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Research article

Effect of microwave drying on drying characteristics, volatile compounds and color of holy basil (*Ocimum tenuiflorum* L.)

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Abstract

Microwave is expected to be a novel method for drying of holy basil leaves. This study evaluated its drying efficiency and effects on aroma characteristics of the basil. Microwave drying was conducted on holy basil leaves, with the drying characteristics, volatile compounds and color properties compared to those of freeze drying. A constant rate in the drying period was used for microwave drying. In both drying methods, the Page model provided a good fit to the changes in the moisture content (coefficient of determination = 0.996–0.998). The kinetic value increase with microwave power indicated a quadratic function. The major volatile flavor compounds detected using gas chromatography-mass spectrometry were α-humulene, α-selinene, methyl eugenol and eugenol. These compounds were enhanced during the drying process but then declined during storage for 4 wk. Microwave-dried basil had the largest decline (e.g., the peak area of α-humulene declined from $15.2\pm2.6\times10^7$ to $1.7\pm0.1\times10^7$). The color change of freeze-dried basil leaves was very noticeable while that of microwave-dried leaves was subtle. Overall, it was shown that microwave drying could be used commercially in the preparation of basil leaves, as the technique was inexpensive, easily applied and capable of maintaining leaf color better than freeze drying.

Introduction

As a consequence of globalization, national cuisine of many countries has gained popularity around the globe. Consequently, there is a requirement to develop preservation methods for authentic ingredients to supply international demands. Thai food is ranked in the top 10 most popular cuisines worldwide (Zoe, 2018). Recently, the global estimate of Thai restaurants was approximately 14,900 (Thairath Online, 2016). As a result, various herbs and spices required for use as raw cooking materials are being exported to many different countries. Holy basil (*Ocimum tenuiflorum* L.) is a traditional herb used in Thai cuisine, preferred for enriching the flavor of dishes. However, holy basil deterioration and dehydration immediately after harvesting impair its flavor and appearance. Drying is an effective method for maintaining the quality of herbs, as dried products are safe from putrefaction due to the reduction in water activity (Ratti, 2001; Zotarelli et al., 2012). Therefore, it is possible to store dried products for long periods (at least several months). As a result of drying, the reduced weight and volume of the products also make it possible to readily transport large amounts of dried herbs. Furthermore, transport vibration and temperature damage and gas control for reduced respiration need not be considered for dried products.

Hot air drying is a common technique used for fruits and vegetables (Imaizumi et al., 2015). Although this technique is inexpensive, the dried objects are exposed to high temperatures for long periods which leads to the decomposition of nutrients and releases volatile compounds from the products (Pirbalouti et al., 2013; Orikasa et al., 2014; Telfser and Galindo, 2019). Freeze drying is often used

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to maintain the quality of dried herbs. This method can remove water without applying a high temperature by lowering the evaporation temperature through reduced pressure (Ratti, 2001). Products usually maintain very good quality due to the low processing temperatures and removal of oxygen (Lenaerts et al., 2018). However, this method involves a long drying process and high energy consumption to maintain the low temperature and vacuum, resulting in higher operating costs (Ratti, 2001).

Recently, microwave drying has gained interest due to its reduced drying times and retainment of product quality (Funebo and Ohlsson, 1998; Maskan, 2001; Orikasa et al., 2018; Ando et al., 2019b). For example, Wang et al. (2019) showed that microwave-assisted drying greatly improved the drying rate of shitake mushrooms. Additionally, Lenaerts et al. (2018) succeeded in improving the sensory quality and chemical components of black tea through microwave drying. A microwave electric field penetrating a dielectric object is converted into heat due to dielectric loss (Yoshida et al., 2017). As this involves an internal heating process, heat loss is extremely low compared to that of external heating and the energy efficiency is high (Imaizumi et al., 2013). Thus, microwave heating can accomplish a rapid temperature increase in an entire object over a shorter drying time. In addition, a shorter drying time generally leads to reduced decomposition of nutrients (Imaizumi et al., 2015). Only a few studies have reported on microwave drying of holy basil leaf. In the current study, microwave drying was applied to holy basil leaf, with the drying kinetics analyzed using the Page model. This model has been widely utilized for the thin layer drying of biological materials such as spinach leaves (Doymaz, 2009) and bay leaves (Demir et al., 2004). Additionally, the quality of microwave-dried leaves was compared to that obtained using freeze-drying.

