

ORIGINAL RESEARCH PAPER

ENHANCEMENT OF APPEARANCE AND FLAVOR RETENTION OF
DRIED KAFFIR LIME (*CITRUS HYSTRIX* D.C.) LEAVES BY LOW
IMPACT DRYING PROCESS

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The effects of low-impact drying on enhancement of appearance and flavor retention of dried kaffir lime (KL) leaves were investigated. Low-Impact Drying (LTLT) had more uniform drying rate than High-Temperature Drying (HTST). LTLT samples were more preferred for color and higher flavor retention than HTST. Significantly lower ΔE in color and less microstructural pore shrinkages and crushed layers were found in LTLT KL ($p < 0.05$). LTLT samples also had significantly higher flavor retention of desirable compounds (dl-Limonene, β -Citronellol, Geraniol, Citronellyl acetate, B-myrcene and Decanal) than HTST. Thus, LTLT was more promising than HTST with enhancement of appearance and flavor retention for superior high-value dried herbs.

Keywords: kaffir lime leave, low impact drying, low temperature, color retention, flavor retention

Introduction

Kaffir lime (*Citrus hystrix* DC.), belongs to citrus family, and is found abundantly in countries belonging to Southeast Asia and South Asia region. Both the fruit and leaves of this plant are widely used in these countries for medicinal and cooking purposes. Because of a strong and distinct aroma of kaffir lime leaves, they are used as a main ingredient in traditional Thai soups, sometimes as a whole or by cutting them into small pieces (Raksakantong *et al.*, 2012). The kaffir lime tree leaves possess a very strong citrus-like aroma. It is estimated that in the leaves of kaffir lime the essential oil is 0.08 to 0.66% (Phoungchandang *et al.*, 2008;

Waikedre *et al.*, 2010) which is used as a flavoring agent. Citronella is the most abundant odorous compound found in its essential oil while some of the other key odorants are myrcene, sabinene, limonene, β -cetronellol, β -pinene, β -cubebene, isopulegol and linalool (Jirapakkul *et al.*, 2013).

Fresh kaffir lime leaves only have a shelf life of three days hence hot air drying is one of the processes used to preserve them for a longer period of time. It is more effective than solar drying, which is weather dependent with less-constant drying rate. In addition, due to its relatively capital cost, the hot air drying method is usually preferred for manufacturing over other drying methods such as freeze drying, which is much more expensive. Despite the process efficiency of hot air drying, the organoleptic properties including the appearance, color, flavor or rehydrating ratio of dried culinary herbs such as kaffir lime leaves becomes less desirable for consumers as compared to fresh products. It is well known that while hot air drying reduces the growth of microorganism, it also causes biochemical changes in the herbs, altering the qualities of dried products. Many research attempts to find the optimum drying methods or conditions to preserve the superior qualities of dried herbs as well as maintaining the efficiency of drying process. Raksakantong *et al.* (2012) reported about their work on drying of kaffir lime leaves that the types of drying method affected the properties of finished leaves such as the composition of fatty acids, antioxidant level, color as well as the volatile compounds. Phoungchandang *et al.* (2008) also found that the concentration of the most abundant volatile compound, which in their case is citronella, in dried kaffir lime leaves decreased with increasing of drying temperature. Moreover, Jirapakkul *et al.* (2013) reported that drying temperature for hot air drying plays an important role in the volatile compound qualities of dried products such as kaffir lime leaves. However, there is the contradiction about the effect of thermal treatment on the antioxidants in some vegetables that thermal treatments could improve the antioxidants (Chuah *et al.*, 2008). However, heat treatments are also found to be the causes of reduction in the antioxidants levels in vegetables (Chuah *et al.*, 2008; Turkmen *et al.*, 2005; Wachtel-Galor *et al.*, 2008). Hence, not only is the drying method affects the qualities of herbs, but also the temperature could result changes in the physicochemical properties of herbs.

Despite the publications about the volatile compounds and their changes in kaffir lime leaves due to different heat treatments, none has yet explained well on how those volatile compounds and their changes affected the preference of consumers. Moreover, the effect of drying conditions on changes in other properties of products such as destruction in microstructure level, color, and appearance remains unclear. Most of published research either focuses on increasing the process efficiency of drying or identifying the types and amount of volatiles compounds remains in dried products without correlating those factors to consumer preference. Therefore, this study aims to investigate the impact of various drying conditions for hot air drying on the process efficiency, physicochemical and organoleptic properties, and how to enhance the appearance and flavor retention of dried kaffir lime leaves to achieve desirable dried culinary herbs.

