Influence of Maturity and Drying Temperature on Antioxidant Activity and Chemical Compositions in Ginger

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Abstract

The objective was to study the effect of maturity at harvest and drying temperature on antioxidant activity and physiochemical properties of ginger (Zingiber officinale). The effects of two different maturities of harvest, i.e. 6 months and 9 months were examined. It was found that the contents of carbohydrate and fiber values increased in the 9-month sample. The 9-month sample was higher in total phenolic contents (5.08 µmol Tannic acid/g), DPPH (85.33% inhibition), ABTS (42.23 µmol Trolox/g) and FRAP (13.78 µmol Trolox/g) than the younger 6-month sample. In essential oil constituents, the 9-month sample contained higher amounts of geranial (28.31%), neral (15.29%), β-phellandrene (13.32%), campene (7.77%) and α-zingiberene (6.17%) in comparison to the 6month samples. The 9-month sample contained the highest level of 6-gingerol (24.36 mg/g). Therefore, the ginger harvested at 9 months was selected for the study of different drying temperatures. Drying times and temperature to achieve moisture content below 10% at 308 min, 60°C were proven to be the best condition as it achieved the highest total phenolic content (12.21 μmol Tannic acid /g), DPPH (91.35%), ABTS (223.50 μmol Trolox/g) and FRAP (42.39 μmol Trolox/g). Ginger contains 45 volatile compounds and the highest main compounds being αzingiberene (18.28%), α-farnesene (10.73%) and geranial (12.42%) when drying temperature was at 60°C. The content of 6-gingerol was also found to be the highest in the sample dried at 60°C (12.57 mg/g).

Keywords: Zingiber officinale, ginger, maturity, antioxidant, volatile compounds, drying DOI 10.14456/cast.2019.4

1. Introduction

Ginger (Zingiber officinale Roscoe) is a unique aromatic herb used around the world in food and spices. It contains both non-volatile and volatile compounds. The unique aroma is provided by the

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essential oils and the pungency contained in the non-volatile compounds. The odor of ginger depends on its major volatile oil, the content of which changes 1-3%. More than 50 volatile compounds were found and characterized in ginger by using gas chromatography massspectrometry. The essential oil contains monoterpenoid and sesquiterpene hydrocarbons. The monoterpenoids include β-phellandrene, camphene, cineole, geraniol, citral, terpineol and borneol. The sesquiterpenoids include α -zingiberene, β -sesquiphellandrene, β -bisabolene, (E,E)- α -farnesene, ar-curcumene and zingiborol [1]. Ginger also contains a variety of oleoresins, namely gingerol, shogaol, paradol and zingerone. Gingerol is the rhizome compound responsible for pungency. Gingerol compounds are transformed at high temperatures to shogaol [2]. Period from planting to maturity could affect the antioxidant activities and bioactive compounds. The main bioactive compounds of Jamaica ginger harvested at 6-8 months after planting depend on the variety of location and the age of rhizome [3] and the harvest duration depends on the end-use. In Thailand, there are no studies on the proximate, essential oil composition and bioactive compounds of fresh ginger cultivars of 6- and 9- month maturity period. Generally, fresh ginger consists of 85-95% moisture content and is easily affected by microbial spoilage and chemical decomposition. The drying method reduces the moisture content resulting in bacterial growth inhibition and delays deteriorative biochemical reactions. However, the drying temperature can cause deterioration of the physical and chemical properties and some of the oil constituents were also changed to less volatile compounds. The other researchers reported that the antioxidant activity showed highest activity in 80% methanolic ginger extract because it is suitable extracting solvent capable of extracting the lipophilic antioxidant composites [4]. Therefore, the purpose of this study is to investigate the effects of maturity at harvest and drying temperature on physical, chemical and essential oil composition of ginger. In addition, the total phenolic contents, antioxidant properties (DPPH, ABTS and FRAP), essential oil compositions and main bioactive compounds were also measured.

2. Materials and Methods

2.1 Raw materials

The ginger rhizomes (Zingiber offinale Roscoe) were divided into two groups according to their maturity (6 months and 9 months). These ginger rhizomes were obtained from the same source in Chiang Rai, Thailand.

2.2 Effects of physiochemical properties of different maturity

The peeled gingers (6 months and 9 months) were washed in distilled water and cut into 0.2 x 4 x 0.4 cm. The fresh ginger was then kept in the vacuum package and stored in refrigerator at 0-4 °C (Mitsubishi electric, MRFV 22M/BR, Japan) for 24 h before further analysis.

2.3 Effects of physicochemical properties of different drying temperatures

The drying process was as per the method reported earlier with some modifications [5]. Samples were spread in the hot air dryer (Armfield, Hamshire, England). Drying experiments were performed with a sample of 200 g for ginger rhizomes dried in a thin layer at 40, 50 and 60°C. The moisture content was evaluated every 30 min to monitor the moisture loss. The weight loss of the sample was recorded every 10 min using a data logger (DT 800, Data taker, Scoresby, Victoria, Australia). The drying was finished when the weight was stable and the moisture content of the ginger had been decreased to 10% wet basis. The dried ginger was ground, sieved at 1.2 mm, kept

in the vacuum aluminum foil package and stored in a desiccator at ambient temperature for at least 24 h before further analysis.

2.4 Physical characteristics and proximate analysis

The color was expressed in L*, a*, b* by the colorimeter (Konica Minolta CR-400 Series, Japan). Water activity was measured using an AquaLab Water Activity Meter (Decagon, USA). Proximate analysis was determined by using standard AOAC methods [6].