Materials and Methods

Sample preparation

Holy basil leaves (initial moisture content: 6.52 kg water/kg dry matter) were harvested from a farm at King Mongkut's University of Technology Thonburi, Bangkok, Thailand. The basil leaves were cut from the stems, then washed with tap water. After washing, excess water was removed using a salad hand spinner and the leaves were used immediately for experiments.

Drying characteristics

The drying characteristics of the basil leaves were investigated based on weight changes during the drying process. For microwave drying, 30 g of basil leaves were placed on a dish in a microwave oven (MG23F301EAS; Samsung, Seoul, South Korea), and dried at 300 W, 450 W, 600 W and 800 W. For freeze drying, 30 g of basil leaves were pre-frozen in liquid nitrogen and then placed in a freeze dryer with a 6-port manifold (Labconco Corp.; Kansas City, MO, USA). To evaluate the drying characteristics of basil leaves, changes in leaf weight were measured using an electric balance (OHAUS Corp.; Parsippany, NJ). In each drying process, the samples were weighed every 8.33×10^{-3} h (30 s) and every 1 h for microwave drying and freeze drying, respectively. Weight changes were converted into changes in moisture content (kilograms water per kilogram dry matter).

Changes in the moisture content during drying are often expressed using mathematical models (Imaizumi et al., 2015; Orikasa et al., 2014). In this study, a Page model was applied, as shown in Equation 1 (Simal et al., 2005):

$$\frac{M - M_e}{M_0 - M_e} = \exp(-kt^n) \quad (1)$$

where M is the moisture content (kilograms water per kilogram dry matter), k is a rate constant (hr^{-n}), t is the drying time (hours), n is a constant and the subscripts e and 0 express equilibrium and initial values, respectively. Three replicates of this experiment were performed. The relationship between the microwave power and the rate constant was fitted to an arbitrary quadratic function by the least-squares method.

Quality evaluation of dried basil leaves

Volatile compounds and color were measured to compare the effects of the drying methods on the quality of dried basil. As described before, basil leaves were dried using microwave drying at 800 W and freeze drying for 6.67×10^{-2} h (4 min) and 13 h, respectively. Then, the dried leaves were stored in polyethylene zip bags at room temperature. The measurements were conducted immediately after drying (W0) and after storage for 1 wk (W1), 2 wk (W2) and 3 (W3) wk.

Volatile compounds

The volatile compounds from the dried basil leaves were analyzed in accordance with Laohakunjit et al. (2017). Gas chromatography-mass spectrometry (GC-MS) (7890A GC/5975C MS; Agilent Technologies; Santa Clara, CA, USA) with headspace solid-phase micro-extraction (SPME) was used to analyze the volatile compounds in the basil leaves. Each sample (0.25 mg) was prepared in a 20 mL vial. Extraction of headspace volatile compounds was performed using an SPME fiber (50/30 µm DVB/CarboxenTM/PDMS StableFlexTM200; Supelco Inc.; Bellefonte, PA, USA) for 20 min using the autosampler of the Agilent GC-MS system. Compounds adsorbed by the fiber were desorbed in the injection port of the gas chromatograph for 5 min at 240°C, with the purge valve off (splitless mode). The compounds were separated using a DB-5ms capillary column (30 m × 0.25 mm, 0.25 µm film thickness; J&W Scientific Inc.; Folsom, CA, USA). Helium was used as the carrier gas with a constant flow rate of 0.5 mL/min. The GC was equipped with a mass spectrometer and CTC CombiPAL autosampler (Agilent Technologies; Basel, Switzerland) for SPME sample collection. The GC oven temperature program was maintained at 50°C for 1 min, ramped to 210°C at 5°C/min and then held at 210°C for 2 min. Data analysis was performed using the MSD ChemStation E.02.00.493 software (Agilent Technologies; Santa Clara, CA, USA). Volatile compounds were identified through comparison with mass spectra from the Wiley 275 and NIST libraries using a percentage quality match > 90%.