Materials and methods

Sample preparation

The fresh kaffir lime leaves were sourced from local markets in Phitsanulok, which locate in the lower-Northern region of Thailand. The maturity of kaffir lime leaves was controlled. Visual sorting of samples was done to select only the leaves with high qualities including color, maturity, glossiness and size. The kaffir lime leaves were equally divided into three portions for the experiments. First portion was kept under frozen condition (-4-0 °C) to retain its original fresh qualities to be used as control. Two other portions were pretreated by hot water blanching and rapidly cooling down before drying in order to help preserve the color and the aroma of the dried leaves (Ratanatriwong and Abu-Ali, 2016). Then, they were divided and processed through two various drying condition which were low impact drying condition and relatively high impact drying condition. The treated samples were kept in aluminium foil bags and stored at -20⁰C before further analyses. In this study all the analyses performed were in triplicates. The analytical results of samples were reported on the dry weight basis.

Drying Processes

The hot-air oven (ABC, A728.002, France) with minimum output of 250W were used for the drying purpose. Four round drying trays with dimensions of 34.5cm x 6 cm were used for each condition. Each tray contained about 400g of pretreated samples. The pretreated sample leaves were spread evenly as a thin layer onto the drying surface with total area of 935 cm². Two drying conditions were used in this study that were 1) a low-impact drying condition in which low-temperature-long-time (LTLT) condition was used with temperature of 40⁰C for 10 hour, and 2) a relatively high temperature drying condition was used, using high-temperature-short-time (HTST) of 60⁰C for 6 hour. Kaffir lime leaves were dried to have target moisture content about 6-8%. Treated samples in each condition were taken out every hour to determine the color, water activity and moisture content. Fresh kaffir lime leaves as control were used to compare the results. Before further analysis the treated samples were kept at -20⁰C.

The moisture content and the water activity determination

The AOAC (2000) method was used for the determination of moisture content and water activity of samples. The infrared moisture analyzer (MB 45, Ohaus, USA) was used for moisture content determination. While the water-activity meter (LabStart-aw, Novasina, Switzerland) was utilized for measuring the water activity of the samples. All the analyses were performed in three replications. Prediction of moisture loss rate was analyzed using linear regression model.

Colorimetric parameters determination

The color measurements were carried out using a colorimeter (Model CR-300, Konica Minolta, Sensing, Inc. Osaka, Japan). The color in CIE system (L^* , a^* , b^* , hue angle and chroma) were reported, and the change of color (ΔE) was calculated using the values of L^* , a^* and b^* in the Hunter-Scotfield equation (Hunter, 1975). Chroma (C) and Hue angle (H^0) were calculated using equations 2-3, respectively.

The colorimeter was calibrated before use against a white standard. The average value from 10 individual measurements were recorded.

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

$$C^* = \sqrt{(a^*)^2 + (b^*)^2} \quad (2)$$

$$H^* = \tan^{-1} \left(\frac{b^*}{a^*} \right) \quad (3)$$

The microstructure of kaffir lime leaves determination

Scanning electron microscope (SEM) Model 1455VP (Leo Electric Systems, Cambridge, UK) was employed to illustrate the microstructure (surface and cross section) of fresh and dried kaffir lime leaves samples. Before SEM identification, all leave samples were prepared by being cut into 10×10 mm size and then freeze-dried. The freeze-dried samples were preserved in desiccators for further experiment. Prior to the experiment, double-sided scotch tape was used on mounted samples and placed separately on sample holder. The inside structure was faced upward and sputter-coated with gold for 2 min (2mbar) prior to observation. Three replications of kaffir lime leaves sample were studied in this analysis. Surface and cross-section pictures of samples were recorded.

Volatile compounds extraction from kaffir lime leaves

The volatile compounds from both treated and untreated leaf samples were extracted using Likens's and Nickerson's apparatus by the Simultaneous Distillation Extraction (SDE) process. Sample of 10-20 g was mixed in a blender for half a minute and then transferred into a 1L round bottom flask having a neck size of 24/40 (scotch Germany), after that half liter of distilled water was added in the blend. The internal standard used was 2-methyl-3-heptanone (200µl) in concentration=7.0 µg/µl. In the 500ml flask of Likens and Nickerson's distillation assembly dichloromethane (200ml) was placed and the simultaneous distillation extraction process was started which lasted for three hours. After the distillation process was completed the dichloromethane (200 ml) was frozen at -20° for one hour to freeze any free water present in it. The extract (200 ml of dichloromethane containing the aroma compounds) was than subjected to purification process using the stream of nitrogen gas at 25°C for two hours until 20 ml of dichloromethane was left (as dichloromethane was evaporated under the nitrogen gas purging at room temperature leaving behind only the volatile compounds). After that it was again kept at -20°C for 1 hour to ensure that any present water was frozen. The 20 mL extract was again purified with nitrogen gas until a final volume of 2 ml was only left behind. The remaining 2ml dichloromethane solution was passed through powdered anhydrous sodium sulphate (2g) and was collected in a (2.5ml) vial. The final sample before injection in the GC machine was kept at -20°C.