2.5 Total phenolic content (TPC)

Ten grams of ground fresh ginger was accurately weighed and transferred into 100 ml of 80% methanol [4]. The sample was shaken for 3 h at an ambient temperature. Following this technique, it was centrifuged at 4,000 rpm for 20 min and then filtrated through Whatman No. 1 paper. Then the ginger extracts were used in total phenolic compound analysis and antioxidant activities (DPPH, ABTS and FRAP). Total phenolic compounds were examined using the method described by Re *et al.* [7]. The ginger solution (200 μl) and 10% Folin-Ciocalteu reagent (1 ml) (Loba chemie, India) were mixed and 2% Na₂CO₃ was then added with a water:methanol (4:6) diluting solvent to make a total volume of 10 ml. Absorbance was recorded at 740 nm after 30 min using a spectrophotometer (UV-Vis model 1601, Shimadzu, Japan). The absorbance was then compared with the tannic acid standard curve. The result was shown as μmol per gram of dried weight.

2.6 Antioxidant activities

2.6.1 DPPH radical-scavenging activity

Free radical scavenging activity of the extracts was determined using stable free radical 2, 2-Diphenyl-picrylhydrazyl (DPPH) (Sigma-aldrich, U.S.A.) with some modifications [4]. Four ml of extract solution and 1 ml of DPPH solution were mixed (0.1 mM in methanol) by a vortex mixer and then stood at room temperature in dark storage for 30 min. The absorbance was recorded at 520 nm. The percentage of scavenging effect was calculated using the following equation (1) shown below:

Radical scavenging activity (%) =
$$[A_0 - A_1]/A_0 \times 100$$
 (1)

Where A_0 was the absorbance of control solution (DPPH without sample) and A_1 was the absorbance of the ginger extract in DPPH solution.

2.6.2 ABTS method

Antioxidant activity was evaluated using 2, 2'-azinobis (3-ethylbenzothiazoline-6-sulphonic acid) diammonium salt (ABTS) (Fluka, Germany) according to the modified method from Re $\it et al.$ [7]. A mixture between 7 mM ABTS and 2.45 mM potassium persulphate, the ABTS+ solution, was stood in dark place for 14 ± 2 h before use. Afterwards, the ABTS+ solution was diluted with ethanol to measure an absorbance of 0.700 ± 0.02 at 734 nm. Ginger extract (150 μ l) was allowed to react with 4,850 μ l of the ABTS+ solution for 6 min and then read by a spectrophotometer at 734 nm. The absorbance was compared with the Trolox standard curve and the results are shown in μ mol per gram dry weight.

2.6.3 Ferric reducing ability power assay (FRAP assay)

The FRAP assay was determined by the modification method from Benzie and Strain [8]. The FRAP reagent, consisting of 300 mM acetate buffer (1.22 g $C_2H_3NaO_2 \cdot 3H_2O$ and 8.05 ml $C_2H_4O_2$), pH 3.6, 10 mM TPTZ (2, 4, 6-tripyridyl-s-triazine) solution in 40 mM HCl, and 20 mM FeCl₃· 6H₂O solution in the ratio of 10: 1: 1, was warmed at 37 °C before use. The FRAP solution (3 ml) was added to 150 μ l of ginger extract for 10 min at 37 °C and the absorbance was recorded at 593 nm. The results are shown as μ mol per gram dry weight.

2.7 Analysis of volatile compounds from essential oil in ginger using gas chromatography

The volatile compounds from essential oil in ginger were studied using gas chromatography according to the method modified from Kiran *et al.* [1]. Peeled fresh rhizomes were chopped and approximately 200 g (30 g for ground dried ginger) of this material was hydrodistilled for 3 h. The essential oil was kept in micro tubes and frozen at -20 °C. Prior to analysis, the extracts were dissolved with hexane at appropriate concentration and filtered by 0.45 µm. pore size filter before being measured by the gas chromatography-mass spectrometry (GC-MS) and gas chromatography (GC-FID).

Essential oils were studied using Shimadzu GC-MS model GC-17A and equipped with mass spectrophotometer model GC-MS QP 5050A. The carrier gas was helium at a flow rate of 1 ml/min using capillary column (BPX-5, SGE, Ltd., Melbourne, Victoria, Australia) which had a dimension of 30 m \times 0.250 mm inner diameter \times 1.00 μ m for this analysis. A split ratio of 1:50 was used. The oven temperature was set from 80°C (holding time of 1 min) to 220°C at 5 °C/min and held for 10 min. The injection port was heated to 250°C. The interface of temperature was 270°C with an ionization voltage of 70 eV. The constituents were identified by comparison with authentic standards, retention indices (RIs) and mass spectra (MS) of National Institute of Standards and Technology (NIST) sources, flavors and fragrance of natural and synthetic compounds (FFNSC) references.

Essential oils were studied using a gas chromatograph model Shimadzu GC-2010. Helium was used as the carrier gas at a flow rate of 1 ml/ min. A capillary column (60 m \times 0.22 mm inner diameter \times 1.00 μ m df) was used for the analysis (BPX- 5, SGE, Ltd., Melbourne, Victoria, Australia), and a flame ionization detector (FID) was used. A split ratio of 1:50 was used. The oven temperature was started at 80°C and held for 1 min before heating to 220°C at a rate of 5°C/min and held for 10 min. The injection port was heated to 250°C, and the detector temperature was set at 300°C. Identification of compound was calculated by retention indices using a C_8 - C_{20} alkane standard solution.