Color

The color of the basil leaves was measured using a colorimeter (CR-300; Konica Minolta; Tokyo, Japan). Measured points were selected at random; each measurement was performed in triplicate. The measured values were expressed as L^*, a^* and b^* values. Additionally, the color difference (ΔE^*ab) was calculated using Equation 2 below:

$$\Delta E^*ab = \sqrt{(L_0 - L^*)^2 + (a_0 - a^*)^2 + (b_0 - b^*)^2} \quad (2)$$

where subscript 0 indicates the initial color value (fresh sample).

Statistical analyses

Each experiment was conducted in triplicate and the data were presented as mean ± SD. Data were analyzed using one-way analysis of variance. Then the Student's t test was applied to test intraday differences between microwave and freeze drying, while interday differences were compared using the Tukey-Kramer method. All statistical analyses were conducted using the R (ver. 3.4.3.) software (R Core Team, 2017).

Results and Discussion

Drying kinetics of basil leaves

Fig. 1 displays the changes in the moisture content during microwave drying and freeze drying. Although freeze drying required 13 hr to reach a moisture content < 0.1, only a few minutes were required for the same level of drying using the microwave method. Furthermore, drying proceeded more rapidly at a higher power (800 W) microwave setting. Drying rates were calculated from the changes in moisture content data. Fig. 2 shows the relationship between the drying rate and moisture content of the holy basil samples. For freeze drying, the drying rate decreased linearly from the initial moisture content, whereas for microwave drying a constant drying rate was maintained up to a moisture content of approximately 2.0 kg water/kg dry matter. During this period, moisture transferred from inside the material to the surface earlier than that of surface evaporation (Nakano et al., 2015). During microwave heating, the sample surface is harder to heat than the inside of the sample (Imaizumi et al., 2013). Therefore, surface hardening, which leads to drying rate reduction in conventional drying (Imaizumi et al., 2015), was considered to be suppressed and thought to contribute to improved moisture mobility due to destruction of the internal structure (Ando et al., 2019a).

