Effect of drying methods on Thai dried chilli's hotness and pungent odour characteristics and consumer liking

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Abstract

Dried chilli is widely used for its hotness and pungent odour enhancing properties. In an attempt to get a broad overview of drying effects on hotness and pungent odour characteristics contributing to dominant features of various types of Thai dried chilli, major volatile flavour compounds responsible for perceived hotness and pungent odour of the dried chilli-(freeze (FD), hot air (HD) and sun (SD) dried samples) were identified by trained panel (n=15) in conjunction with HS-SPME/GC-MS and LLE/GC-MS. Consumer liking of the three dried chilli was assessed on the basis of pungent odour and hotness characteristics by Thai consumers (n=120) using 9 point–category hedonic scale. The GC-MS and trained panel data were analyzed by PLS and results show that capsaicin was the main compound responsible for the oral hotness sensation, while 1-penten-3-one compound was found to be an indicator of strong pungent odour. The FD sample presented the highest intensity of hotness characteristic (p≤0.05). The content of 1-penten-3-one in FD was also higher than that of HD and SD. Although trained panellists could not differentiate between the intensities of pungent odour of FD and HD (p>0.05), consumers had a tendency to prefer HD. This research proposes that there is substantial value to analysis such as this that integrates human sensorial perception with objective measurement. The outputs of this research can be used to provide guidance on dried chilli product development based on consumer acceptance criteria.

Introduction

Chilli (*Capsicum* spp.) is a common spice in Thai cuisine and it is widely consumed as a food component throughout the world, particularly in South East Asia and Latin-American countries (Laohavechvanich *et al*., 2006). It is one of economic plants and involves with Thai society, especially with its daily cooking. Generally, it is mostly consumed in the form of dried powder or fine flake as a condiment (Turhan *et al*., 1997). Fresh chilli is normally preserved by drying immediately after harvest to obtain the dried chilli, to decrease overflow of fresh chilli in the market and also to control the market price (Charmongkolpradit *et al*., 2010). The most conventional drying method applied to chilli is sun drying. The duration of sun drying which is required to reduce moisture content in fresh chilli, depends on the quality of sunlight, temperature and air humidity. Hence the sun dried chilli present various moisture content levels between and within processing batches. In addition, they might be contaminated with dust, dirt, rainfall,

animals, birds, rodents, insects and microorganisms (Mangaraj *et al*., 2001). Alternatively, thermal drying method such as hot air drying has been identified as a more efficient drying method (Toontom *et al*., 2012). This is due to the fact that heat and mass transfer can be controlled during the drying process to achieve desired product quality (Tunde-Akintunde *et al*., 2014). The other option in producing dried chilli is using freeze drying. It is claimed to be the best process for retention dried chilli quality (Park and Kim, 2007), however it is also the most expensive process for drying (Ratti, 2001).

Certainly, hotness and volatile flavour compounds (i.e. perceived pungent odour) of dry chilli are important characteristics when applied dry chilli in food products (Lin and Durance, 1998; Szumny *et al*., 2010; Toontom *et al*., 2012). The hotness compounds such as capsaicin (Wang *et al*., 2009) and volatile flavour compounds such as pungent odour (i.e. 1-penten-3-one) (Luning *et al*., 1995) available in chilli have been found to be exposed to greater thermal and oxidative degradation, therefore; as far

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as dried chilli quality concerns, the decompositions of hotness and pungent odour compounds in dried chilli may influence on different sensorial property perceived by consumers and may not meet the requirement of the consumers in terms of flavour attributes. Therefore, a sensory profile in term of hotness and pungent odour attributes of dried chilli was observe as well as consumer acceptance.

With respect to the objective measurement of volatile flavour compounds, solid-phase microextraction (SPME) has been recommended for the quantitative analysis of flavour and fragrance compounds (Zhang *et al.*, 1994). However, only SPME may not success to get a good representation of the volatile compound in a sample. Many researchers recommended application of SPME extraction combined with liquid-liquid extraction (LLE) to obtain a more complete volatile flavour profile of samples (Kuwayama *et al.*, 2008; Domínguez and Agosin, 2010; Lee *et al.*, 2012). Since, extraction by SPME method mainly depends on the volatility and sorption to SPME fibre of the volatile flavour compounds and LLE depends on the pKa and solubility in extraction solvent. Therefore, combination of usage the both method can improve reliability of the profiling results (Lee *et al.*, 2012). The first objective of this work was to study the effects of drying methods; freeze, hot air and sun drying, on the volatile flavour compounds and sensory characteristics of dried chilli. The associations of instrumental parameters and the key sensory characteristics were statistically studied by Partial Least Square (PLS) regression method. Another objective was to determine consumer liking on hotness and pungent odour attributes of the chilli products after drying process.

