International Journal of Agricultural Technology 2021Vol. 17(4):1577-1590
Available online http://www.ijat-aatsea.com
ISSN 2630-0192 (Online)

Effects of drying methods on active odorants, phytochemicals and antioxidant properties of *Litsea petiolata Hook*. f. leaves locally used as a substitute to male giant water bugs in pungent chili pastes

Sangsila, A.[1*], Chumroenphat, T.[2] and Jorjong, S.[3]

[1]Division of General Science, Faculty of Education, Buriram Rajabhat University, Buriram 31000, Thailand; [2]Laboratory Equipment Center, Division of Research Facilitation and Dissemination, Mahasarakham University, Mahasarakham 44150, Thailand; [3]Faculty of Natural Resources, Rajamangala University of Technology Isan, Sakon Nakhon Campus, Sakon Nakhon 47160, Thailand.

Sangsila, A., Chumroenphat, T. and Jorjong, S. (2021). Effects of drying methods on active odorants, phytochemicals and antioxidant properties of *Litsea petiolata* Hook. f. leaves locally used as a substitute to male giant water bugs in pungent chili pastes. International Journal of Agricultural Technology 17(4):1577-1590.

Abstract The plant species *Litsea petiolata Hook. f.* has been traditionally used as a flavouring ingredient in chili paste as a substitute to male giant water bugs in many parts of Thailand and some Southeast Asian countries, due to the male giant water bug-like fragrance of its various parts. However, the knowledge about its odor, phytochemical quality, and antioxidant activity of this plant has not been known widely. The current study compared the odorant profiles between male giant water bugs and dried leaves of *L. Petiolata* and compared the phytochemicals and antioxidant properties of the leaves from three different drying methods. Nine odorants were common in male giant water bugs and dried leaf samples, with undec-10-en-2-one and undecanone detected at high levels, thus serving as important odorant contributors. The best efficient way among the three methods to keep the odorants, phenolic acids (chologenic, gallic, rutin and ferulic acid), flavonoids (catechin hydrate, *p-coumaric* acid and myracetin), and vitamin C was drying in the frozen temperatures. Moreover, the leaves dried by the cool method contained high amount of phenolic acids, flavonoid, and the quantity of antioxidant, as compared to other drying methods. The research results revealed that *L. petiolata*'s leaves were rich in phytochemicals and antioxidant capacities which provided benefits to people. Moreover, It is elucidated the contribution for drying processes, and the stability of other qualifications of the plants, such as the biological components and the number of antioxidant.

Keywords: Antioxidants, Drying methods, Odorants, Phytochemicals

Introduction

Litsea spp. is an evergreen tree under *Lauraceae* and there are about 200-400 kinds of these trees growing plentifully in the torrid zones of Asia and

[*] **Corresponding Author**: Sangsila, A.; **Email:** sangsi.a19@gmail.com, Arunrussamee.ss@bru.ac.th

America continents (Wang *et al*., 2016). Many species have been widely used for medicinal purposes and have been used as a spice in cooking too (Kong *et al*., 2015; Yang *et al*., 2014). In Thailand, 35 types of this plant were recorded and most of them are used as vegetables, flavouring agents, and folk medicines (Ngernsaengsaruay *et al*., 2011). The species *Litsea petiolata* Hook. f, locally called 'Thammung', which is native to Malaysia and the southern region of Thailand and have been traditionally used as a flavouring ingredient in chili pastes across the regions of Thailand, especially in the northeastern area, due to the male giant water bug-like fragrance of its various parts (Panyamongkol, 1994; Pimpaka *et al*., 2007).

For the bugs, their families are *Lethocerus indicus* Lep. and Serv. They were mostly found in Southeast Asia, as in Thailand, Laos, Vietnam, and Cambodia. These bugs are one of the most popular edible insects consumed in almost all parts of Thailand, particularly the northern and northeastern regions (Hanboonsong *et al*., 2013). Thai people like to consume it because of the distinguished and desirable odor. They smashed the bugs together with their chili paste to make them smell good and tasty; and the bugs are also put in other food (Wishram *et al*., 2013). Nowadays, male giant water bugs are rapidly declined in number as a result of intensive capture, habitat decline, and water pollution (Kiatbenjakul *et al*., 2015). Most of the insects are now imported from neighboring countries, such as Laos and Cambodia, making the insects more expensive. Hence, local people are currently utilizing Thammung leaves as an alternative source of this strong and desirable odor that can provide a sustainable substitute to the male giant water bugs.