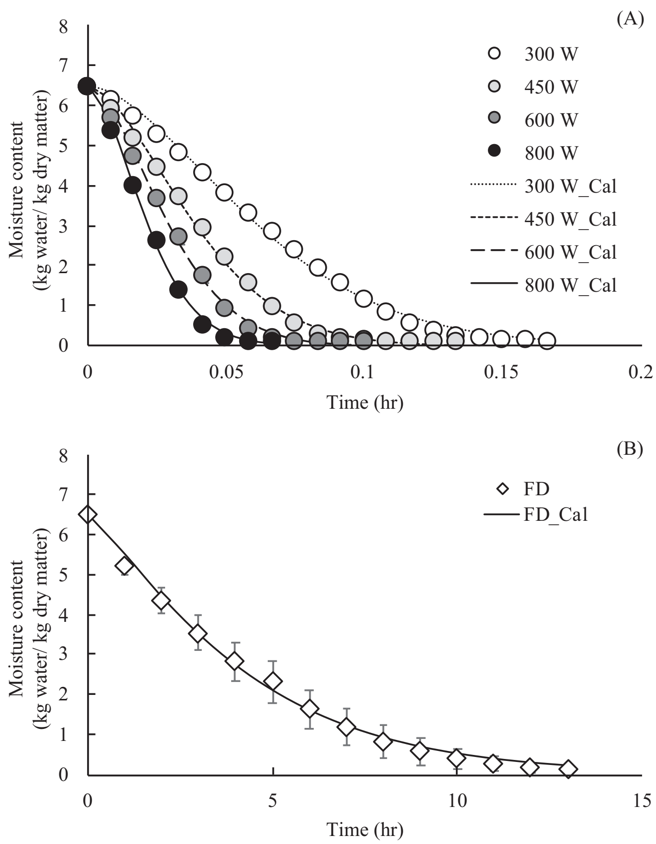

Fig. 1 Changes in moisture content during: (A) microwave drying; (B) freeze drying (FD), where lines are calculated values fitted using a Page model (_Cal) and vertical error bars = ± SD

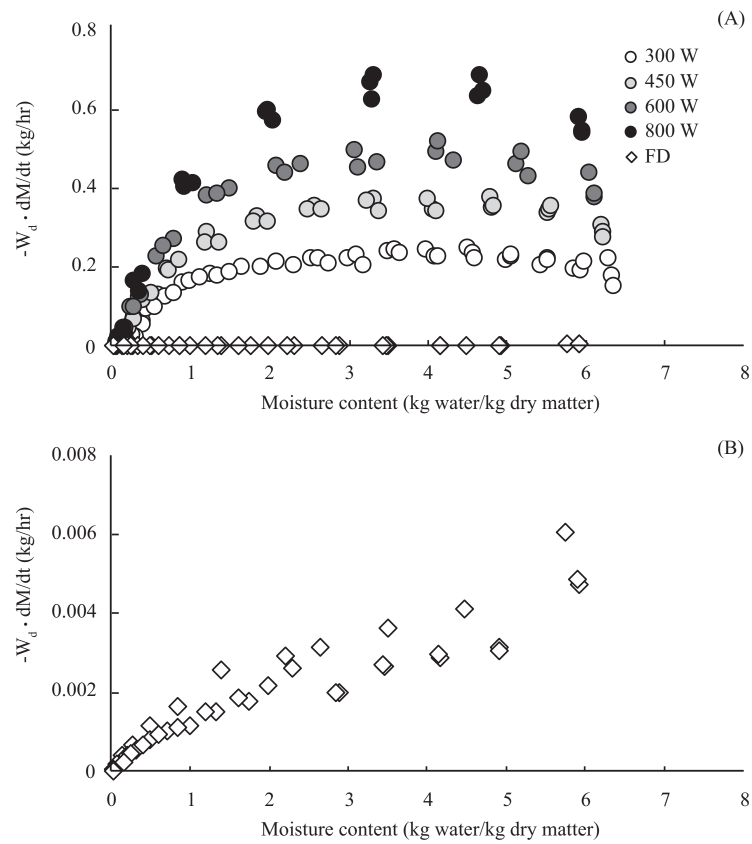

Fig. 2 Relationship between drying rate and moisture content of basil samples: (A) microwave drying at 300–800 W and freeze drying (FD); (B) Freeze drying

The Page model was fitted to the measured values using the least squares method (Fig. 1). A good fit was obtained for each moisture change (coefficient of determination = 0.996–0.998), and the kinetic parameters were determined. Rate constants were 77.3–445.6 hr^{-n} for the microwave method and 0.167 hr^{-n} for freeze drying. Therefore, the microwave method promoted moisture migration over 1,000 times that of freeze drying. Fig. 3 shows plots of the *k* values with respect to microwave oven power. The values were expressed as quadratic functions of the power and indicate that a higher power resulted in a higher drying efficiency. It could be assumed that the wall material, sample tray and air in the oven also absorbed microwaves; however, their relative absorption ratios decreased at higher powers. Based on these results, 800 W was selected for the microwave drying method in subsequent experiments.