Materials and Methods

Raw materials and chilli drying with different methods

Chee fah chilli (*Capsicum annuum* Linn. var. Acuminatum Fingerh.) was purchased from a contacted distributor from local market in Songkhla province, Thailand. Fully ripened chilli (70-90 days at the time of harvesting) with red colour of at least 75% of surface pod was used. The average diameter of pods was 1.5 ± 0.24 cm and an average length was 10.4 ± 0.98 cm. The raw material specifications in this research were based on Thai Agricultural Commodity and Food Standards of fresh chilli (TACFS 1502, 2004). The chilli was equally divided into 4 random batches; for the control sample (fresh chilli) and for processing batches of the three drying treatments. Fresh chilli was washed, removed the

stem and prepared to further analyses, immediately. In addition, the whole pod of fresh chilli was washed, removed the stem, blanched using hot water at 90°C for 3 minutes and then cooled in cold water and drained on a perforated tray. The chilli samples then were cut into the lengths of 2 cm and prepared for drying using three alternative methods; hot air drying (HD); freeze drying (FD); and sun drying (SD). Fresh chilli (F) without drying was used as a control. SD was processed by spreading blanched-cut chilli on a net in a single layer and exposed directly to sunlight (approximately 37°C). The thermometer was placed on an empty tray beside a net of chilli in order to record the range of drying temperature. The SD sample was dried for 8 hours per day for 8 days. For Hot air drying (HD), the blanched and cut chilli was placed on a perforated tray on an area of approximately 0.2 m² subjected to 60°C hot air dryer. Freeze-drying chilli was processed at -50°C, 5 Pa in a freeze dryer. All the dried chilli samples were taken out of the processes and packed when the moisture contents had dropped within a range of 10-13%, in accordance with the Thai Industrial Standards Institute (TISI 456, 1983). The individual final product from each drying treatment was packed together as a whole lot in aluminium laminated bags under vacuum condition and stored at -20°C. All chilli samples were freshly ground just before used in every experiment. Then, the samples were passed to sieve in order to get a typical 80-mesh size of chilli powder, in accordance with Thai Community Product Standard of ground chilli (TCPS 492, 2004).

Determination of capsaicin content

Ten grams of ground sample was placed in a 250 ml flask with 100 ml of acetone. The sample was stirred for 1 hour at room temperature. It was filtered by vacuum and the volume of the supernatant was reduced to approximately 5 ml by removing the excess acetone using nitrogen gas. The final solution was filtered through a 0.45 mm filter before injection to high performance liquid chromatography (HPLC). Ten microliters of extracted sample was injected for analysis by HPLC equipped with a Luna C18 column (5 μ, 250 × 4.6 cm) and a UV detector at 284 nm. The mobile phase used a mixture of methanol and water (80:20 v/v) and a flow rate of 1.5 ml/min (Betts, 1999). The capsaicin in each sample was identified and quantified by comparing it with capsaicin standard compounds (≥95.0%, from *Capsaicum* sp., Sigma, USA). A standard curve was prepared using serial dilutions of 0.15, 0.31, 0.63, 1.25, 2.50, 5.00, 10.00 and 20.00 mg/l capsaicin concentrations. The pungency level in SHU was calculated by the amount

of capsaicin (%dry weight) multiplies by 160,000 (Nwokem *et al.*, 2010; Toontom *et al.*, 2012). The measurements were taken in triplicate and results were averaged.

Volatile flavour compounds analysis

Liquid-liquid extraction (LLE)

Forty grams of ground sample were mixed with 200 ml of the solvent (distilled water and ethanol; 80:20 v/v), filtered by vacuum, centrifuged with 1500 rpm for 10 minute at 4ºC and removed the supernatant to use for determination. Then, clear solution was used for volatile flavour compounds isolation, immediately. The 100 ml clear solution was mixed with 100 ml solvent (dichloromethane and pentane; 1:2 v/v) using magnetic stirrer. The extracting condition was at room temperature and the mixture was extracted for 90 minutes and left to equilibrate for 30 minutes. Solvent phase was collected and sediment was re-extracted twice. The combined solvent phase was dried on sodium sulfate anhydrous, kept overnight at -20°C, cold- filtered and concentrated by purging of nitrogen gas. The concentrate was kept at -20°C prior to analysis (Adapted from Nabavi *et al.*, 2010; Ibarz *et al.*, 2006; Oliveira *et al.*, 2008; Genisheva and Oliveira, 2009). The 1 µl was directly injected into a gas chromatography-mass spectrometry (GC-MS).

Solid phase microextraction (SPME

Three grams of ground sample were adsorbed onto a solid phase microextraction (SPME; Supelco Inc., Bellefonte, PA) holder for GC analysis using the MS detector. Each SPME sampler consisted of a length of fused silica fibre absorption fibres coated with divinylbenzene, carboxen and polydimethylsiloxane (DVB/CAR/PDMS) 50/30 µm as a solid adsorbent. The sample was taken for sampling in a glass vial of 20 ml capacity and capped with a Teflon-lined septum and crimped. The vial was incubated at 40°C for 20 minutes in the heating block chamber before the introduction of fibre into the headspace vial. The volatile flavour was adsorbed at 40°C for 20 minutes and subsequently thermally desorbed at 220°C for 5 minutes in a GC injection port. Desorption time was optimized to ensuring there would be no carry-over effect to the next sampling (Toontom *et al.*, 2012).

Gas Chromatography–Mass Spectrometry (GC-MS):

Volatile flavour compounds were identified by Gas Chromatography-Mass Spectrometry (GC-MS) method using an Agilent 6890 plus GC/HP 5973 MSD (Agilent, USA). The carrier gas was helium at a flow rate of 1.5 ml/minute with a split ratio of 1:1 at 220°C. The separation of volatile flavour compounds was achieved on a fused silica capillary column (25 m x 0.32 mm i.d.) coated with cross-linked polyethylene glycol modified with nitroterephthalic acid as a stationary phase (20 M) at a film thickness of 0.50 µm (HP-FFAP; J&W Scientific, Folsom, CA). The oven was programmed as follows: 45°C for 2 minutes, ramped to 130°C at 3°C/minute and held for 1 minute; ramped to 220°C for 3 minutes at 20°C/minute; and then ramped to 230°C for 1 minute at the same rate. The mass selective detector capillary direct-interface temperature was 280°C. Acquisition was performed in the electronic impact (EI) mode. The mass range used was 20-550 a.m.u. and the acquisition rate was 4.33 scan/second. The identification was tentatively based on a comparison of the mass spectra of unknown compounds with those in the Wiley 275.L mass spectral database (Hewlett-Packard Co.) (Toontom, 2008).