Most Thais believe that their local plants such as herbs, vegetables, fruits are their healthy food because they are sources of antioxidants. The parts of these plants, including leaf, root, rhizome, flower, fruit, seed, and bark serve as local medicine like antioxidants (Balange and Benjakul, 2009). Herbs generally have their distinct taste and aroma. These properties are both volatile and nonvolatile (Longo and Sanroman, 2010). *Litsea* species are one of plants variety that provide of volatile and nonvolatile properties such as flavonoids, monoterpenes, alkaloids, lignans and fatty acids (Agrawal *et al*., 2011). Nevertheless, information about the volatile and nonvolatile compounds of *L. petiolata* is limited. The volatiles in *L. petiolata* leaves that are responsible for the strong and favourable odor as in the male giant water bugs have not yet been clearly identified. Moreover, *L. petiolata*'s leaves are frequently dried before storage to improve their shelf life by retarding some growth in a small scale and protecting reactions of biochemical which possible affected smell properties (Diaz-Maroto *et al*., 2003), different drying methods employed for this purpose may cause an unexpected effect on the volatiles, phytochemicals

and the amount of antioxidant in the leaves and therefore, appropriate drying methods must be carefully selected.

The purposes of research findings were identified and compared the quality of the male giant water bugs volatiles and dried *L. petiolata*'s leaves to select the best drying method that yielded high level of phytochemicals and antioxidant activities of the dried leaves.

Materials and methods

Raw material and chemicals

L. petiolata's leaves were collected from Chanthaburi Province, while the male giant water bugs were purchased from the local morning markets in the Mahasarakham Province, Thailand. The used chemicals were analytical grade, which 6-Hydroxy-2, 5, 7, 8-tetramethyl-chroman-2-carboxylic acid (trolox), 2, 2-diphenyl-1-picrylhydrazyl hydrate (DPPH) and Folin–Ciocalteu reagent were purchased from Fluka. Phenolic standards [gallic acid, chlorogenic acid, ferulic acid, rutin and quercetin], flavonoid standards [(+)-catechin hydrate, syringic acid, *p*-coumaric acid, myricetin and kaempferol] and ascorbic acid were purchased from Sigma–Aldrich. Solvents for High Performance Liquid Chromatography (HPLC) and Gas Chromatography-Mass Spectrometry (GC-MS) analyses chromatographic grade were used.

Drying methods and extraction procedure

The leaf samples were examined by three types of drying methods: sunlight drying, hot air oven drying, and freeze drying. For the sunlight drying, the samples were distributed on trays (30×45 cm^2) and let them dried under the direct sunlight with the temperature between 32 and 35 ℃ for one day, approximately 12 hours of daylight. For oven drying, there were placed on trays and put in a hot air oven at a temperature of 60 ℃ for 12 hours. Freeze drying, the samples were dried in a freeze dryer (Scanvac, Model CoolSafe 100-9 Pro) at -92 ℃ and 0.035 mbar vacuum pressure for 12 hours. The leaf samples were smashed and kept in a desiccator until analysis. The extraction of antioxidant phenolic compounds and flavonoids from samples were carried out as described by Sánchez-Salcedo and others (2015). Briefly, approximately 1 gram of the dried leaves was obtained with 10 mL of 80% aqueous methanol acidified with 1% formic acid in a sonicator bath at 40 ℃ for 20 minutes. The supernatant was collected and filtered through 0.22 µm Nylon filter and kept at -18 ℃ for further analysis after doing centrifugation at 10,000 rounds per minute for 5 minutes.

GC-MS analysis

The volatile substances gained from the leaf subjects were analyzed using a GC–MS system comprising a model QP2010 gas chromatograph and quadrupole MS detector (Shimadzu, Japan). The volatile compounds were separated by a 5% diphenyl, l-95% of dimethylpolysiloxane fused-silica capillary column (Rtx-5Ms 30 m × 0.25 mm i.d., Restek, USA). The temperature in hot air oven was maintained at 40 ℃ for 2 minutes, then increased to 200 ℃ at 10 ℃/min and 280 ℃ at 15 ℃ / minutes, and kept for 20 minutes, the total running time was 41.33 minutes. The injector and transfer line temperatures were held at 200 ℃. The ion source temperature was set at 200 ℃. Helium was delivered as the carrier gas at a continuous pressure of 52.2 kPa with a flow rate of 1 mL/min. The data of mass spectra was collected by electron impact at 70 eV. The mass range was from m/z 40 to m/z 500 in scanning mode. The volatile components were identified by comparing the whole spectra of the subjects with the data system (NIST 11) and the stability indices.

HPLC analysis

The contents of phenolic mixed substance flavonoids and vitamin C were evaluated by the Shimadzu HPLC system with a diode array detector. The Inertsil ODS-3 C-18 column (250 mm × 4.6 mm i.d., 5 μm) and guard column were used for analysis. For phenolic compounds, temperature in columns was set at 40 ℃, and 20 μL injection volume with 0.8 mL/min of flow rate. UV-Vis spectra were recorded at 280 nm. Acetonitrile and acidified water (1% (v/v) acetic acid, pH 2.58) were utilized as moving phases A and B, respectively. For determination of flavonoids, the temperature of column was kept at 40 ℃, the injection amount was 20 μL with a flow rate of 0.6 mL/min, and the UV-Vis spectra were recorded at 254 nm. Acetonitrile/water (2/97.8, v/v) consisted 0.2% phosphoric acid, and acetonitrile/water (97.8/2, v/v) consisted 0.2% phosphoric acid which were used as mobile phases A and B, consecutively.