Changes in volatile compounds during storage

Table 1 shows the volatile compounds in basil leaves detected using GC-MS before and after drying. The amounts of volatile compounds were compared using a similar method to that of Choi et al. (2019) and Guijarro-Real et al. (2019). In fresh leaves, α-humulene, α-selinene, methyl eugenol and eugenol were confirmed. After drying, these compounds were released either equally or to a greater extent with other natural flavor compounds such as β-pinene, clovene and β-caryophyllene that were also detected. Both drying methods enhanced the aromatic properties. In addition, caryophyllene had the largest peak in the microwave-dried basil leaves, while eugenol had the largest in the freeze-dried leaves. β-Myrcene and (-)-calamenene appeared only in microwave-dried basil, whereas borneol and isoeugenol were found in freeze-dried basil. Qu et al. (2019) indicated that the drying method significantly affected the volatile compound content of black tea and its aroma. Therefore, the flavor of basil most likely also differs based on drying method. Fig. 4 shows changes in the peak areas of β-caryophyllene, α-selinene and eugenol during storage at room temperature. In the freeze-dried leaves, there was no significant decrease in these compounds. Conversely, β-caryophyllene and α-selinene declined sharply in the microwave-dried leaves after 1 wk. Due to this, the flavor of microwave-dried leaves was significantly lower than that of freeze-dried leaves at W3. Additionally, eugenol became the dominant aroma in microwave-dried leaves at W3 because amounts of the other compounds reduced during storage. Tamaki et al. (2012) reported that microwave drying has the effect of increasing the porosity of vegetable tissue. Nathakaranakule et al. (2019) observed that microwave drying created a highly porous structure in durian chips. In the current study, these effects might have influenced the holding power of the volatile components in the basil leaves. To increase the usefulness of microwave drying for basil, it is necessary to develop technologies that enable the retention of aroma compounds even during storage. In the current study, changes in aromatic components during drying and storage were only briefly evaluated. However, in future studies, it is desirable to further clarify these changes through quantitative analysis.

Color changes of basil leaves

Fig. 5 shows the color changes of the dried leaves. Although the lightness (L^*) of freeze-dried leaves showed no significant difference from that of fresh leaves, it was significantly higher than that of microwave-dried leaves. An increased L^* value by freeze drying was also reported for pumpkin

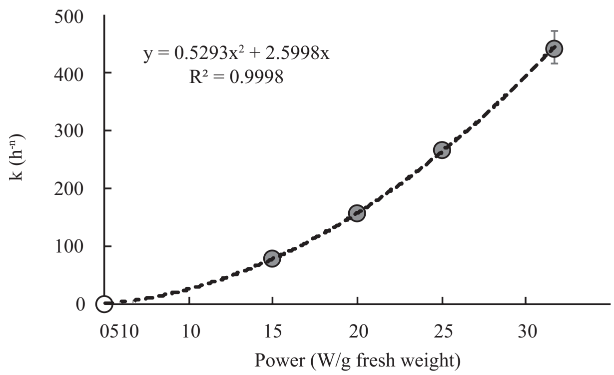

Fig. 3 Plots of *k* value against microwave oven power, where vertical error bars = ± SD
Curve: The quadratic function fitted by the least-squares method

Table 1 Volatile compounds of basil leaves detected using gas chromatography-mass spectrometry before and after drying

No	RT	RI	Compounds	Peak area (×10⁷)		
				Fresh	MW	FD
1	5.9	1025	α-Pinene		3.28	3.22
2	7.3	1076	Camphene		1.76	1.15
3	8.6	1125	β-Pinene		1.90	1.46
4	8.9	1168	(+)-Sabinene			0.647
5	10.6	1192	β-Myrcene		1.52	
6	11.6	1216	(+)-Linonene		2.88	1.08
7	12.0	1254	1, 8-Cineol			1.21
8	20.2	1301	Clovene		0.285	1.32
9	22.7	1353	β-Caryophyllene		178	21.0
10	24.4	1358	α-Humulene	4.96	15.2	3.93
11	25.3	1359	Borneol			1.72
12	25.7	1360	α-Selinene	2.67	25.7	4.28
13	28.1	1367	(-)-Calamenene		0.239	
14	31.9	1380	Methyleugenol	5.36	4.40	3.57
15	34.8	1384	Eugenol	3.51	42.5	71.2
16	38.1	1402	Iso-Eugenol			9.45

RT = retention time; RI = retention index; MW = microwave drying; FD = freeze drying.