Sensory evaluation

Fifteen participants were screened and recruited form PSU panel database, based on their sensory sensitivity and ability to describe sensations perceived from food products. A generic descriptive analysis method (Lawless and Heymann, 2010) was applied. Panellists undertook a 42-hour training programme. A ten-centimetre vertical Labelled Magnitude Scale (LMS) with six verbal descriptors, at following points of; barely detectable = 0.13 cm, weak = 0.59 cm, moderate = 1.64 cm, strong = 3.35 cm, very strong = 5.21, and strongest imaginable = 10 cm was developed and used in the training sessions to evaluate the sensory intensity (Adapted from Green *et al.*, 1993). After the training, the panel's performance was tested to determine reliability and validity on all attributes. If the panel performance was consistent and valid on the test samples (known intensities), then they would be qualified to start working on the sample evaluation. During the sample evaluation sessions, the panellists worked individually in separate booths and were instructed to independently rate samples by placing marks on the LMS to indicate the perceived intensity of the sensory attributes. The data obtained from individual panellists were transformed into logarithmic form and the attribute intensity mean scores were presented in geometric means. The temperature in the sensory evaluation room was controlled at 25ºC, and the room was free from distracting noises and odours. The pungent odour and hotness-related intensity were separately evaluated in different sessions. In pungent odour-related intensity evaluation, 2.5 g of each ground sample was served in a glass bottle. Each sample

was presented in an aluminium foil-covered glass bottle to mask any influences of colour and to control the transferring of any odorant. The panellists were asked to sniff and hold their breath for 3-5 seconds. They were required to clear their nasal cavities with soft tissue papers between samples (Cometto-Muñiz *et al*., 2000). To evaluate the hotness -related intensity, 2.5 g of each ground sample was served in a plastic cup. All samples were presented in the booths with red light to mask any colour interference. The panellists were asked to rate the hotness intensities perceived via mouth. After testing each sample, the panellists were required to rinse their mouths once with sucrose solution (10% sucrose w/w in water) (Nasrawi and Pangborn, 1990) and 5 times with water and then wait for 5 minutes before evaluating the next sample. An inter-stimulus interval of 5 minutes was allowed to permit any residual to subside (Lawless *et al*., 2000; Allison *et al*., 1999).

Consumer liking

One hundred and twenty chilli consumers of whom 82 females and 38 males, age between 19 to 35 years old, were recruited. The samples were individually served one at a time with balance first-order and carry-over-effect design (MacFie *et al*., 1989). The 3-digit random codes and red masking light in sensory booths were applied during the evaluation to reduce interference effects. Two and a half grams of the three samples, i.e. FD, HD and SD were presented in clear plastic containers to all consumers. Consumers were asked to taste 2.5 g per sample and to rate their liking on pungent odour and hotness attributes of samples after testing on 9-point category hedonic scale. The procedure to rinse their mouths, cleanse their noses and time breaks between samples were similar to the test protocol that the trained panellists had applied.

Statistical analysis

A Completely Randomized Design (CRD) was planned for the HPLC determination of capsaicin contents. The experimental designs of Descriptive Analysis by trained panellists and consumer liking test were Randomized Complete Block Design (RCBD). Data were subjected to analysis of variance (ANOVA) and Post-hoc Duncan's New Multiple Range Test (DMRT), with a level of significance of 0.05. Statistical analyses were performed using the Statistical Package for Social Science (SPSS 11.0 for windows, SPSS Inc., Chicago, IL, USA, 2002). Partial Least Squares (PLS) was applied to analyse both data sets derived from the instrumental measurements (as predicting variables) and from the trained panellists data (as dependent variables) to reveal the relationships between subjective and objective measurements using XLSTAT software (XLSTAT Pro 2008).