2.8 Analysis of active compounds in ginger using high performance liquid chromatography method (HPLC)

Ground ginger (10 g) was weighed and extracted with 100 ml of 95% ethanol by electronic shaker for 30 min. The solvent was concentrated in a rotary evaporator and filtered through a Whatman No.1. After filtering through a 0.2 μm syringe filter, the final sample was used for injection. Standards of 6- gingerol, 6- shogaol, paradol and zingerone were prepared. The method was performed on HPLC (HPLC, Agilent Technologies, Santa Clara, CA, USA) with a photodiode-array detector. The optimal HPLC system contained a C₁₈ reverse phase column (Water C₁₈, 250x4.6 mm i.d., 5 μm particle size). The gradient elution was acetonitrile and water at a flow rate of 1.0 ml/min and a detection of 282 nm. The mobile phase contained water (A) and acetonitrile (B). Both solutions were filtered by 0.45 μm pore size filter. The gradient elution program was set as follows:

from 0 to 25 min, B was isocratic at 33%; from 25-35 min, solution B was changed from 33% to 55%; from 35 to 60 min, B followed changed linearly from 55% to 90%; from 60 to 65 min, B was a linear change from 90% to 33%; and from 65 to 70 min, B was isocratic at 33% [9].

2.9 Statistical analysis

The data was shown as mean±standard deviation for at least triplicate analyses. Mean comparisons of physical and chemical properties (proximate analysis, total phenolic content, DPPH, ABTS, FRAP and bioactive compounds) were analyzed using ANOVA for a Completely Randomized Design (CRD) in SPSS version 17.0 (SPSS Inc., Chicago, USA). Statistical significance was analyzed for p≤0.05 using Duncan's New Multiple Range Test (DMRT).

3. Results and Discussion

3.1 Effects of 6 and 9-month maturity on the physiochemical properties of ginger rhizome cultivars

The physicochemical properties of ginger harvested at different maturities are shown in Table 1. The different harvest time of the ginger rhizome affected the color (L*, a* and b*), moisture content, water activity, carbohydrate, protein, fat, fiber and ash. The color of the ginger indicated a light yellow trend. The L* movement showed lightness and b* direction denoted a change to yellow. Ginger harvested at 9 months had higher yellow and lightness than the 6-month samples. The ginger harvested at 6 months had higher moisture content and water activity than the samples at 9 months. There was also significant change in crude fiber as their composition increased in the more mature sample. Harvest for dried herbs and essential oil is best at long maturity with increase of the fiber content [10]. The carbohydrate content increased from 31.36% (6-month sample) to 39.09% (9month sample). The fiber content increased from 38.41% (6-month sample) to 43.73% (9-month sample). However, the protein, fat and ash contents of the sample were decreased in the more mature ginger. Increase of crude fiber and decrease of fat and protein contents of rhizome were observed after 6 months and 15 days [11]. Other researchers found that dry recovery, carbohydrate and crude fiber were positively correlated with maturity whereas essential oil, oleoresin and protein were negatively related with maturity [12, 13]. The values for composition of ginger are reported in the following range: 7.2 to 8.7, 5.5 to 7.3 and 2.5 to 5.7 g/100 g dried weight for protein, fat and ash, respectively [1]. Our study showed that ginger harvested at 9 months contained a higher total phenolic content (TPC) (5.08±0.28 µmol tannic acid/g) than ginger harvested at 6 months (2.61±0.12 µmol tannic acid/g) (Table 1). This finding is in an agreement with Chumroenphat et al. [14] who reported that matured ginger had a higher total phenolic content than immature ginger. Antioxidant activities of plant extracts were usually correlated to their phenolic content. Hydrogen donating characteristics of the phenol compounds can inhibit free radical induced to scavenge free radicals and donate oxygen [1]. However, it is known that non phenolic antioxidants might also lead to the antioxidant activity of plant extract [15, 16]. Ginger harvested at 9 months was able to reduce the stable free radical 2, 2- diphenyl-1-picrylhydrazyl (DPPH) to yellow-colored DPPH (70.48%). DPPH radical scavenging activity in ginger harvested at 9 months was found to be 70.48%, compared to 55.40% found in ginger harvested at 6 months. In addition, the ginger harvested at 9 months also displayed the highest radical scavenging potential of ABTS (42.23±7.80 µmol Trolox/g) and FRAP (13.78±0.66 µmol Trolox/g). According to Chen et al. [17], DPPH values of methanolic extract were found between 32% and 90.1% of 18 different ginger types.

Table 1. Physicochemical properties of fresh ginger at different maturities.

Characteristics	6-month maturity	9-month maturity
L*	55.40 ± 0.01^{b}	70.48 ± 0.06^{a}
a*	7.29 ± 0.31^{a}	-1.54 ± 0.90^{b}
b*	9.39 ± 0.18^{b}	23.21 ± 3.76 ^a
Water activity	1.000 ± 0.01^{a}	0.996 ± 0.01^{b}
Moisture (% wet basis)	94.88 ± 0.15^{a}	87.25 ± 1.03^{b}
Carbohydrate (% dry basis)	31.36 ± 0.98^{b}	39.09 ± 0.58^{a}
Protein (%dry basis)	11.60 ± 0.37^{a}	8.95 ± 0.71^{b}
Fat (%dry basis)	4.27 ± 0.20^{a}	2.33 ± 0.11^{b}
Fiber (%dry basis)	38.41 ± 0.41^{b}	43.73 ± 0.64^{a}
Ash (%dry basis)	14.35 ± 0.82^{a}	6.63 ± 0.13^{b}
Total Phenolic content (μmol Tannic acid/g)	2.61 ± 0.12^{b}	5.08 ± 0.28^{a}
DPPH (%inhibition)	62.97 ± 1.49^{b}	85.33 ± 2.47 ^a
ABTS (µmol Trolox/g)	7.57 ± 3.03^{b}	42.23 ± 7.80^{a}
FRAP (µmol Trolox/g)	8.40 ± 0.59^{b}	13.78 ± 0.66^{a}

a-b Different letters show significant difference between means in the same row (p≤0.05).