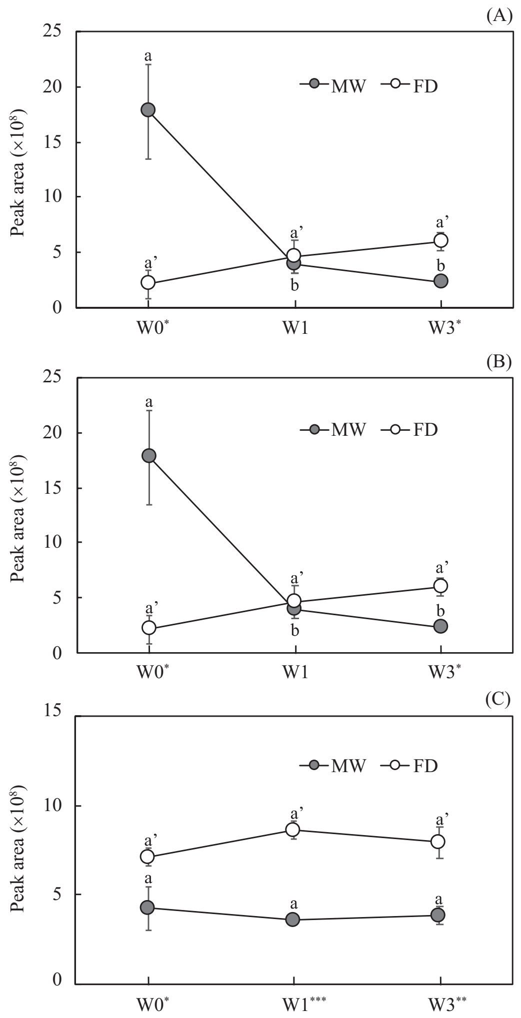

Fig. 4 Changes in peak area of volatile compounds in microwave-dried and freeze-dried samples before (W0) and after storage for 1 wk (W1) and 3 wk (W3): (A) α-humulene; (B) α-selinene; (C) eugenol, where MW = microwave drying; FD = freeze drying; vertical error bars = ± SD; different letters within each drying method indicate significant ($p < 0.05$) differences among the storage periods; asterisks indicate significant differences between microwave drying and freeze drying at each storage period, where * = $p < 0.05$, ** = $p < 0.01$, *** = $p < 0.001$

and green pepper (Guiné and Barroca, 2012). Additionally, the freeze-dried samples exhibited large error bars, leading to concern about the uniformity of quality. Comparatively, microwave drying maintained the same value as fresh leaves throughout storage. The a^* value was increased by drying in both methods and gradually increased during storage. Negative a^* values represent green color, with rising values towards zero representing loss of green coloration. As chlorophyll is a green component of plants, it was inferred that the loss of green coloration was due to chlorophyll decomposition during drying and storage. In addition, the greenness of basil leaves was retained to a greater extent following microwave drying than from freeze drying.

The positive b^* values, indicating yellow color, decreased due to drying, but were maintained during storage. In plants, chlorophyll is present as a mixture of blue-green chlorophyll a and yellow-green chlorophyll b (Püntener and Schlesinger, 2000). Thus, the decrease