Results and Discussion

Effect of drying on capsaicin content

The capsaicin contents of all samples which were calculated based on dry weight basis are shown in Table 1. It was found that the drying method did not affect the capsaicin content (p>0.05). Furthermore, the capsaicin content and hotness of dried chilli from all the drying methods were higher than in the fresh chilli sample (p≤0.05). The capsaicin content of fresh chilli (as a raw material and a control sample) used in this research was of similar range of fresh Chee fah chilli (0.52-1.07 mg/g; 7,500-16,500 SHU) reported elsewhere (Kaewprasit and Kumngern, 2009; Noichinda *et al*., 2012). Interestingly though based on dry weight basis calculation, the fresh chilli contained lesser amount of capsaicin than the three dried chilli samples. This can be explained in relation to the activity of peroxidase enzyme and temperature during blanching and drying processes. Since, vanillyl moiety of capsaicin was easily oxidized by the peroxidise enzyme and the enzyme could contribute to capsaicin degradation (Bernal *et al*., 1993a; Bernal *et al*., 1993b; Diaz *et al*., 2004). The dried chilli samples were blanched before drying, hence the peroxidise enzyme was inactivated, whereas the enzyme was still active in case of the fresh chilli. Other possible explanation for an increase of capsaicin content in dried chilli is the hydrolysis of glycosides in the fruit from blanching and drying heat. During fruit development, not only free capsaicin can be formed, but free capsaicin can be bound with sugars and other compounds via hydroxyl group in capsaicin structure (Diaz *et al*., 2004). The glycosdic linkages in bound capsaicin can be destroyed by blanching and drying conditions, hence increase free capsaicin compounds to be available in such the system. In addition, a dehydrated condition of food samples might improve extractability of capsaicin by cell disruption during the solvent extracting process (Harrison and Harris, 1985; Lee and Howard, 1999; Schweiggert *et al*., 2006). The capsaicin contents of dried chilli found in this research varied between 0.98 and 1.29 mg/g (p>0.05) and is of similar range reported by Margied *et al*. (2014) and Yaldiza *et al*. (2010).

Effect of drying on volatile flavour compounds

The volatile fractions of fresh and dried chilli extracted using SPME and LLE techniques are

Table 1. Capsaicin content, hotness level and mean liking scores of pungent odour and hotness using 9-point hedonic scale in fresh and dried chilli from different drying methods

Samples	Capsaicin contents	Hotness levels	Liking scores	
			Pungent odour	Hotness
F	0.58 ± 0.10^b	9280.84 ± 705.71^b	ND	ND
FD	1.29 ± 0.20^a	20640.54 ± 743.87^a	5.65 ± 1.76^a	5.63 ± 1.93^a
HD	1.17 ± 0.20^a	18720.20 ± 335.46^a	5.89 ± 1.79^a	5.74 ± 1.69^a
SD	0.98 ± 0.21^a	15680.79 ± 650.60^a	4.32 ± 1.65^b	5.15 ± 1.78^a

Note: Different superscripts within a column show significant difference ($p \leq 0.05$).
ND refers to not determined.

shown in Table 2. From a qualitative viewpoint, volatile compounds namely; acids, alcohols, ketones, aldehydes, esters, pyrroles, sulphur and hydrocarbon were detected using LLE and SPME.

In general, the number of components obtained by the SPME was lower than those extracted by LLE, none the less, higher amount of several alcohol, aldehyde and hydrocarbon compounds were found in SPME profiles. This perhaps due to the intermediate polarity of divinylbenzene/carboxen/ polydimethylsiloxane (CAR/DVB/PDMS) coating (Junior *et al.*, 2011). Whereas, more compounds of acids, ketone and hydrocarbon were presented in the LLE profiles. In addition, 1-penten-3-one (pungent odour) was only analysed and detected by LLE/GC-MS. The result indicates that the SPME is not able to extract the volatile flavour component 1-penten-3-one (1P3O) which is a principle of pungent odour compound found in chilli. Since 1P3O has low volatility, then it solubilises more in the mixture of organic solvent between dichloromethane and pentane. The mixed solvent has high extraction efficiency for a wide range of non-polar to polar compounds (Martín-del-Campo *et al.*, 2011; Popescu *et al.*, 2011). The discrepancy of LLE and SPME can be explained by the fact that compounds detected by SPME are mainly related to their volatility, while with LLE, the extraction of compounds mostly relies on its solubility in the organic phase (Lee *et al.*, 2012). However, both techniques are able to detect compounds that are known to be dominant in chilli.

The volatile flavours affected by drying can be distinguished in two groups of the decreased or disappeared compounds and the increased or formed compounds. There were two compounds which found and detected after the three drying processes, namely 5-methyl-undecane and 2,3-dihydro-3,5-dihydroxy-6-methyl-4(4H)-pyranone, corresponding to herbal and caramel odours (Acree and Arn, 2004; Almonds, 2009). The 1P3O (pungent odour) was a volatile compound which decreased after drying. Whereas, acetic acid was mainly presented in all samples and increased after drying, particularly in the SD sample. The volatile flavour compounds which completely disappeared after drying were cyclobutylbenzene

(sweet odour), 4-vinyl-2-methoxy-phenol (clove-liked odour), phenol (acrid or tar-like odour), 2-methoxy-phenol (smoky odour), n-hexyl acetate (herbal odour), hexadecanal (cardboard-liked odour), 2-docecen-1-al (woody odour), 2-pentyl-furan (green-liked odour), hexanal (leafy odour), hexanol (herbal odour) and hexadecanoic acid (waxy odour). The volatile flavour compounds found only in the FD sample were 1,6-dimethyl-naphthalene (woody odour), hexadecane (mild waxy odour), 5-ethyl-undecane (herbal odour), 2-furanmethanol-acetate (horseradish-like odour), phenylethyl alcohol (fresh odour), 2-ethyl-1-hexanol (citrus-like odour), 5-methyl-2-(1-methyl) cyclohexanol (camphoraceous-liked odour), 2-octanol (spicy odour), and 2-methyl-butanoic acid (cheesy odour). Some volatile flavour compounds were also only found in HD sample. These were 1-methoxy-4-(1-propenyl)-benzene (aniseed-like odour), alpha-gurjunene (woody odour), undecane (herbal odour), 2-acetyl furan (balsamic-like odour), benzeneacetaldehyde (flora-like odour), 1,3-cyclohexadiene-1-carboxaldehyde (herbal odour), 5-methylfurfural (spicy odour), benzaldehyde (flora-like odour) and 2-methyl-butyric acid (cheesy odour). On the other hand, it can be concluded that the sun drying process decomposed highly volatile flavour compounds. The compounds found in SD sample were 2-methyl-tridecane (mild waxy odour), 2,3-butanediol (onion-like odour), dihydro-2(3H)-furanone (creamy odour), 1-methyl-1H-pyrrole (herbal odour), tetramethylpyrazine (nutty odour) and 2-methyl-propanoic acid (cheesy odour). It is possible that 2-methylpropionic and 2-methylbutyric acid were present because of Strecker degradation as they were found to form upon further oxidation during drying (Luning *et al.*, 1995). The formation of volatile flavour compounds, namely 2-acetyl pyrrole and furfural, were only detected in the FD and HD samples due to the Maillard reaction.