Essential oil composition is one significant parameter to determine variation among different ginger cultivars. By GC-MS and GC-FID analyses and it was found that there were 44 volatile compounds in the essential oil of ginger cultivars. The percentage compositions of various components from ginger after planting at 6 and 9 months are shown in Table 2. The data shows that there are 34 and 36 volatile compounds in ginger harvested at 6 and 9 months, respectively. The data clearly reports a difference in essential oil composition with different maturity periods. The major volatile compounds of 6-month cultivars are geranial (24.20%), nerol (13.34%), 3-carene (10.53%), β -phellandrene (10.25%), campene (6.08%) and α -zingiberene (4.32%). The main volatile compounds of 9-month cultivars are geranial (28.31%), neral (15.29%), β-phellandrene (13.32%), campene (7.77%), α-zingiberene (6.17%) and α-pinene (3.47%). A higher undecanone concentration (0.27%) was found in 6-month cultivars when compared to 9-month cultivars (0.17%) which is in an agreement with previous research [1]. Ginger essential oil was also shown to be a combination of sesquiterpenic compounds that contain α -zingiberene, β -sesquiphellandrene, neral and geranial as well as monoterpenoid compounds with camphene, geranial and geranyl acetate as main constituents [2]. Ginger harvested at 6 months and 9 months showed higher monoterpene content than sesquiterpene content. The data clearly shows ginger harvested at 9 months has more main volatile compounds than at 6 months. Higher contents of geranial (28.31%), neral (15.29%), β -phellandrene (13.32%), campene (7.77%), α-zingiberene (6.17%), α-farnesene (3.30%), β sesquiphellandrene (2.72%) and ar-curcumene (1.94%) were found in ginger harvested at 9 months in comparison to the ginger harvested at 6 months. The low molecular weight of monoterpenes:

Table 2. Essential oil composition of ginger cultivars harvested at 6-month and 9-month maturity using GC-MS and GC-FID.

No.	Compound	Composition (%)		RIa	Identification
		6-month	9-month		
		maturity	maturity		
1	tricyclene	0.09	0.16	927	RI
2	α- pinene	3.16	3.47	938	RI
3	camphene	6.08	7.77	951	RI
4	4-carene	-	0.12	-	MS ^b
5	β- pinene	0.54	0.59	976	RI
6	myrcene	3.26	3.17	990	RI, MS
7	α- phellandrene	0.36	0.46	1007	RI, MS
8	β-phellandrene	10.25	13.32	1032	RI
9	cineole	0.28	-	1034	RI
10	2-cymanene	0.61	-	1086	RI
11	2-nonanone	3.93	1.59	1093	RI
12	3,3-dimethyl-1octene	0.18	0.20	1151	RI, MS
13	citronellal	-	0.85	1154	RI, MS
14	iso borneol	0.44	-	1162	RI
15	borneol	0.33	0.11	1162	RI, MS
16	α -terpineol	0.89	0.81	1189	RI, MS
17	citronellol	0.66	0.96	1229	RI, MS
18	neral	7.65	15.29	1226	RI, MS
19	3-carene	10.53	-	-	MS
20	nerol	13.34	2.58	1255	RI, MS
21	bornyl acetate	0.16	0.16	1272	RI, MS
22	2-undecanone	0.27	0.17	1285	RI, MS
23	citronellyl propionate	0.99	-	1255	RI, MS
24	geranial	24.20	28.31	1272	RI, MS
25	β- elemene	-	0.22	1337	RI
26	citronellyl acetate	-	1.66	1358	RI
27	copaene	-	0.08	1388	RI
28	geranyl acetate	-	0.51	1381	RI
29	ar-curcumene	0.95	1.94	1393	RI, MS

Table 2. (cont.)

30	chamigrene	0.36	0.15	1478	RI
31	zingiberene	4.32	6.17	1487	RI, MS
32	e-e-α- farnesene	2.39	3.30	1494	RI
33	γ-cadinene	-	0.18	1514	RI
34	β -sesquiphellandrene	1.31	2.72	1507	RI
35	selina-3,7(11)-diene	0.14	-	1384	RI
36	trans-cadinene ether	0.09	-	1400	RI
37	hexadecane	0.50	-	1447	RI
38	β-bisabolene	0.15	1.33	1456	
39	γ-elemene	-	0.14	1586	RI
40	β-gurjunene	-	0.12	1596	RI, MS
41	β-cadin-4-en-10-ol	0.53	0.14	1627	RI, MS
42	caryophyllene	0.17	0.57	1638	RI, MS
43	β-eudesmol	-	0.61	1662	RI, MS
44	trans-α-bergamotene	0.54	0.16	1693	RI, MS

^aRetention indices, ^bmass spectral data

α-pinene, cineole, borneol, neral and geranial are shown in various proportions leading to aroma characteristics in the products. For instance, citral with its two isomers neral and geranial, was found high (between 6.6-20.7% citral) in different areas of Brazilian-grow cultivars while it was found only 1.9-4.3% in Chinese cultivars [19]. Australian oils also had high citral content (27%) with an average of 19% [20, 21]. The citral content affecting the lemony odor of ginger is popularly used in the food manufacturing [22]. In summary, the citral content increased with the age of rhizomes and is higher than zingiberene at 9-month maturity. These data indicated that the volatile compounds of each cultivated ginger depend on the age of rhizome, environmental origin, and climate surroundings. Cultivars of ginger at 9 months contained significantly higher concentrations of volatile compounds than 6-month cultivars and duration of 8-9 months is suitable for essential oil production [10].