The extraction of vitamin C was prepared by mixing approx. 1 g of dried sample with 5 mL of 2% metaphosphoric acid using a vortex for 2 min. After filtration, the mixture was collected for HPLC analysis. Mobile phase was 0.1 M KH_2HPO_4/methanol (3:97, v/v). Chromatograms were recorded at 280 nm.

Total phenolic content (TPC)

The level of TPC in the leaf samples was analyzed by modified the method of Abu Bakar *et al.* (2009). An equal amount (12.5 μL) of the extract

and Folin-Ciocalteu reagent were mixed and kept at a room temperature for 5 minutes. Thereafter, 125 µL of 7% Na_2CO_3 solution and 100 µL of water were put to the mixture and placed in a normal room temperature for 90 minutes. The supernatant was collected and determined at 760 nm. Gallic acid equivalent (GAE) values were justified from the gallic acid standard curve and presented in milligram GAE per 100-gram dry weight.

Total flavonoid content (TFC)

The level of TFC in the leaf samples was measured using a colorimetric assay which was modified from Jorjong *et al.* (2015). The ability of the samples to absorb light was recorded at 510 nm. Catechin equivalent (CE) values were carried out from the (+)-catechin standard curve, and revealed in milligram CE per 100-gram dry weight.

DPPH radical scavenging assay

The DPPH assay was managed accordingly to Jorjong *et al.* (2015) method, 0.2 mM DPPH methanolic solution and the sample extract were thoroughly mixed in equal amounts (100 µL) and maintained in the dark for 1 hour. The mixture of DPPH and methanol in equal amounts (100 µL) served as the control. The absorbance was recorded at 520 nm. Ascorbic acid equivalent antioxidative capacity (AAEAC) values were determined from the ascorbic acid standard curve and presented in millimole AAEAC per gram dry weight.

ABTS $^{\bullet+}$ scavenging assay

The ABTS assay was performed in accordance with Seeram *et al.* (2006) with minor modification. ABTS $^{\bullet+}$ reagent was produced by putting 7.4 mM ABTS $^{\bullet+}$ solution with 2.6 mM potassium persulfate aqueous solution in the proportion of 1:1 (v/v) in the dark at a general room temperature for 12 hours before use. Trolox prepared in a range of 0–500 µM acted as an antioxidant standard. A 10 µL volume of the sample extract and 190 µL of ABTS $^{\bullet+}$ radical cation solution were thoroughly mixed and incubated for 2 hours. The absorbance of the resultant mixture was determined at 748 nm. Trolox equivalent antioxidative capacity (TEAC) values were justified from the Trolox standard curve, and presented in millimole TEAC per gram dry weight.

In this context, volatile mixed substance was investigated employing gas chromatography (GC-MS) while phytochemicals including phenolic acids, flavonoids and vitamin C were examined through liquid chromatography

(HPLC). Antioxidant existing of the dried leaves was determined by applying DPPH and ABTS $^{\bullet}$ radical scavenging assays.

Statistical analysis

Data was analyzed through a computer statistical program. All determinations were carried out in triplicate and presented by the mean ± SD. One-way analysis of variance (ANOVA) with Duncan's multiple range test was applied to identify differences between the means. In all cases, $p < 0.05$ was set to declare statistical significance of the hypotheses.

Results

Volatile compounds in male giant water bugs and L. petiolata leaf samples

The unstable mixtures found in the freeze-dried male giant water bugs and *L. petiolata* leaf samples subjected to three different drying methods were identified using GC-MS analysis. The volatiles responsible for potent odorants in male giant water bugs included undec-10-en-2-one (fatty, citrus-like), undecanone (fruity, waxy), α-cubebene (herbal, waxy), copaene (woody, spicy honey), caryophyllene (spicy, pepper-like, woody) and tridecan-2-one (waxy, earthy, mushroom). Undec-10-en-2-one and undecanone were the most powerful odorants, and were significant peaks in the total ion chromatograms (Table 1) serving as important odorant contributors (imparting characteristic citrus-like odors), Nine volatile compounds (undec-10-en-2-one, undecanone, copaene, caryophyllene, 2-norpinene, dodec-11-en-2-one, tridecan-2-one, (1R, 2R, 4S) -1-ethenyl-1-methyl-2, 4-bis (1-methylethenyl)-cyclohexane and 2, 4-di-tert-butylphenol) were detected in both male giant water bugs and *L. petiolata* leaf samples.

Notably, the levels of the two major odorants undec-10-en-2-one and undecanone were affected by drying methods, with the sun-dried samples observed to be abundant with these odorants, indicating that odorants could be easily altered by drying method.