Effect of drying on sensory characteristics

Eight sensory attributes of fresh and dried chilli which were developed and agreed by all trained panellists are shown in Table 3. The sensory lexicon

Table 2. Volatile flavour compounds and their relevant attributes indentified in fresh and dried chilli samples from different drying methods

RT[A]	RI[B]	Volatile flavour compound	Attributes[C]	LLE/GC-MS				SPME/GC-MS			
				F	FD	HD	SD	F	FD	HD	SD
		Acids									
2.78	1081	2-Methyl-butanoic acid	Cheesy	nd	nd	nd	0.42	nd	1.63	nd	0.35
17.80	1596	Acetic acid	Vinegar-like	nd	nd	nd	1.32	0.02	4.72	2.37	10.71
20.93	1127	2-Methyl-propanoic acid	Cheesy	nd	nd	nd	2.44	nd	nd	nd	2.44
24.80	1662	2-Methyl-butyric acid	Cheesy	nd	nd	0.63	nd	nd	nd	0.54	nd
23.73	1652	Propanoic acid	Rancid, Sour	0.30	nd	nd	nd	nd	nd	nd	nd
39.25	2145	Octadecanoic acid	Mild fatty	nd	1.66	nd	nd	nd	nd	nd	nd
40.32	2672	Tetradecanoic acid	Coconut	nd	nd	nd	2.76	nd	nd	nd	nd
40.99	2740	Pentadecanoic acid	Waxy	nd	nd	nd	3.91	nd	nd	nd	nd
42.20	3160	9,12-Octadecadienoic acid	Rancid	0.84	34.64	nd	9.13	nd	nd	nd	nd
43.45	2931	n-Hexadecanoic acid	Waxy	0.64	1.40	1.90	28.85	0.08	nd	nd	nd
44.16	3157	9-Octadecenoic acid	Faint fatty	nd	6.11	nd	2.68	nd	nd	nd	nd
		Alcohols									
17.97	1600	2-Ethyl-1-hexanol	Citrus	nd	nd	nd	nd	nd	0.26	nd	nd
10.14	1396	2-Octanol	Spicy	nd	nd	nd	nd	nd	0.2	nd	nd
12.09	1448	Hexanol	Herbal	nd	nd	nd	nd	1.12	nd	nd	nd
20.26	1621	Linalool	Floral	0.37	nd	0.27	nd	0.41	nd	0.03	nd
23.45	1649	5-Methyl-2-(1-methyl) cyclohexanol	Camphor	nd	nd	nd	nd	nd	0.01	nd	nd
31.62	1778	2-Methoxy-phenol	Smoky	0.10	nd	nd	nd	0.04	nd	nd	nd
32.64	1821	2,6-Bis(1,1-dimethylethyl)-4-methylphenol	Camphor	0.13	nd	nd	nd	0.04	0.19	nd	nd
32.07	1781	Benzenemethanol	Sweet and fruity	0.53	nd	0.30	nd	0.59	nd	0.24	nd
33.35	1870	Phenylethyl alcohol	Floral odour	nd	0.16	nd	3.95	nd	0.39	nd	nd
34.74	2008	1-Hexadecanol	Waxy floral	nd	nd	nd	2.31	nd	nd	nd	nd
		Ketones									
3.74	1167	1-Penten-3-one	Spicy, pungent	42.93	38.18	37.57	0.90	nd	nd	nd	nd
11.15	1424	3-Hydroxy-2-butanone	Creamy	nd	nd	nd	1.53	0.16	nd	0.17	1.00
23.03	1646	Dihydro-2(3H)-furanone	Creamy	nd	nd	nd	0.63	nd	nd	nd	1.43
25.32	1666	4-Ketoisophorone	Musty, woody, sweet	nd	0.05	nd	nd	nd	nd	nd	nd
32.98	1844	Beta-ionone	Sweet	0.10	0.08	nd	nd	0.11	0.09	nd	nd
35.96	2198	2,3-Dihydro-3,5-dihydroxy-6-methyl-4(4H)-pyranone	Caramel	nd	0.65	nd	0.92	nd	0.71	0.23	0.89
		Furans									
7.90	1330	2-Pentyl-furan	Green	nd	nd	nd	nd	0.42	nd	nd	nd
18.72	1607	2-Acetyl furan	Balsamic	nd	nd	nd	nd	nd	nd	0.22	nd
5.00	1230	Isoamylacetate	Banana-like	1.18	1.33	0.58	nd	2.41	2.52	1.99	nd
		Esters									
9.46	1376	n-Hexyl acetate	Herbal	0.06	nd	nd	nd	0.07	nd	nd	nd
19.95	1618	2-Furanmethanol-acetate	Horseradish	nd	nd	nd	nd	nd	0.04	nd	nd
