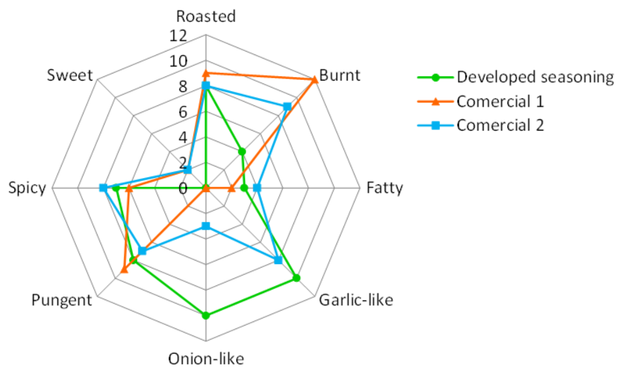

Figure 3. Sensory descriptive aroma profile comparison of roasted chili seasonings.

together with (E)-2-heptenal and 1-(2-furanyl)-Ethanone contributed to pungent note in the sample. Diallyl disulfide (sulfur-containing volatiles) has been reported to contribute to spicy note in garlic (Calvo-Gómez *et al.,* 2004), whereas several groups of volatile flavour compounds, i.e. alcohols, aldehydes, ketones and esters have been described as sweet odour.

4. Conclusion

Characteristic aroma compounds responsible for the scent of Thai roasted chilli seasoning were analysed by SPME followed by GC-MS. 49 volatile compounds were detected composed of 17 aldehydes and ketones, 8 sulfur -containing compounds, 7 esters, 6 alkanes, 5 miscellaneous compounds, 3 alcohols, 2 acids, and 1 terpene hydrocarbon compound. Acetic acid with a pungent aroma note showed the highest intensity in this seasoning, followed by prop-2-en-1-ol and diallyl disulfide contributing to garlic and spicy note respectively. These compounds played a key role in the scent of Thai roasted chilli seasoning. In addition, the final product in this study contained very low aflatoxin content where aerobic plate count, yeasts and moulds were not detected. The relationship between the aroma compositions of Thai roasted chilli seasoning to flavour attributes indicated that Strecker aldehydes are responsible for roasted odour. Acetic acid, prop-2-en-1-ol, diallyl disulfide and allyl methyl trisulfide responsible for pungent and spicy might be obtained from chilli, garlic and shallot.

Conflict of interest

The authors declare no conflict of interest.

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References

Apichartsrangkoon, A., Chaikham, P., Srisajjalertwaja, S., Chunthanom, P. and Dajanta, K. (2013). Aroma volatile profiles of thai green chili paste (nam prig noom) preserved by ultra-high pressure, pasteurization and sterilization. *International Food Research Journal*, 20(4), 1739–1746.

Bailey, M.E. (1994). Maillard reactions and meat flavour development. In Shahidi, F. (Eds.). Flavor of Meat and Meat Products. Boston, USA: Springer. https://doi.org/10.1007/978-1-4615-2177-8_9

BAM. (2001a). Bacteriological Analytical Manual Chapter 3: Aerobic Plate Count. In FDA Bacteriological Analytical Manual. Retrieved from FDA website: http://www.cfsan.fda.Gov/~ebam/bam-3.html

BAM. (2001b). Bacteriological Analytical Manual Chapter 18: Yeasts Molds and Microtoxins. In FDA Bacteriological Analytical Manual. Retrieved from FDA website: http://www.cfsan.fda. gov/ebam/bam-18.html

Block, E., Ahmad, S., Catalfamo, J.L., Jain, M.K. and Apitz-Castro, R. (1986). Antithrombotic organosulfur compounds from garlic: structural, mechanistic and synthetic studies. *Journal of the American Chemical Society,* 108(22), 7045–7055. https://doi.org/10.1021/ja00282a033

Brodnitz, M.H., Pascale, J.V. and Derslice, L.V. (1971). Flavor components of garlic extract. *Journal of Agricultural and Food Chemistry*, 19(2), 273–275. https://doi.org/10.1021/jf60174a007

Calvo-Gómez, O., Morales-López, J. and López, M.G. (2004). Solid-phase microextraction-gas chromatographic-mass spectrometric analysis of garlic oil obtained by hydrodistillation. *Journal of Chromatography A*, 1036(1), 91–93. https://doi.org/10.1016/j.chroma.2004.02.072

Caporaso, N., Paduano, A., Nicoletti, G. and Sacchi, R. (2013). Capsaicinoids, antioxidant activity, and volatile compounds in olive oil flavored with dried chili pepper (*Capsicum annuum*). *European Journal of Lipid Science and Technology*, 115(12), 1434–1442. https://doi.org/10.1002/ejlt.201300158