Phytochemical contents in dried L. petiolata's leaves

Phenolic compounds

L. petiolata 's leaf samples processed through different drying methods were investigated for five major phenolic acids, namely gallic acid, chlorogenic acid, rutin, ferulic acid and quercetin. As given in Table 2, the phenolic acids except quercetin were detected in all leaf samples with differences in their relative levels. It was noted that chlorogenic acid was most abundant in all leaf

samples, followed by gallic acid and rutin, with ferulic acid detected at lowest levels. Evidently, the dried leaf samples collected from different drying methods showed variations in phenolic acids with thermal processing observed to cause a significant loss. The cool drying method was found as a promising way for conserving *L. petiolate* leaves, exhibiting a total of phenolic acids of 271.72 mg/100 g DW. The total data of the phenolic acids investigated through hot air oven and sunlight dried leaf samples were compared.

Table 1. Mass fragmentation of GC-MS identified volatile compounds from hexane soluble extracts of male giant water bugs and *L. petiotala* leaves

No [a]	Volatile compounds	RI [b]	Base peak (m/z)	Content (%)			
				Giant water bug	Freeze drying	Oven drying	Sun drying
1	8-Nonen-2one	1092	43.00	nd	nd	0.27	nd
2	2-Nonanone	1101	58.05	nd	nd	1.39	nd
3	Undec-10-en-2-one	1290	58.05	46.63	55.96	nd	63.34
4	Undecanone	1297	58.00	34.78	28.09	nd	30.70
5	Cyclohexadecanone	1305	71.05	nd	nd	53.54	nd
6	9-Octadecanole	1311	57.95	nd	nd	17.54	nd
7	9-Decen-2-one	1332	43.05	nd	2.88	5.77	nd
8	α-Cubebene	1362	119.00	0.18	nd	nd	nd
9	Copaene	1391	119.05	0.91	nd	0.42	0.92
10	α-copaene	1391	119.10	nd	0.46	nd	nd
11	β-copaene	1404	161.10	0.20	nd	nd	nd
12	Caryophyllene	1438	93.05	0.59	0.40	nd	0.69
13	β-Caryophyllene	1449	41.05	nd	nd	0.31	nd
14	2-Norpinene	1448	119.10	0.20	0.11	0.11	nd
15	1,1,4,8-Tetramethyl-cis, cis,4,7,10-cycloundecatriene	1474	93.00	nd	0.10	nd	0.18
16	Dodec-11-en-2-one	1496	58.00	6.09	5.03	11.40	nd
17	Tridecan-2-one	1502	58.00	7.93	4.76	2.50	2.68
18	(1R,2R,4S)-1-ethenyl-1-methyl-2,4-bis(1-methylethenyl)-cyclohexane	1517	121.10	0.97	0.40	nd	nd
19	2,4-Di-tert-butylphenol	1521	191.05	1.52	0.22	nd	1.03
20	9-Decen-2-one	1536	43.05	nd	1.56	6.64	nd
21	Cubenene	1541	161.10	nd	nd	nd	0.30
22	(+)-Spathulenol	1604	43.05	nd	nd	nd	0.15
23	Spathulenol	1612	43.05	nd	nd	0.12	nd

nd, not detected

[a] Number correspond to those labeled on the total ion chromatogram obtained by Head space GCMS

[b] Identification by mass spectrum (tentative), retention indices, Index standard (the series of hydrocarbons C_7-C_{30}) as a reference

Retention indices using a nonpolar dimethylpolysiloxane column

Percentage of mass spectral matching quality against NIST11 Mass Spectral Library

Flavonoids

Three flavonoids were examined from the dried leaf samples, including (+)-catechin hydrate, *p-coumaric* acid and myricetin, with (+)-catechin hydrate observed to be the most abundant compound (Table 2). Evidently, all the drying processes were differently affected in flavonoid contents in *L. petiolata* leaves. Among the drying methods, freeze drying was more efficient than the others due to its beneficial effect to maintain the major flavonoids in comparison to hot air oven and sunlight drying methods, even though myricetin in the cool-dried method was compared to hot air oven and sunlight dried leaf samples.

Vitamin C

The vitamin C contents in freeze, hot air oven and sunlight dried *L. petiolata* leaves are presented in Table 2. The study found that after drying through the freeze-dried, the samples had higher vitamin C content (3.64 mg/100 g DW) than the samples dried in hot air oven (5.78 mg/100 g DW), and the samples dried by sunlight (3.88 mg/100 g DW). However, the vitamin C capacity obtained from the freeze-dried method, and the hot air oven dried method was not statistically significantly different.

Table 2. Phytochemical constituents (phenolic-acids, flavonoids and vitamin C) of dried *L. petiotala* leaves processed through three different drying methods

Phytochemical constituents		**Drying methods**		
		Freeze drying	**Oven drying**	**Sun drying**
Phenolic acids (mg/100g DW)	1. Gallic acid	46.78 ±3.42a	29.15 ±3.10b	51.11 ±1.28a
	2. Chlorogenic acid	169.43 ±20.11a	128.49 ±7.54b	105.74 ±15.34b
	3. Rutin	40.57 ±3.11a	23.69 ±3.72b	30.34 ±0.69c
	4. Ferulic acid	14.93 ±0.54b	20.57 ±0.82a	13.95 ±3.82b
	5. Quercetin	nd	nd	nd
	Total	271.72	201.91	201.13
Flavonoids (mg/100g DW)	1. (+)- Catechin hydrate	839.69 ±36.55a	603.56 ±62.99b	555.20 ±44.25b
	2. Syringic acid	nd	nd	nd
	3. P-cumaric acid	105.53 ±7.22a	64.26 ±6.74b	70.77 ±7.27b
	4. Myracetin	9.53 ±0.17a	10.73 ±1.14a	11.32 ±1.13a
	5. Kaemperol	nd	nd	nd
	Total	954.75	678.55	637.30
Vitamin C (mg/100g DW)		6.34 ±0.48a	5.78 ±0.57a	3.88 ±0.11b

nd, not detected.