Our HPLC method allows the finding of all four ginger's pungency at both maturity periods (Figure 1). The content of 6-gingerol increased with the age of the rhizome significantly ($p \le 0.05$). The content in 9-month sample ($24.36\pm0.56\,\text{mg/g}$) was higher than the 6-month sample ($21.55\pm2.80\,\text{mg/g}$). This suggests that manufacturers should purchase ginger rhizome with higher level of 6-gingerol throughout the growth period. However, the previous research found that 6-gingerol content directly associated with oleoresins amounts and reduced by the effects of long term planting with an exception in some locations [1]. In Hawaii, 6-gingerol in rhizomes increased over period examined, while it reached a peak at 4 months on a dry matter after planting, then decreased and increased again to reach a second peak at 6 months [23]. Similarly, the maximum oleoresin content was reached after 7 months. Time from planting to maturity may be highly affected by the type of soil [24]. High levels of 6-shogoal were detected in 6-month samples ($9.02\pm1.01\,\text{mg/g}$) compared to low levels in 9-month samples ($5.59\pm0.09\,\text{mg/g}$). Shogaol is often found at low

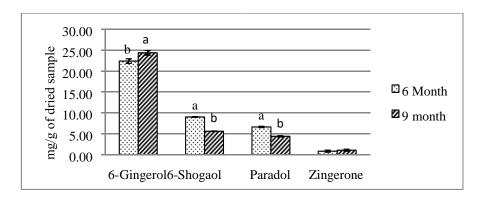


Figure 1. Variations in 6-gingerol, 6-shogaol, paradol and zingerone contents of ginger during maturation (6 and 9 months).

concentration in fresh ginger during long term storage of dried ginger [14]. The 6-month samples contained higher levels of paradol (6.62±0.07 mg/g) than the 9-month samples (4.38±0.16 mg/g). Zingerone is found in low level in fresh ginger and is also produced from gingerol during the process; this compound is less pungent and has a spicy aroma [25]. Contents of zingerone observed in the samples were not significantly different in both maturations. In summary, fiber and the levels of volatile compound and bioactive compound are the most main criteria for the determination of suitable ginger rhizomes for production [26]. The best time to harvest ginger for the extracts of high quality is 9 months. By allowing for a longer harvesting time, it will ensure maximum returns for manufacturers. Therefore, we selected 9 -month maturity ginger for further investigation because the unique aroma of this cultivar comprises significantly higher levels of pungent 6-gingerol, which create commercial interest of aroma ingredients [27].

3.2 Effects of physicochemical properties of different drying temperatures

The drying curves of ginger using a tray dryer to achieve moisture content of 10% are shown in Figure 2. Drying times to achieve moisture content below 10% were 510, 394 and 308 min for 40°C, 50°C and 60°C, respectively. The experimental results showed that drying at high temperature decreased the moisture content faster than at low temperature, resulting in shorter drying time.

From Table 3, both L*(brightness/darkness) and b*(yellowness/blueness) values were affected by thermal treatment. With high temperature the ginger turned darker with a slight yellowness with lower L* and higher b* because the browning of the ginger was greater at a higher drying temperature [5]. The color changes caused by heat processing may have affected the nonenzymatic browning of food and the deterioration of pigment in the food [28]. The proximate compositions of the sample studied are also presented in Table 3. The dried ginger exhibited water activity between 0.307-0.387 which was in an alignment with dried food specifications (a_w <0.6). The moisture content of the dried ginger ranged between 7.01-8.39% (wet basis) conforms to Thai agricultural standards of dried ginger powder which is limited to 10% [29]. Furthermore, there were no significant changes in the composition of carbohydrate (73.16-73.93%), protein (9.509.53%), fat (2.22-2.25%) and fiber (9.11-9.13%) after drying temperature was increased. The ash content decreased significantly (p<0.05) from ginger subjected to higher temperature drying, which reduce the micronutrients represented in the investigation of the ash [30]. The previous reported

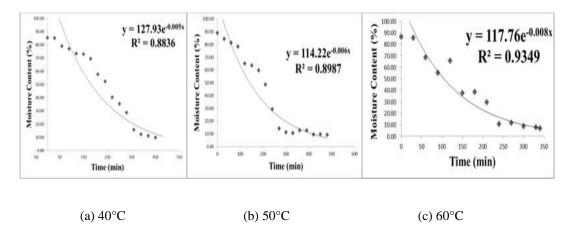


Figure 2. Drying time of ginger at different temperatures (a) 40°C (b) 50°C (c) 60°C

Table 3. Physicochemical properties of dried ginger at different drying temperatures.