28.37	1694	2-Hydroxybenzoic acid methyl ester	Wintergreen like	nd	nd	3.18	nd	0.16	0.36	0.1	nd
37.36	1891	Ethyl linoleolate	Faint odour	nd	nd	nd	1.58	nd	nd	nd	nd
40.48	1962	Dibutyl phthalate	Faintly fruity	nd	nd	0.77	1.81	nd	nd	nd	nd
		Pyrroles									
5.48	1248	1-Methyl-1H-pyrrole	Herbal	nd	nd	nd	nd	nd	nd	nd	1.27
33.62	1889	2-Acetylpyrrole	Licorice-like	nd	0.44	0.27	nd	nd	0.47	0.28	nd
		Aldehydes									
2.83	1095	Hexanal	Leafy	nd	nd	nd	nd	0.03	nd	nd	nd
15.46	1536	2-Docecen-1-al	Fatty	nd	nd	nd	nd	0.04	nd	nd	nd
17.26	1583	Furfural	Almond	nd	nd	nd	nd	nd	0.26	3.73	nd
19.25	1612	Benzaldehyde	Almond	nd	nd	nd	nd	nd	nd	0.04	nd
21.41	1631	5-Methylfurfural	Caramel	nd	nd	0.31	nd	nd	nd	0.01	nd
23.43	1649	1, 3-Cyclohexadiene-1-carboxaldehyde	Herbal	nd	nd	0.26	nd	nd	nd	0.13	nd
23.62	1651	Benzeneacetaldehyde	Flora	nd	0.03	nd	nd	nd	nd	0.04	nd
34.11	1937	Hexadecanal	Cardboard	nd	nd	nd	nd	0.51	nd	nd	nd
43.78	1119	3-Chloro-benzaldehyde	Pungent	nd	nd	4.78	nd	nd	nd	nd	nd
		Sulfur containing compounds									
22.76	792	2,3-Butanediol	Onion	nd	nd	nd	1.21	nd	nd	nd	7.40
12.26	1453	2-Methyl-tridecane	Mild waxy	nd	nd	nd	1.78	nd	nd	nd	1.78
35.85	2180	1,2-Diiodo-ethane	Faint ether-like	1.48	nd	nd	nd	nd	nd	nd	nd
33.38	1872	1,6-Dimethyl-naphthalene	Woody	nd	nd	nd	nd	nd	0.04	nd	nd
		Hydrocarbon compounds									
30.85	1754	1-Methoxy-4-(1-propenyl)-benzene	Aniseed	nd	nd	nd	nd	nd	nd	0.03	nd
39.21	1789	1-Octadecene		0.11	nd	nd	1.70	nd	nd	nd	nd
23.76	1652	4,7,10-Cycloundecatriene	Woody	nd	0.04	nd	nd	nd	nd	nd	nd
4.06	1191	5-Ethyl-undecane	Herbal	nd	nd	nd	nd	nd	0.33	nd	nd
3.61	1157	5-Methyl-undecane	Herbal	nd	nd	nd	0.44	nd	2.6	0.06	0.34
18.55	1605	Alpha-gurjunene	Woody	nd	nd	0.40	nd	nd	nd	0.41	nd
21.07	1628	Beta-caryophyllene	Spicy	nd	1.86	nd	nd	nd	2.01	1.13	nd
29.84	1723	Cyclobutylbenzene	Sweet	nd	nd	nd	nd	0.05	nd	nd	nd
4.27	1203	Dodecane	Woody	nd	0.96	nd	nd	nd	0.17	0.92	nd
3.30	1134	Heneicosane	Waxy	nd	0.06	nd	nd	nd	nd	nd	nd
21.73	1634	Hexadecane	Mild waxy	nd	0.2	nd	nd	nd	0.11	nd	nd
17.26	1583	Tetramethylpyrazine	Nutty	nd	nd	nd	nd	nd	nd	nd	3.56
30.35	1839	Trans-anethole	Herbal	0.10	nd	nd	nd	0.11	nd	nd	nd
10.22	1399	Tridecane	Mild waxy	nd	0.71	nd	nd	nd	0.79	0.09	nd
40.28	1100	Undecane	Herbal	nd	nd	2.50	nd	nd	nd	0.21	nd

Note: LLE/GC-MS refers to liquid-liquid extraction with a gas chromatography-mass spectrometry; SPME/GC-MS refers to solid phase microextraction with a gas chromatography-mass spectrometry; RT[A] refers to retention time (min); RI[B] refers to retention index that was based on a series of alkane (C8-C40); nd refers to not detected; [C] Reference: *http://www.flavournet.org/flavournet.html*

Table 3. Definitions and references of fresh and dried chilli's sensory attributes