Values are expressed as mean ± SD of triplicate measurement.

Different letters in the column indicate significant differences at $p < 0.05$.

Antioxidant capacity of dried L. petiolata leaves

Total phenolic and total flavonoid contents

The tested plants produced a variety of phenolics and flavonoids at various levels depending on their families and planting procedures, identification and quantitative analysis of the major phenolics and flavonoids may not be sufficient to declare the effects of drying methods on the antioxidant content of the studied plant samples. In this study, the volatility of t phenolic and flavonoid contents of the dried *L. petiolata* leaf samples were affected by three different drying methods (Table 3). The scales of phenolic contents measured on the *L. petiolata* leaves subjected to different drying methods were significantly differed. The hot air oven dried leaves showed the highest amount of gross phenolics (188.38 mg GAE/100 g DW). On the other hand, the levels of gross phenolic contents in the freeze-dried method (154.19 mg GAE/100 g DW) and the sunlight dried method (154.17 mg GAE/100 g DW) leaves were compared.

Table 3. Comparison of total phenolics, total flavonoids and antioxidant activities of dried *L. petiotala* leaves processed through three different drying methods

	Drying methods		
Antioxidant activity	**Freeze drying**	**Oven drying**	**Sun drying**
[a] TPC (mg GAE/100g DW)	154.19±18.75b	188.38±13.02a	154.17±14.23b
[b] TFC (mg GAE/100g DW)	300.03±6.74a	301.20±4.49a	294.30±8.88a
[c] DPPH (mM AAE/100g DW)	151.15±1.10a	148.85±0.18ab	147.50±2.05b
% Inhibition DPPH	76.07±0.53a	74.98±0.08ab	74.33±0.98b
[d] ABTS (mM TE/100g DW)	777.67±4.16a	772.67±6.35a	736.33±27.65b
% Inhibition ABTS	86.19±0.46a	85.63±0.70a	81.59±3.07b

Values are expressed as mean ± SD of triplicate measurement.
TPC, total phenolic content, TFC, total flavonoid content; DPPH, DPPH radical scavenging; ABTS, ABTS radical scavenging.
Different letters in the column indicate significant differences at $p < 0.05$.

ABTS $^{\bullet+}$ assay and DPPH radical scavenging activity

ABTS $^{\bullet+}$ radical cation assays, expressed as TEAC values, were used to assess free radical-scavenging capacity of the dried *L. petiolata* leaf samples. As shown in Table 3, significantly differences in the antioxidant activities were examined among the dried leaf samples processed by different drying methods. The antioxidant activity among the dried samples varied from 736.33 to 777.67 mM TEAC/100 g DW, with the highest antioxidant contents obtained from the freeze-dried method.

DPPH radical scavenging assay also conducted to determine the antioxidant activities of the dried *L. petiolata* leaves. The values of DPPH measured in the dried leaf samples ranged from 147.5 to 151.15 mM AA/100 g DW, with the percentage inhibition of DPPH which ranged from 74.33 to 76.07. The freeze-dried samples were found to have the strongest antioxidant capacity as compared to the hot air oven dried and sunlight dried samples (Table 3).

Discussion

The research finding presented the comparison between profiles of volatile compounds in *L. petiolata* leaves and male giant water bugs. Moreover, the effects of three drying methods, sunlight drying, hot air oven drying and freeze drying on the active odorants, bioactive compounds, and antioxidant reactions of the leaves were investigated.

It was noted that cool drying was the best method among the other two methods for conserving the phenolic and flavonoid contents of the leaves, which was consistent with an earlier study (Gümüşay *et al*., 2015) which elucidated that cool drying was the most efficient in maintaining the phenolic quality in tomatoes and ginger. Moreover, Routray *et al*. (2014) found that the freeze-dried blueberry leaves had the highest content of total phenolics along with high antioxidant quality, as compared to the microwave-dried method. Similarly, Julkunen-Tiitto and Sorsa (2001) demonstrated that high contents of (+)-catechin were observed in the freeze-dried leaves of purple willow but was not detected in the heat-dried leaf samples. However, cool drying method and oven or tray drying method at 30 ℃ were reported no significant influence on the phenolic capacity (total phenols, salicylates, and quercetin) of meadowsweet and willow as compared to traditional air-drying (Harbourne *et al*., 2009). On the contrary, shade drying was found to be a promising method to preserve phenolic acids (chlorogenic acid and rutin) in *Stevia rebaudiana* leaves, as it was compared to freeze- and air-drying methods (Periche *et al.*, 2016). Besides, jujubes processed through explosion puffing, a processing system which facilitates hot air drying of fruits and vegetables, were reported to have higher levels of gallic, *p*-hydroxybenzoic, vanillic, *p*-coumaric, ferulic acids, and rutin amount than the sun-dried jujubes (Du *et al*., 2013). Similarly, Mohd Zainol *et al.* (2009) revealed that the oven drying method yielded the highest flavonoid reduction followed by vacuum and cool drying methods, with catechin and rutin found to be the most consistent flavonoids.