Characteristics	Drying Temperature (°C)		
	40	50	60
L*	81.23 ± 0.25^{a}	78.03 ± 1.81^{b}	$75.15 \pm 1.46^{\circ}$
a*ns	3.53 ± 0.85	2.19 ± 0.49	2.89 ± 0.57
b*	15.65 ± 0.12^{c}	21.61 ± 1.22^{b}	24.53 ± 1.10^{a}
Water activity	0.387 ± 0.027^{a}	0.378 ± 0.031^{a}	0.307 ± 0.014^{b}
Moisture (% wet basis)	8.39 ± 0.92^{a}	8.13 ± 0.39^{ab}	7.01 ± 0.10^{b}
Carbohydrate (%dry basis) ^{ns}	73.16 ± 0.72	73.38 ± 0.49	73.93 ± 0.74
Protein (%dry basis) ^{ns}	9.51 ± 0.27	9.50 ± 0.15	9.53 ± 0.26
Fat (%dry basis) ^{ns}	2.25 ± 0.22	2.22 ± 0.06	2.23 ± 0.16
Fiber (%dry basis) ^{ns}	9.11 ± 0.38	9.14 ± 0.33	9.13 ± 0.20
Ash (%dry basis)	5.96 ± 0.17^{a}	5.75 ± 0.07^{b}	5.19 ± 0.20^{b}
Phenolic content (µmol Tannic acid/g)	9.66 ± 1.01^{b}	10.34 ± 0.94^{b}	12.21 ± 0.22^{a}
DPPH (%inhibition) ^{ns}	92.07 ± 0.54	92.02 ± 0.54	91.35 ± 0.50
ABTS (µmol Trolox/g)	$134.80 \pm 22.80^{\circ}$	127.35 ± 5.13^{b}	223.50 ± 18.81^{a}
FRAP (µmol Trolox/g)	35.30 ± 2.70^{b}	$23.64 \pm 0.86^{\circ}$	42.39 ± 0.78^{a}

a-c Different letters mean significant difference between means in the same row (DMRT, p≤ 0.05).

ns letters show non-significant difference between means in the same row (DMRT, p>0.05).

values for composition of ginger (dry weight basis) were 72.84% for carbohydrate, 6.58% for protein, 5.53% for fat, 6.40% for ash and 8.30% for fiber [31]. In the present report, protein and fat contents were slightly changed from previous research.

The highest total phenolic content was observed in drying at 60°C (12.21±0.22 µmol tannic acid/g dry basis) and the lowest was observed in drying at 40°C (9.66±1.01 µmol tannic acid/g dry basis). This can be explained by the rehydration process causing the release of phenols from the cell walls and an increase in free hydroxyphenols and the transformation of new composites such as shogaol and zingerone [32]. For optimal phenolic content, shorter time at higher drying temperatures is required while the total phenolic content and antioxidant activities of ginger rhizomes were higher when drying was done at higher temperatures [14]. The DPPH radical inhibition (%) was not different among variables drying temperatures. The values of %inhibition ranged between 91.3592.07%. The ABTS and FRAP radical scavenging activities of ginger slices gave direct effect on total phenolic content. The highest ABTS was observed in drying at 60°C (223.50±18.81 μmol Trolox/g) and was the lowest when observed in drying at 40°C (134.80±22.80 µmol Trolox/g) and at 50° C ($127.35\pm5.13 \mu mol\ Trolox/g$). For FRAP values at 60° C ($42.39\pm0.78 \mu mol\ Trolox/g$) showed significantly (p≤0.05) higher than at 40°C and 50°C. It was observed that drying at various temperatures did not affect the DPPH radical inhibition (%) but rather influenced total phenolic content, ABTS and FRAP in this experiment. Therefore, the highest temperature (60°C) measured can be the most visible since it decreases the time and the costs of processing, resulting in higher total phenolic content and antioxidant activities than other temperatures (40 and 50°C). The usage of high temperatures in extraction and food storage leads to the damage of total phenolic content. However, significant changes were confirmed between the different temperatures and the use of lower temperatures caused a slower loss of antioxidant activities [30]. Some researcher reported that the drying temperature in the range of 60-80°C had no effect on total phenolic content, DPPH and ABTS scavenging inhibitions [33].

In the analysis of volatile compounds of ginger, 63 compounds are identified by GC-MS and GC-FID. The high contents of zingiberene, geranial, β -sesquiphellandrene and ar-curcumene comprise the important aromas of ginger [18]. Different drying temperature caused variations of volatile compounds. In drying at 60°C, 45 compounds were found and the main compounds were α -zingiberene (18.28%), geranial (12.42%), α -farnasene (10.79%), β -sesquiphellandrene (8.61%), neral (8.58%), ar-curcumene (6.09%). In drying at 60°C, zingiberene, α - farnasene, β -sesquiphellandrene, ar-curcumene and β -bisabolene were more prevalent in comparison to other dried samples. It was found that drying at 60°C conserved better levels of sesquiterpenes (zingiberene, farnesene, curcumene, β -sesquiphellandrene and β -bisabolene) while 44 and 46 compounds were detected when drying at 40°C and 50°C, respectively. This indicates that lower temperatures slightly decreased some sesquiterpenes and increased monoterphenes (campene, myrcrene, borneol, neral and geranial). The most geranial contents were found when drying at 50°C. Generally, the mild drying temperature had less effect on the volatile compounds despite its long treatment period whereas the strong drying process exerted significant effect on volatile compounds [34].

The 6-gingerol content was studied with several drying temperatures and the data are presented in Figure 3. The content of 6-gingerol increased as the drying temperature increased. The highest 6-gingerol content was found with drying at 60°C. It was previously assumed that the high temperature and long exposure to heat would accelerate the deterioration and change of 6-gingerol [34]. In the previous research, 6-gingerol seems to decompose as the drying temperature increased to 60°C [35] and transforms into 8-gingerol and 10-gingerol. Another research studied the effect of drying ginger rhizome on 6-gingerol contents and found that a long drying time can reduce the amount of this compound [36]. The 6-shogaol content ranged from 2.28 to 2.54 mg/g and is similar to the results from previous report (about 2-2.5 mg/g in ethanolic extract) [21]. Drying temperature of ginger directly affects the yield of 6-shogaol. The content of 6-shogaol slightly decreased as the

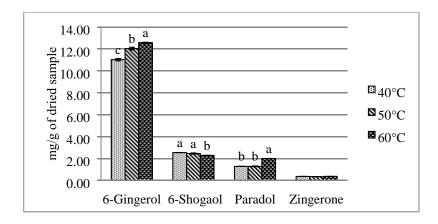


Figure 3. Variations in 6-gingerol, 6-shogaol, paradol and zingerone contents of ginger after drying at 40 °C, 50 °C and 60 °C

temperature increased (60°C). Various drying temperatures have been tested to obtain different bioactive compounds such as paradol. Paradol contents tended to increase at the drying temperature of 60°C (2.00±0.01 mg/g). Zingerone was much more stable than other active compounds during drying and the minimal content ranged from 0.35 to 0.38 mg/g in ginger.