Chaethong, K. and Pongsawatmanit, R. (2015). Influence of sodium metabisulfite and citric acid in soaking process after blanching on quality and storage stability of dried chili. *Journal of Food Processing and Preservation*, 39(6), 2161–2170. https://doi.org/10.1111/jfpp.12460

Choi, J.H. and Kyung, K.H. (2005). Allyl alcohol is the sole antiyeast compound in heated garlic extract. *Journal of Food Science,* 70(6). 305 – 309. https://doi.org/10.1111/j.1365-2621.2005.tb11452.x

Chung, I., Kwon, S.H., Shim, S.T. and Kyung, K.H. (2007). Synergistic antiyeast activity of garlic oil and allyl alcohol derived from alliin in garlic. *Journal of Food Science*, 72(9), 437–440. https://doi.org/10.1111/j.1750-3841.2007.00545.x

Cremer, D.R. and Eichner, K. (2000). Formation of volatile compounds during heating of spice paprika (*Capsicum annuum*) powder. *Journal of Agricultural and Food Chemistry*, 48(6), 2454-2460. https://doi.org/10.1021/jf991375a

Cuicui, L. and Lixia, H. (2018). Review on volatile flavor components of roasted oilseeds and their products. *Grain and Oil Science and Technology*, 1 (4), 151-156. https://doi.org/10.3724/SP.J.1447.GOST.2018.18052

Fennema, R.O. (1996). Food Chemistry. 3rd ed. New York, USA: Mercel Dekker, Inc.

Ge, S., Chen, Y., Ding, S., Zhou, H., Jiang, L., Yi, Y., Deng, F. and Wang, R. (2020). Changes in volatile flavor compounds of peppers during hot air drying process based on headspace-gas chromatography-ion mobility spectrometry (HS-GC-IMS). *Journal of the Science of Food and Agriculture,* 100(7), 3087-3098. https://doi.org/10.1002/jsfa.10341

Hiraga, C., Mahakanjanakul, W., Sathonsaovapark, S. and Sittipod, S. (2004). Development of quality and safety standard for Nam Prik products under OTOP projects. Retrieved from website: https://kukr.lib.ku.ac.th/kukr_es/kukr/search_detail/dowload_digital_file/329866/64453 [In Thai].

Hye-Ryun, K., Sanjeev, K. D., Il-Doo, K. and In-Joo, P. (2016). Physicochemical and sensory characteristics of pepper oil sauce prepared from perilla oil. *African Journal of Food Science*, 10(12), 352–358. https://doi.org/10.5897/ajfs2016.1500

Jun, H.R., Cho, I.H., Choi, H.K. and Kim, Y.S. (2005). Comparison of volatile components in fresh and dried red peppers (*Capsicum annuum* L.). *Food Science and Biotechnology*, 14(3), 392-398.

Klieber, A. (2000). Chilli spice production in Australia: a report for the Rural Industries Research and Development Corporation. Barton, Australia: Rural Industries Research and Development Corporation.

Kyung, K.H., Kim, M.H., Park, M.S. and Kim, Y.S. (2002). Alliinase-independent inhibition of *Staphylococcus aureus* B33 by heated garlic. *Journal of Food Science,* 67(2), 780 – 785. https://doi.org/10.1111/j.1365-2621.2002.tb10676.x

Kyung, M.K. and Hyung, H.B. (2014). Aroma quality assessment of Korean fermented red pepper paste (gochujang) by aroma extract dilution analysis and headspace solid-phase microextraction-gas chromatography-olfactometry. *Food Chemistry*, 145, 488–495. https://doi.org/10.1016/j.foodchem.2013.08.087

Labuza, T.P., Heidelbaugh, N.D., Silver, M. and Karel, M. (1971). Oxidation at intermediate moisture contents. *Journal of the American Oil Chemists Society,* 48(2), 86–90. https://doi.org/10.1007/BF02635692

Mateo, J., Aguirrezabal, M., Domínguez, C. and Zumalacárregui, J.M. (1997). Volatile compounds in Spanish paprika. *Journal of Food Composition and Analysis*, 10(3), 225–232. https://doi.org/10.1006/jfca.1997.0535

Molina, C.M., Priego, C.F. and Luque de Castro, M.D. (2017). Headspace−GC–MS volatile profile of black garlic vs fresh garlic: Evolution along fermentation and behavior under heating. *LWT - Food Science and Technology*, 80, 98–105. https://doi.org/10.1016/j.lwt.2017.02.010