Volatile compound profile determination of kaffir lime leaves

The volatile compounds analysis was carried out using a chromatograph (GC-2010) joined with GC/MS-QP2010 (Shimadzu, Japan). The column used for the

volatile compounds separation was DB-5MS column with having the thickness 0.25 μ m, length 30.0m and diameter 0.25 μ m. The scanning process was carried out between 35 to 350 m/z. The column flow rate for the carrier gas (Helium) was maintained at 2ml/minute and a split ratio of 100. Initially the oven temperature was held was 40 $^{\circ}$ C for 5 minutes and then increased to 100 $^{\circ}$ C with a rate of 2 $^{\circ}$ C/min and from 100 $^{\circ}$ C to 225 $^{\circ}$ C the rate of temperature increase was 5 $^{\circ}$ C/min. The temperature of 225 $^{\circ}$ C was kept for 10 minutes and the injector was started at 250 $^{\circ}$ C, respectively. Every time 1 μ l of sample was injected. The internal standard was used for calculating the relative concentrations of the detected volatile compounds. Three compounds including hexanal, l-linalool and citronellal were calculated as the relative concentration of authentic standards. The mass spectrum of detected volatile compounds was compared in the Wiley 7 library for the identification. For the calculation of the Odor activity values (OAVs) the odor threshold level in fresh water was used.

The consumer preference determination of dried kaffir lime leaves

The consumer preference of dried KL leaves in different drying conditions were evaluated by 40 untrained panelists. Panelists assessed dried leaf samples by observing and smelling without tasting the samples in aspects of color, overall appearance, overall flavor and overall acceptance. Student's t-test at 95% confidential level was used to analyze the data.

Statistical analysis

The data collected from every experiment except sensory evaluation was analyzed by ANOVA, analysis of variance with the help of SPSS software version 17.0 (IBM Corporation, New York, NY, USA). Duncan's multiple range tests (DMRT) at a confidence level of 95% ($p < 0.05$) was used to calculate the difference among the samples. The analysis were conducted in triplicates, and results were reported in mean and standard deviation.

Results and discussion

Effect of drying conditions on process efficiency and physico-chemical properties of dried kaffir lime leaves

It is well known that drying condition including drying temperature and drying time plays an important role on drying process efficiency for hot-air drying. Due to the direct relationship between the process efficiency and production cost, most manufacturers focus more on drying process with high process efficiency rather than the qualities of dried product. However, in the case of drying culinary or medicinal herbs which their selling prices directly related to their premium qualities, the effect of drying process on the organoleptic properties including appearance, odor, flavor or tastes of these herbs cannot be ignored. Kaffir lime (KL) leaf is one of the abundant culinary herbs in Thailand. Prior to drying process, all KL leaves were pretreated by hot water blanching and rapidly cooling down in order to preserve color and aromas. Then all KL samples were treated at various drying conditions to obtain dried samples with final moisture content of 6-

8% (Figure 1a-b) and water activity below 0.5 (Figure 2) based on the industrial standard of dried herb.

Drying temperature directly increased drying rate (Figure 1b) which directly reduced drying time (Figure 1a). The moisture contents of dried Kaffir lime leaves treated by both drying conditions decreased as drying temperature and time increased. The moisture content of samples decreased due to the excitation of molecules that the water molecules were in the increased state of excitation when drying temperature increased, resulting in a rising of the distance between molecules and a reduction of the attraction force between them. Therefore, due to higher drying rate, higher drying temperature in HTST condition required shorter time than relatively-low drying temperature in LTLT condition to obtain samples with similar final moisture content. It was due to the fact that high temperature results in a higher moisture loss as the water molecules absorb the heat energy quickly which result in their fast evaporation and leading to higher evaporation rates (Zarein *et al.*, 2015). This was found in Figure 1a-b, and was also supported by the prediction regression models between moisture content of samples (Y_{LTLT} and Y_{HTST}) and drying time (x) for each drying condition (LTLT and HTST) (Equations 4 and 5).