4. Conclusions

In this paper, age and drying temperature were examined as they relate to the physical properties, proximate compositions, total polyphenol contents, antioxidant properties, essential oil compositions and bioactive compounds of ginger. The results found that the main volatile compounds of 9-month ginger were geranial, neral, β -phellandrene, campene, α -zingiberene, α -farnesene. Maximum content of 6-gingerol can be achieved with ginger at 9-month maturation. The optimal drying temperature at 60°C for 308 min in hot air dryer results in a sample rich in phenolic compounds with strong antioxidant. Our findings reveal that shorter drying time conserved major volatile compounds. The major volatile compounds were α -zingiberene, geranial, α -farnasene, β -sesquiphellandrene, neral and ar-curcumene when drying at 60°C. Drying temperature at 60°C gave the highest 6- gingerol content. To produce ginger powder, using ginger which has 9- months of maturation before drying at a temperature of 60°C is recommended.

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References

- [1] Kiran, C.R., Chakka, A. K., Padmakumari, A., Menon, A.N., Kumar, M.M.S. and Venogopalan, V.V., 2013. Influence of cultivar and maturity at harvest on the essential oil composition, oleoresin and [6]-gingerol content from Northeast India. *Journal of Agricultural and Food Chemistry*, 61, 4145-4154.
- [2] Semwal, R.B., Semwal, D.K., Combrinck, S. and Viljoen, AM., 2015. Gingerols and shogaols: Important nutraceutical principles from ginger. *Journal of Phytochemistry*, 117, 554-568.
- [3] Bailey-shaw, Y., Williams, L., Jonor, G., Green, C., Hibbert, S., Salmon, C. and Ann marie, S., 2008. Changes in the contents of oleoresin and pungent bioactive principles of Jamaican ginger (*Zingiber officinale* Roscoe.) during maturation. *Journal of Agricultural and Food Chemistry*, 56, 5564-5571.
- [4] Shirin, AP. and Jamuna, P., 2010. Chemical composition and antioxidant properties of ginger root (*Zingiber officinale*). *Journal of Medicinal Plants Research*, 4, 2674-2679.
- [5] Phoungchandang, S., Nongsang, S. and Sanchai, P., 2009. The development of ginger drying using tray drying, heat pump-dehumidified drying, and mixed-mode solar drying. *Journal of Drying Technology*, 27(10), 1123-1131.
- [6] AOAC., 2000. Official Methods of Analysis. 17th ed. Gaithersberg, MD, USA: Association of Analytical Communities.
- [7] Re, R., Pellegrini, N., Proteggente, A., Pannala, A., Yang, M and Rice-Evans, C., 1999. Antioxidant activity applying an improved ABTS radical cation decolorization assay. *Journal of Free Radical Biology and Medicine*, 26, 1231-1237.
- [8] Benzie, I.F. and Strain, J.J., 1996. The Ferric Reducing Ability of Plasma (FRAP) as a measure of "antioxidant power": The FRAP assay. *Journal of Analytical Biochemistry*, 239, 70-75.
- [9] Kajsongkram, T., Rotamporn, S., Limbunruang, S. and Sirinan, T., 2015. Development and validation of a HPLC method for 6-gingerol and 6-shogaol in joint pain relief gel containing ginger (*Zingiber officinale*). *International Journal of Medicine and Health Science*, 9(12), 813-817.
- [10] Mazaud, F., Rottger, A. and Steffel, K., 2002. *GINGER: Post-Production Management for Improved Market Access*. [online] Available at: https://www.scribd.com/document/209439414/Post-Harvest-Compendium-Ginger
- [11] Jogi, B.S., Singh, I.P., Dua, H.S. and Sukhija, P.S., 1972. Changes in crude fibre, fat and protein content of ginger at different stages of ripening. *Indian Journal of Agricultural Science*, 42, 1011 1015.
- [12] Nybe, E.V., Nair, P.C.S and Mohankumaran, N., 1982. Assessment of yield and quality components of ginger. In: Nair, M.K., Premkumar, T., Ravindran, P.N. and Sarma, Y.R., *Proceedings of National Semiliar on Ginger and Turmeric*, Kasaragod, India: Central Plantation Crops Research Institute.
- [13] Ratnambal, M.J., Gopalam, A. and Nair, M.K., 1987. Quality evaluation in ginger (*Zingiber officinale Rosc.*) in relation to maturity. *Journal of Plant and Crops*, 15, 108-117.
- [14] Chumroenphat, T., Khanprom, I. and Butkhup, L., 2011. Stability of phytochemicals and antioxidant properties in ginger with different drying methods. *Journal of Herbs, Spices and Medicinal Plants*, 17(4), 361-374.
- [15] Harish, R. and Shivanandappa, T., 2006. Antioxidant activity and hepatoprotective potential of *Phyllanthus niruri*. *Journal of Food Chemistry*, 97, 122-129.
- [16] Hassimotto, N., Genovese, M. and Lajolo, F., 2005. Antioxidant activity of dietary fruit, vegetables, and commercial frozen fruit pulps. *Journal of Agricultural Food Chemistry*, 53(8), 2928-1935.