The current study revealed the interesting finding that thermal methods appeared to incur a significant loss in vitamin C contents as compared to freeze drying, which was well supported by Demiray's study which posited that the

presence of oxygen and heat caused the reduction of vitamin C (Demiray *et al.*, 2013; Veras *et al*., 2012). Chang *et al.* (2006) claimed that vitamin C contents were reduced by 66% when employing heat drying. Kaya *et al.* (2010) also postulated that increasing drying air temperature generated more loss in vitamin C in the dried kiwi fruits. On the contrary, vitamin C reduction was decreased by increasing the humidity of drying air method.

After analyzing the results of drying methods on the whole phenolic and flavonoid capacity, it was found that oven drying method was the most suitable for keeping the total phenolics in *L. petiolata* leaves, which was well supported by an earlier study (Ling *et al*., 2015) demonstrating that the oven-dried red seaweed (Kappaphycus alvarezii; 'crocodile' morphotype) samples contained higher values of total phenolic contents when it was compared with other drying methods. Similarly, Lutz *et al.* (2015) elucidated that convective drying gave rise to the enhancement of the whole phenolic capacity in fruits and vegetables. However, oven drying was found to result in a decrease in overall phenolic amounts in Neem leaves as it was compared to shade drying (Sejali and Anuar, 2011). Even though the oven-dried leaf samples appeared to have the highest level of total flavonoid contents (301.20 mg GAE/100 g DW), there were no significant differences between the sun-, oven- and freeze-dried leaf samples, indicating that flavonoids were stable to a certain degree. This finding was in accordance with the previous study (Alonzo-Mac ás *et al*., 2013) which showed insignificant differences of total flavonoid contents in strawberry as affected by thermal and cool drying. On the contrary, thermal drying technique was reported to significantly increase the level of total flavonoids in raspberry when it was compared to other drying methods (Si *et al.*, 2016). By contrast, cool-dried pomegranate peels were found to have the highest total flavonoid concentrations as it was compared to the peels dried by the oven (Mphahlele *et al*., 2016).

The findings of his study revealed that the cool drying method was the most appropriate for keeping the antioxidants in *L. petiolata* leaves, which was consistent with the study of Sogi (Sogi *et al.*, 2013) who claimed that the mango kernel obtained the highest antioxidant contents when it was dried by cool drying method. Moreover, Yi and Wetzstein (2011) postulated that the oven-drying method reduced significantly the amount of antioxidant in rosemary (*Rosmarinus officinalis*), motherwort (*Leonurus cardiaca*), and peppermint (*Mentha piperita*); herbs dried by sun and 40 ℃ dried by the oven exhibited higher TEAC capacity as compared to 70 ℃ oven-dried herbs. Mediani *et al*. (2014) also showed that freeze drying of ulam raja (*Cosmos caudatus*) resulted in its higher free radical scavenging activity against 1, 1-diphenyl-2-picrylhydrazyl or DPPH (IC50 = 0.0223 mg/mL) as compared to oven drying. Higher DPPH scavenging activity was reported to be detected in

dried black chokeberries processed using freeze drying (Thi and Hwang, 2016). On the other hand, oven drying gave rise to higher DPPH scavenging activity of Iranian quince in comparison to sun drying (Gheisari and Abhari, 2014). However, thermal drying was found to cause a significant decrease in DPPH scavenging activity of raspberry fruits as compared to fresh raspberry fruits, in which hot-air drying method, hot air and explosion puffing drying, infrared radiation drying, and infrared radiation and microwave vacuum drying methods found to observed to better retain the antioxidant activity of dried raspberry fruits than freeze drying method (Si *et al*., 2016).

In conclusion, this research highlighted the major volatile mixed substance in the male giant water bugs and the dried *L. petiolata* leaf samples which might be the agents of their strong and desirable fragrance. Nine major volatiles were identified in both the male giant water bugs and the dried leaf samples, and Undec-10-en-2-one and Undecanone were the most abundant volatile compounds. Freeze drying was the most effective method in maintaining the level and type of volatiles. This method was also useful to keep the high amount of phytochemicals (phenolic acids, flavonoids and vitamin C) and antioxidants. These results showed that *L. petiolata* leaves are rich in bioactive compounds with high levels of antioxidants that are beneficial to human health.