Morales, S.E., Kebede, B., Ugás, R., Grauwet, T., Van Loey, A. and Hendrickx, M. (2018). Flavor characterization of native Peruvian chili peppers through integrated aroma fingerprinting and pungency profiling. *Food Research International*, 109(2017), 250–259. https://doi.org/10.1016/j.foodres.2018.04.030

Muangkote, S., Vichitsoonthonkul, T., Srilaong, V., Wongs-Aree, C. and Photchanachai, S. (2018). Influence of roasting on chemical profile, antioxidant and antibacterial activities of dried chili. *Food Science and Biotechnology*, 28(2), 303–310. https://doi.org/10.1007/s10068-018-0475-1

Muriel, E., Atequera, T., Petrón, M.J., Andrés, A.I. and Ruiz, J. (2004). Volatile compounds in Iberian dry-cured loin. *Meat Science*, 68(3), 391–400. https://doi.org/10.1016/j.meatsci.2004.04.006

National Bureau of Agricultural Commodity and Food Standards. (2016). Food Consumption Data of Thailand. Retrieved from website: https://www.acfs.go.th/files/files/attach-files/867_20190606145951_625162.pdf

Park, Y. (2008). Moisture and Water Activity. In Nollet, L.M.L. and Toldra, F. (Eds). Handbook of Processed Meats and Poultry Analysis, p. 35–67. England: CRC Press. https://doi.org/10.1201/9781420045338.ch3

Rattanathawornkiti, J. (2010). Shelf-Life Evaluation of Fried Chili Paste and Chili Oil Using Q_{10} and Kinetic Model. Bangkok, Thailand: King Mongkut's University of Technology Thonburi, MSc. Dissertation.

Rotsatchakul, P., Chaiseri, S. and Cadwallader, K.R. (2008). Identification of characteristic aroma components of Thai fried chili paste. *Journal of Agricultural and Food Chemistry*, 56(2), 528–536. https://doi.org/10.1021/jf072499n

Samdaeng, W., Maireng, T., Pimpa, B. and Lekjing, S. (2015). Study on shelf life of common split gill mushroom (*Schizophyllum commune*) roast curry and chili paste products. *Burapha Science Journal*, 20(2), 33-47.

Schenker, S., Heinemann, C., Huber, M., Pompizzi, R., Perren, R. and Escher, F. (2002). Impact of Roasting

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Conditions on the Formation of Aroma Compounds in Coffee Beans. *Journal of Food Science*, 67(1), 60-66. https://doi.org/10.1111/j.1365-2621.2002.tb11359.x

Schnuggy. (2012), Namphrick pad for noodles. Retrieved on December 18, 2020. from Bloggang Website: http://www.bloggang.com/main.blog.php?id=schnuggy&month=07-08-2012&group=37&gblog=56

Singh, P., Amar, S., Singh, J. and Singh, S. (2014). Effect of Drying Characteristics of Garlic-A Review. *Journal of Food Processing and Technology*, 5(4), 1-6. https://doi.org/10.4172/2157-7110.1000318

Surasereewong, S., Sukchum, N. and Chaethong, K. (2021). Optimization of roasting conditions for Thai chili seasoning. *Journal of Current Science and Technology*, 11(2), 218-229.

Thai community product standard. (2013). Namphrik phat (Pub. L. No. 321–2013). Retrieved from http://www.fio.co.th/web/tisi_fio/fulltext/TIS429-2548.pdf

Toontom, N., Meenune, M., Posri, W. and Lertsiri, S. (2012). Effect of drying method on physical and chemical quality, hotness and volatile flavour characteristics of dried chilli. *International Food Research Journal*, 19(3), 1023–1031.

Vanderhaegen, B., Neven, H., Coghe, S., Verstrepen, K.J., Verachtert, H. and Derdelinckx, G. (2003). Evolution of chemical and sensory properties during aging of top-fermented beer. *Journal of Agricultural and Food Chemistry*, 51(23), 6782–6790. https://doi.org/10.1021/jf034631z

Wu, J.L., Chou, C.C., Chen, M.H. and Wu, C.M. (1982). Volatile flavor compounds from shallots. *Journal of Food Science*, 47(2), 606–608. https://doi.org/10.1111/j.1365-2621.1982.tb10133.x

Yusuf, O.K. and Bewaji, C.O. (2011). Evaluation of essential oils composition of methanolic Allium sativum extract on *Trypanosoma brucei* infected rats. *Research in Pharmaceutical Biotechnology,* 3(2), 17–21.

Zhang, N., Sun, B., Mao, X., Chen, H. and Zhang, Y. (2019). Flavor formation in frying process of green onion (*Allium fistulosum* L.) deep-fried oil. *Food Research International*, 121, 296–306. https://doi.org/10.1016/j.foodres.2019.03.006