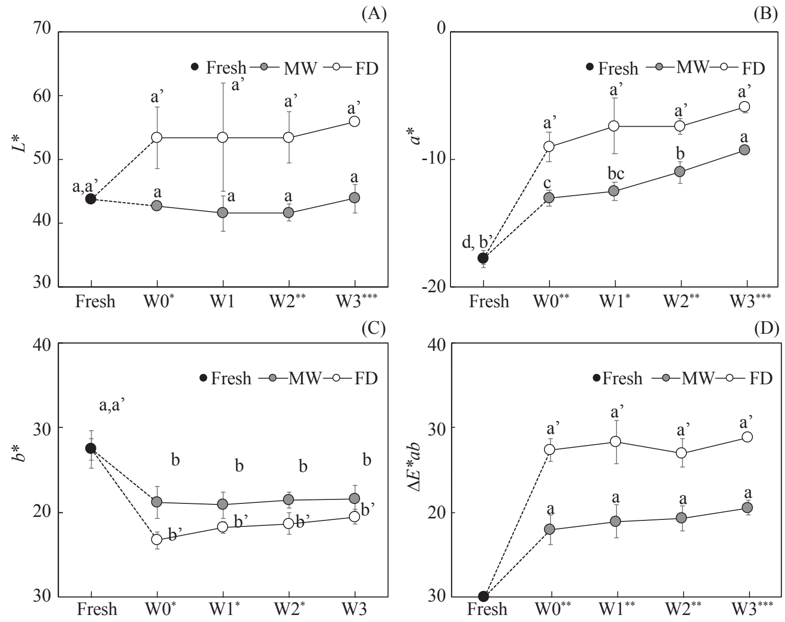

Fig. 5 Changes in color of microwave-dried and freeze-dried samples before (W0) and after storage for 1 wk (W1), 2 wk (W2) and 3 wk (W3): (A) L^*; (B) a^*; (C) b^*; (D) color difference ΔE^*ab, where MW = microwave drying; FD = freeze drying; vertical error bars = ± SD; different letters within each drying method indicate significant ($p < 0.05$) differences among the storage periods; asterisks indicate significant differences between microwave drying and freeze drying at each storage period, where * = $p < 0.05$, ** = $p < 0.01$, *** = $p < 0.001$

in the b^* values was also considered to be related to chlorophyll. The value of ΔE^*ab calculated from these color elements was 8.0–10.5 for microwave-dried leaves and was extremely high for freeze-dried leaves (17.0–18.8). As discussed, color changes of basil leaves might be attributed to degradation of chlorophyll. Decomposition of chlorophyll relates to enzymatic reaction involving chlorophyllase (Tsuchiya et al., 2001). It seemed that this enzymatic reaction was especially high during freeze drying because of the long processing time. Comparatively, microwave heating rapidly inactivates enzymes (Yoshida et al., 2017). Thus, microwave treatment was effective for green color retention in the basil leaves. For both drying methods, changes in color during storage were minimal. In general, enzymatic reactions are suppressed when water activity is low (Lee and Kim, 1995). Since dried products have extremely low water activity, color change was most likely suppressed during storage. Therefore, the color of basil needs to be carefully controlled during the drying process. Although changes in color during drying are inevitable, microwave drying minimized this occurrence in basil leaves.

The current study used microwave drying of basil leaves, and its usefulness was evaluated in terms of drying characteristics, volatile compounds and color change. Microwave drying greatly improved the drying rate compared to that of freeze drying and it is expected to have a great effect on cost reduction. In addition, this effect was enhanced by higher power microwave drying, with 800 W providing the best condition for microwave drying because it maintained the good appearance of the dried basil leaves and reduced the processing time. The flavor of basil leaves is very important for cooking and the current study showed that the volatile components in the basil leaves were not significantly reduced by microwave heating. Additionally, leaf color (deep green) was maintained *via* the microwave method. Therefore, it was concluded that the appearance of microwave-dried products would remain attractive to consumers. Overall, these results suggested that microwave drying can efficiently produce high-quality products, while freeze drying requires a longer time and results in poorer appearance parameters. Further studies will investigate the development of technology to minimize the loss of flavor components in microwave-dried basil during storage.

Conflict of Interest

The authors declare that there are no conflicts of interest.

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