- [17] Chen, I., Chang, C., Wang, C., Shyu, Y. and Chang, T., 2008. Antioxidant and antimicrobial activity of Zingiberaceae plants in Taiwan. *Journal of Plant Food Human Nutrition*, 63(1), 15-20.
- [18] Teferra, T., Kifle, A., Tefera, A., Abate, S., Sima, B., Astatike, A., Habte, T and Samuel, A., 2015. *Ginger (Zingiber Oficinale Rosec.): Production, Postharvest Handling, Processing and Marketing A Comprehensive Extension Package Manual.* [online] Available at: https://www.researchgate.net/publication/279442041_Ginger_Zingiber_Oficinale_Rosec_Production_Postharvest_Handling Processing and Marketing A Comprehensive Extension Package Manual
- [19] Lawrence, B.M., 2000. Progress in essential oils. Ginger oil. *Perfumer and Flavorist*, 25(2), 55-58.
- [20] Connel, D.W. and Jordan, R.A., 1971. Composition and distinctive volatile flavor characteristics of the essential oil from Australian-grown ginger (*Zingiber officinale*). *Journal of the Science of Food and Agriculture*, 22, 93-95.
- [21] Weiss, E.A. 1997. Essential Oil Crops. Oxon: CAB International Publishing.
- [22] Kiran, C.R., Chakka, A.K., Amma, K.P.P., Menon, A.N., Kumar, M.M.S. and Venugopalan., V.V., 2013. Essential oil composition of fresh ginger cultivars from North-East India. *Journal of Essential Oil Research*, 25(5), 380-387.
- [23] Baranowski, J.D., 1986. Changes in solids, oleoresin, and (6)-gingerol content of ginger during growth in Hawaii. *Journal of Horticulture Science*, 21(1), 14-146.
- [24] Weiss, E.A., 2002. Spice Crops. Oxon: CAB International Publishing.
- [25] Bhatt, N., Waly, M., Musthafa, M.E. and Ali, A., 2013. *Ginger: A Functional Herb*. New York: Nova Science Publishers.
- [26] Purseglove, J.N., Brown, E.G., Green, C.L. and Robbins, S.R.J., 1981. *Spices*. London: Longman.
- [27] Wohlmuth, H., Leach, D.N., Smith, M.K. and Myers, S.P., 2005. Gingerol content of diploid and tetraploid clones of ginger (*Zingiber officinale* Roscoe). *Journal of Agriculture and Food Chemistry*, 53(14), 5772-5778.
- [28] Yussef, K.M. and Mokhtar, S.M., 2014. Effect of drying methods on the antioxidant capacity, color and phytochemicals of *Portulaca oleracea* L. Leaves. *Journal of Nutrition and Food Sciences*, 4(6), 1-6.
- [29] National Bureau of Agricultural Commodity and Food Standards., 2015. *Good agricultural practices for ginger*. [online] Available at: http://www.acfs.go.th/standard/download/eng/GAP-GINGER-ENG.pdf
- [30] Reis, R.C., Castro, V.C., Devilla, I.A., Oliveira, C.A., Barbosa, L.S. and Rodovalho, R., 2013. Effect of drying temperature on the nutritional and antioxidant qualities of cumari peppers from Pará (*Capsicum chinense Jacqui*). *Brazilian Journal of Chemical Engineering*, 30(2), 337-343.
- [31] Nwinuka, N.M., Ibeh, G.O. and Ekeke, G.I., 2005. Proximate composition and levels of some toxicants in four commonly consumed spices. *Journal of Applied Science and Environmental Management*, 9(1), 150-155.
- [32] Balladin, D.A., Chang-Yen, I., McGaw, D.R. and Headley, O., 1996. Solar drying of West Indian ginger (*Zingiber officinale* Roscoe) rhizome using a wire basket dryer. *Journal of Renewable Energy*, 7(4), 409-418.
- [33] Phowong, C., Thuwapanichayanan, R. and Jaisut, D., 2012. Effect of drying temperatures on drying kinetics, total phenolic content, antioxidant activities and color of ginger. *Journal of Agricultural Science*, 43(3), 460-463.
- [34] An, K., Zhao, D., Wu, J., Xu. Y. and Xiao, G., 2016. Comparison of different drying methods on Chinese ginger (*Zingiber offinale* Roscoe): Changes in volatiles, chemical profile, antioxidant properties, and microstructure. *Journal of Food Chemistry*, 197, 1292-1300.

- [35] Ok, S. and Joeng, W., 2012. Optimization of extraction conditions for the 6-shogaol-rich extract from ginger (*Zingiber officinale* Roscoe). *Journal of Preventive Nutrition and Food Science*, 17(2), 166-171.
- [36] Puengphian, C. and Sirichote, A., 2008. [6]-gingerol content and bioactive properties of ginger (*Zingiber officinale* Roscoe) extracts from supercritical CO₂ extraction. *Asian Journal of Food and Agro-Industry*, 1(01), 29-36.
- [37] Lee, S., Khoo, C., Halstead, C.W., Huynh, T. and Bensoussan, A., 2007. Liquid chromatographic determination of 6-, 8-, 10-gingerol, and 6-shogaol in ginger (*Zingiber officinale*) as the raw herb and dried aqueous extract. *Journal of AOAC International*, 90(5), 1219-1226.