Sensory attributes	Definition	References
1. Burnt chilli odour	Degree of burnt odour characteristic which is similar to dried chilli	Pure water = 0 (odour) 2.5 g ground dried chilli (80°C, 10 min) = 1 (odour)
2. Fresh chilli odour	Degree of fresh chilli odour which is similar to green odour of fresh chilli and no fermented odour	Vinegar = 0 (odour) 2.5 g ground fresh chilli = 0.94 (odour)
3. Raise-to-nasal pungent odour	Degree of chilli pungent odour characteristic which irritates upper of nose	Pure water = 0 (pungent odour) 2.04 µl/l 1P3O = 0.97 (pungent odour)
4. Sting-pungent odour	Degree of chilli pungent odour characteristic which induces to nasal sting	Pure water = 0 (pungent odour) 2.04 µl/l 1P3O = 0.67 (pungent odour)
5. Warm in mouth	Degree of warm sensation in mouth during keep a sample in the mouth	Pure water = 0 (hotness) 15 mg/l capsaicin = 0.90 (hotness)
6. Oral burn	Degree of burn sensation in mouth during keep sample in mouth	Pure water = 0 (hotness) 15 mg/l capsaicin = 0.70 (hotness)
7. Oral sting	Degree of sting sensation in mouth during keep sample in mouth	Pure water = 0 (hotness) 15 mg/l capsaicin = 0.67 (hotness)
8. Tongue numb	Numbing sensation on tongue	Pure water = 0 (hotness) 15 mg/l capsaicin = 0.82 (hotness)

includes attribute names, agreed definitions and reference samples that illustrated dimensional consensus meaning of each attribute. The trained panel identified two attributes, namely fresh (FO) and burnt (BO)-chilli odour as generic attributes perceived from all dried chilli samples. They described the dried chilli samples' pungent odour characteristic as raise-to-nasal (RNO) and sting (SPO) -pungent odour. Whereas, warm in mouth (WM), oral burn (OB), oral sting (OS) and tongue numb (TN) were related to the hotness characteristics of dried chilli samples. The attribute means from the trained panel of each sample are presented in Figure 1. The fresh and dried chilli samples were differentiated by all attributes. The fresh sample was perceived to have highest FO intensity, while sun dried chilli sample contained the least FO intensity ($p \leq 0.05$). It is possible that the fermentation process was caused by contaminated microorganisms during drying by being exposed to the sun in open air (Mangaraj *et al.*, 2001; Topuz *et al.*, 2004), hence fermented odours could occur in SD sample. The HD sample showed similar intensity of raise-to-nasal and sting-pungent odour attributes to the FD sample. It can be proposed here that there is similar profile of pungent odour-volatile compounds in the HD and FD samples. The compounds are 1-penten-3-one (pungent odour), β-caryophyllene (spicy odour), 2-octanol (spicy odour) and 3-chloro-benzaldehyde (pungent odour) which were identified by GC-MS. HD sample contained higher intensity of burnt chilli odour (BO) than the FD and SD samples,
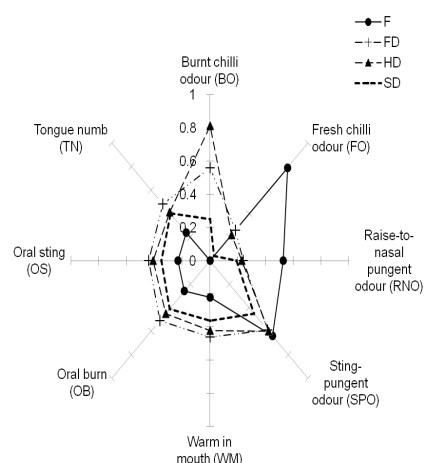

Figure 1. Hotness and pungent odour profile analysed by the trained panel

respectively. The formation of 2-acetyl furan (licorice odour), furfural (almond odour) and 5-methylfurfural (caramel odour) which generated during hot-air drying, might be responsible for the burnt attribute. These compounds are found in chilli samples produced by high temperature process, inducing a Maillard reaction (Apriyantono and Ames, 1993; Elmore *et al.*, 2009). Whereas, the freeze drying is the best drying method for avoiding chemical changes caused by heat, it produces a product with superior physical and chemical qualities of products (Park and Kim, 2007; Antal *et al.*, 2011). Therefore, the fresh chilli odour presented in FD sample was higher than other dried samples. In terms of hotness related-

attributes, FD sample also had the highest intensity in all hotness related-attributes when compared with other dried chilli samples (p≤0.05). These findings are in agreement with the results from HPLC which the FD sample was found to have the highest capsaicin content (1.29 mg/g; 20,640.54 SHU), then followed by HD sample (1.17 mg/g; 18,720.20 SHU), SD sample (0.98 mg/g; 15,680.79 SHU) (p>0.05). The research then concludes that the perceived hotness in samples mainly depends on the capsaicin content.