Acknowledgements

The authors would like to thank the Laboratory Equipment Center of the Division of Research Facilitation and Dissemination of Mahasarakham University for the performance of HPLC and GC-MS analysis. The authors are also grateful to Assistant Professor Dr.Surachai Piyanukool, Faculty of Humanities and Social Sciences, Buriram Rajabhat University, Thailand for kindly proofreading the article.

References

Abu Bakar, M. F., Mohamed, M., Rahmat, A. and Fry, J. (2009). Phytochemicals and antioxidant activity of different parts of bambangan (*Mangifera pajang*) and tarap (*Artocarpus odoratissimus*). Food Chemistry, 113:479-83.

Agrawal, N., Choudhary, A. S., Sharma, M. C. and Dobhal, M. P. (2011). Chemical constituents of plants from the genus *Litsea*. Chemistry & Biodiversity, 8:223-43.

Alonzo-Macías, M., Cardador-Martínez, A., Mounir, S., Montejano-Gaitán, G. and Allaf, K. (2013). Comparative study of the effects of drying methods on antioxidant activity of dried strawberry (*Fragaria* Var. Camarosa). Journal of Food Research, 2:92-107.

Balange, A. and Enjakul, S. (2009). Use of kiam wood extract as gel enhancer for mackerel (*Rastrelliger kanagurta*) surimi. International Journal of Food Science & Technology, 44:1661-9.

Chang, C. H., Lin, H. Y., Chang, C. Y. and Liu, Y. C. (2006). Comparisons on the antioxidant properties of fresh, freeze-dried and hot-air-dried tomatoes. Journal of Food Engineering, 77:478-85.

Demiray, E., Tulek, Y. and Yilmaz, Y. (2013). Degradation kinetics of lycopene, beta-carotene and ascorbic acid in tomatoes during hot air drying. LWT-Food Science and Technology, 50:172-6.

Diaz-Maroto, M. C., Perez Coello, M. S., Gonzalez Vinas, M. A. and Cabezudo, M. D. (2003). Influence of drying on the flavour quality of spearmint (*Mentha spicata* L.). Journal of Agricultural and Food Chemistry, 51:1265-9.

Du, L. J., Gao, Q. H., Ji, X. L *et al.* (2013). Comparison of flavonoids, phenolic acids, and antioxidant activity of explosion-puffed and sun-dried jujubes (*Ziziphus jujuba* Mill.). Journal of Agricultural and Food Chemistry, 61:11840-7.

Gheisari, H. R. and Abhari, K. H. (2014). Drying method effects on the antioxidant activity of quince (*Cydonia oblonga* Miller) tea. Acta Scientiarum Polonorum, Technologia Alimentaria, 13:129-34.

Gümüşay, Ö. A., Borazan, A. A., Ercal, N. and Demirkol, O. (2015). Drying effects on the antioxidant properties of tomatoes and ginger. Food Chemistry, 173:156-62.

Hanboonsong, Y., Jamjanya, T. and Durst, P. B. (2013). Six-legged livestock: edible insect farming, collecting and marketing in Thailand. Food and Agriculture Organization of the United Nations Regional Office for Asia and the Pacific, Bangkok, Thailand.

Harbourne, N., Marete, E., Jacquier, J. C. and O'Riordan, D. (2009). Effect of drying methods on the phenolic constituents of meadowsweet (*Filipendula ulmaria*) and willow (*Salix alba*). LWT-Food Science and Technology, 42:1468-73.

Jorjong, S., Butkhup, L. and Samappito, S. (2015). Phytochemicals and antioxidant capacities of Mao-Luang (*Antidesma bunius* L.) cultivars from Northeastern Thailand. Food Chemistry, 181:248-55.

Julkunen-Tiitto, R. and Sorsa, S. (2001). Testing the effects of drying methods on willow flavonoids, tannins, and salicylates. Journal of Chemical Ecology, 27:779-89.

Kaya, A., Aydın, O. and Kolaylı, S. (2010). Effect of different drying conditions on the vitamin C (ascorbic acid) content of Hayward kiwifruits (*Actinidia deliciosa* Planch). Food and Bioproducts Processing, 88:165-73.

Kiatbenjakul, P., Intarapichet, K. and Cadwallader, K. R. (2015). Characterization of potent odorants in male giant water bug (*Lethocerus indicus* Lep. and Serv.), an important edible insect of Southeast Asia. Food Chemistry, 168:639-47.

Kong, D. G., Zhao, Y., Li, G. H *et al*. (2015). The genus *Litsea* in traditional Chinese medicine: an ethnomedical, phytochemical and pharmacological review. Journal of Ethnopharmacology, 164:256-64.

Ling, A. L. M., Yasir, S., Matanjun, P. and Abu Bakar, M. F. (2015). Effect of different drying techniques on the phytochemical content and antioxidant activity of *Kappaphycus alvarezii*. Journal of Applied Phycology, 27:1717-23.

Longo, M. A. and Sanroman, M. A. (2010). Vegetable flavors from cell culture. In: Hui YH, Chen F, Nollet, LML (eds) Handbook of fruit and vegetable flavors. John Wiley and Sons, Inc., pp.663-680.

Lutz, M., Hernández, J. and Henríquez, C. (2015). Phenolic content and antioxidant capacity in fresh and dry fruits and vegetables grown in Chile. CyTA - Journal of Food, 13:541-7.

Mediani, A., Abas, F., Tan, C. P. and Khatib, A. (2014). Effects of different drying methods and storage time on free radical scavenging activity and total phenolic content of *Cosmos caudatus*. Antioxidants, 3:358-70.

Mohd Zainol, M. K., Abdul-Hamid, A., Abu Bakar, F. and Pak Dek, S. (2009). Effect of different drying methods on the degradation of selected flavonoids in *Centella asiatica*. International Food Research Journal, 16:531-7.

Mphahlele, R. R., Fawole, O. A., Makunga, N. P. and Opara, U. L. (2016). Effect of drying on the bioactive compounds, antioxidant, antibacterial and antityrosinase activities of pomegranate peel. BMC Complementary and Alternative Medicine, 16:143.

Ngernsaengsaruay, C., Middletin, D. J. and Chayamarit, K. (2011). A revision of the genus *Litsea* Lam. (Lauraceae) in Thailand. Thai Forest Bulletin (Botany), 39:40-119.
Panyamongkol, S. (1994). Extraction of essential oil from Thummang (*Litsea petiolata* hook.f.) leaves and twigs as a food flavour. (Master Thesis). Chulalongkorn University.
Periche, A., Castelló, M. L., Heredia, A. and Escriche, I. (2016). Effect of different drying methods on the phenolic, flavonoid and volatile compounds of *Stevia rebaudiana* leaves. Flavour and Fragrance Journal, 31:173-7.
Pimpaka, N., Laohakunjit, N. and Kerdchoechuen, O. (2007). Volatile composition of essential oils from Thammumg (*Litsea petiolata*). Agricultural Science Journal, 38:181-4.
Routray, W., Orsat, V. and Gariepy, Y. (2014). Effect of different drying methods on the microwave extraction of phenolic components and antioxidant activity of highbush blueberry leaves. Drying Technology, 32:1888-904.
Sánchez-Salcedo, EM., Mena, P., García-Viguera, C., Martínez, J. J. and Hernández, F. (2015). Phytochemical evaluation of white (*Morus alba* L.) and black (*Morus nigra* L.) mulberry fruits, a starting point for the assessment of their beneficial properties. Journal of Functional Foods, 12:399-408.
Seeram, N. P., Henning, S. M., Niu, Y., Lee, R., Scheuller, H. S. and Heber, D. (2006). Catechin and caffeine contents of green tea dietary supplements and correlation with antioxidant activity. Journal of Agricultural and Food Chemistry, 54:1599-603.
Sejali, S. N. F. and Anuar, M. S. (2011). Effect of drying methods on phenolic contents of neem (*Azadirachta indica*) leaf powder. Journal of Herbs Spices and Medicine Plants, 17:119-31.
Si, X., Chen, Q., Bi, J., Wu, X., Yi, J., Zhou, L. and Li, Z. (2016). Comparison of different drying methods on the physical properties, bioactive compounds and antioxidant activity of raspberry powders. Journal of The Science of Food and Agriculture, 96:2055-62.
Sogi, D. S., Siddiq, M., Greiby, I. and Dolan, K. D. (2013). Total phenolics, antioxidant activity, and functional properties of 'Tommy Atkins' mango peel and kernel as affected by drying methods. Food Chemistry, 141:2649-55.
Thi, N. D. and Hwang, E. S. (2016). Effects of drying methods on contents of bioactive compounds and antioxidant activities of black chokeberries (*Aronia melanocarpa*). Food Science and Biotechnology, 25:55-61.
Veras, A. O. M., Bétega, R., Freire, F. B., Barrozo, M. A. S. and Freire, J. T. (2012). Drying kinetics, structural characteristics and vitamin C retention of dedo-de-moça pepper (*Capsicum baccatum*) during convective and freeze drying. Brazillian Journal of Chemical Engineering, 29:741-50.
Wang, Y. S., Wen, Z. Q., Li, B. T., Zhang, H. B. and Yang, J. H. (2016). Ethnobotany, phytochemistry, and pharmacology of the genus *Litsea*: an update. Journal of Ethnopharmacology, 181:66-107.
Wishram, W. A., Saengthong, P. and Ngernsiri, L. (2013). Meiotic chromosome analysis of the giant water bug, *Lethocerus indicus*. Journal of Insect Science, 13:39.
Yang, K., Wang, C. F., You, C. X., Geng, Z. F., Sun, R. Q., Guo, S. S., Du, S. S., Liu, Z. L. and Deng, Z. W. (2014). Bioactivity of essential oil of *Litsea cubeba* from China and its main compounds against two stored product insects. Journal of Asia-Pacific Entomology, 17:459-66.
Yi, W. and Wetzstein, H. Y. (2011). Effects of drying and extraction conditions on the biochemical activity of selected herbs. HortScience, 46:70-3.

(Received: 23 August 2020, accepted: 18 June 2021)