Linkages between subjective and objective measurements

The PLS was performed on the 20 volatile compounds derived from the instruments with regard to the intensity of pungent odour and hotness-characteristics, namely FO, BO, RNO, SPO, WM, OB, OS and TN. The volatile flavour compounds obtained from SPME method are correlated well with the sensory attributes perceived from the human nose. The responses from both measurements were then analysed by PLS statistical method in order to predict the relationships between the two sets of variables. In this case, the pungent and hotness characteristics are Y-variables (dependent variables) and volatile flavour compounds are X-variables (predictors). The connection of sensory descriptors and volatile compounds in chilli samples testifies the existence of a relationship between variables. The PLS results as demonstrated in Figure 2 show that the FO and RNO attributes can be predicted by hexanol, benzenemethanol, 2-pentyl-furan, n-hexylacetate, hexanal and trans-anethole ($r^2 > 0.9$). Acree and Arn (2004) and Almonds (2009) supported the notion that hexanol, n-hexylacetate and trans-anethole contributed to herbal odour, while benzenemethanol contributed to fruity odour. Hexanal was also reported to give a glassy, leafy odour which was found in fresh chilli (*C. annuum*) (Mazida *et al.*, 2005). In addition, 2-pentyl-furan was found to contribute to a green odour in fresh chilli and was significantly decreased after cooking (Srisajjalertwaja *et al.*, 2012). The SPO attribute presents highly positive correlation ($r^2 = 0.996$) with 1-penten-3-one which has been described as a pungent odour of fresh chilli (Mazida *et al.*, 2005; Azcarate and Barringer, 2010; Elmore et al., 2009). However, it can be seen from this research that the SPO attribute is negatively associated with acetic acid (vinegar-like odour) ($r^2 = -0.996$). The equation model explaining the relationship between BO and volatile compounds shows moderate goodness of fit statistics ($r^2 = 0.655$). However, it is worth noting that BO attributes are related to 1,3-cyclohexadiene-1-carboxaldehyde, undecane
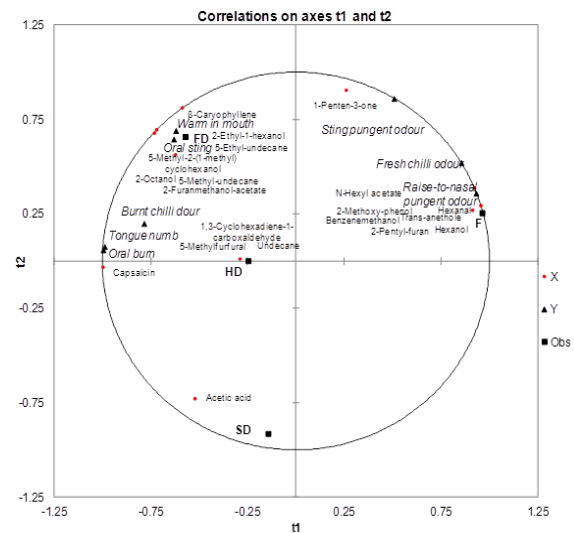

Figure 2. PLS plot of sensory attributes and flavour compounds in fresh and dried chilli samples

and 5-methylfurfural. These compounds are known of their effects contributing caramel and herbal odour in dried chilli. The capsaicin which gives oral hotness sensation (Bosland, 1996; Walsh and Hoot, 2001; Margied *et al.*, 2014) presents high association with the OB ($r^2 = 0.982$) and TN ($r^2 = 0.995$) attributes. The WM and OS attributes show positive relations with β-caryophyllene (spicy odour) ($r^2 = 0.861$ and $r^2 = 0.814$).

Effect of drying on consumer liking

The mean scores of the liking on pungent odour and hotness in the three dried chilli samples are reported in Table 1. Significant differences of liking between samples are detected in all attributes (p≤0.05). Hotness liking, however, shows no significant differences among the three samples with similar range of hotness levels (SD = 0.98 mg/g, 15,680.79 SHU; HD = 1.17 mg/g, 18,720.20 SHU; FD = 1.29 mg/g, 20,640.54 SHU). The consumers disliked pungent odour attribute of SD which could cause by the least fresh chilli odour remained in SD.

In general, SD was the least liked sample on most attributes while HD received the highest liking scores on most attributes, though is not significantly different from FD (p>0.05). The reason why the consumers showed a tendency of liking in all attributes of HD, may relate to familiarity in the dried chilli flavour. Most of dried chilli products available in Thai retailers are produced by conventional hot air drying. Heath *et al.* (2011) stated that the familiarity with taste, odour and flavour of food play role in consumers' liking. Augustyn *et al.* (2010) also reported that increased familiarity results in increased liking, and foods typically must be introduced 10 times before participants accept them.

It is a well-established psychological phenomenon that people express liking for things merely because they are familiar with them (Bornstein and Crave-Lemley, 2004). A positive experience with a product also leads to increased favourability. It is an intuitive finding that people take into consideration things that they used to like or things that they have come to like over time (Hoeffler *et al.*, 2013).

Conclusion

Drying methods influenced on volatile flavour compounds of Chee fah chilli. However, the sun, hot air and freeze drying methods did not affect the capsaicin concentration in all dried chilli. The extraction techniques, LLE and SPME, were able to differentiate and identified the varieties of volatile flavour compounds from Thai dried chilli. The groups of volatile flavour compounds, acids, ketones, pyrroles, furans and aldehydes, dominated sensory characteristics of the dried chilli. These compounds were likely to be the key volatile flavour characteristic of the dried chilli samples. One-penten-3-one was a main pungent odour compound in chilli samples which was only detected by GC-MS. The dominant volatile flavours, such as acetic acid and 2-methylpropionic, were found in SD sample. 2-Acetylpyrrole and furfural were main volatile flavours finding in HD and FD samples. Regarding to the sample hotness and pungent odour profiles, the FD sample contained higher intensity of fresh chilli odour and all hotness attributes than other dried samples. Sun dried chilli was perceived the least fresh chilli odour. The research findings derived from sensory descriptive analysis are in agreement with the instrumental results. The 1-penten-3-one and capsaicin are the most dominant compounds to sensorial quality in chilli samples. Although the intense hotness was presented in FD sample, Thai consumers had given the highest liking scores, on hotness and pungent odour in the HD sample. This finding was discussed in relation to familiarity and positive experience with the sample among Thai chilli consumers.

Acknowledgements

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