$$Y_{LTLT} = -6.437x + 74.669, R^2 = 0.9784 \quad (4)$$

$$Y_{HTST} = -12.605x + 86.122, R^2 = 0.8818 \quad (5)$$

The prediction model of HTST showed that the rate of moisture content changes during drying time was twice higher than that of LTLT. However, the R^2 of LTLT condition was expressively higher than that of HTST condition, indicating that changing of moisture content during drying in LTLT was much smoother. As drying time increased, the moisture content of LTLT-KL samples gradually decreased whereas that of HTST-KL samples sharply decreased during the 2nd-4th hour of drying before level off (Figure 1a). This was because the relatively lower drying temperature of LTLT condition had less effect on moisture reduction, resulting in smoother drying rate (Figure 1b). On the contrary, this was not the case for HTST condition. The drying rate of HTST during the first 2 hours of drying was not significantly different from that of LTLT. However, the drying rate of HTST during the 2nd-4th hour was significantly higher than LTLT, resulting in an abrupt moisture loss. This was shown in Figure 1b that the drying rate of HTST showed a sharp peak during the 2nd-4th drying hour before leveling off which was due to the fact that the free water was already lost so the moisture reduction after that was from the bounded water in the kaffir lime leaves. The abrupt increase and decrease in the drying rate of HTST greatly affected the negative qualities of final products such as the structure destruction and the pore shrinkage which would be discussed further. On the other hand, LTLT drying condition had relatively smoother changes of drying rate during drying so it resulted in less impact on damaging the final dried samples. The results were in compliance with the findings of (Jirapakkul *et al.*, 2013) that, despite the higher drying rate at high temperature treatments, it produced negative effects on the color, appearance, texture and flavor retention of dried kaffir lime leaves. Thus, a higher drying rate was not always the

best solution for drying herbs, and it did not necessarily guaranteed good product qualities.

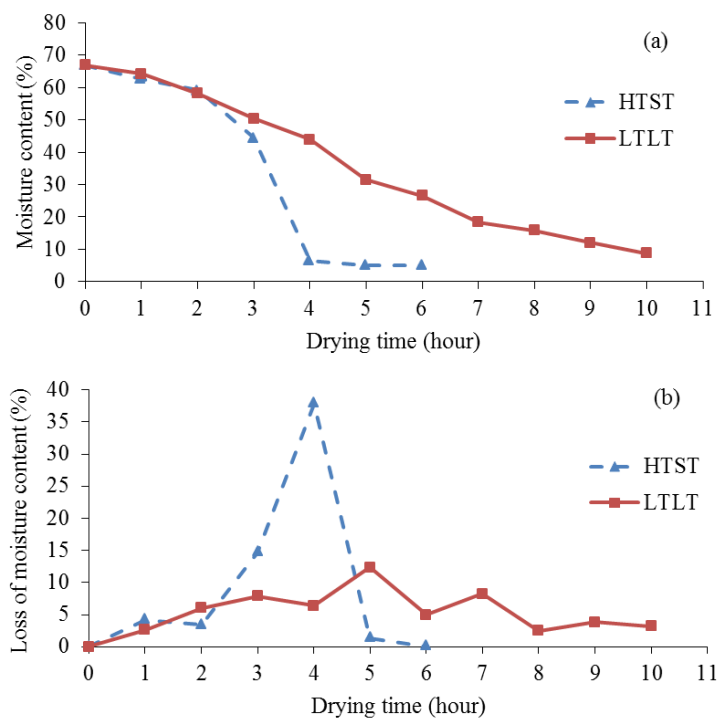


Figure 1. Effect of various drying conditions on reduction of moisture content (a) and drying rate (b) of treated dried kaffir lime (KL) leaves during drying.

Likewise, the water activity (a_w) of kaffir lime leaves decreased with increasing drying time as illustrated in Figure 2. The pattern of reduction in water activity values of HTST-treated sample was considerably different from that of LTLT-treated ones. For LTLT drying treatment, no significant difference among the water activity values of samples taken during the first 6 hours of drying was found; however, after that the water activity values in samples were significantly lower. While for HTST condition, the water activity values of samples during the first two hours were not significantly different; however, it drastically decreased during the 2nd-4th drying hour. This was also supported by the changing of drying rate shown in Figure 1b. Moreover, this indicated that the HTST had a higher drying rate than LTLT condition, resulting in faster and greater moisture reduction in HTST-treated product. This was shown in Figure 2 that the HTST-treated KL leaves had a much lower water activity value than that of LTLT-treated KL leaves even when they had similar moisture content.

Both samples treated with LTLT and HTST conditions had their water activity (a_w) below 0.6, which was below the critical value of mold growth that means all treated samples were safe for consumption. The water activity of a product is a crucial factor which influences the shelf-life of products for example dried

products with the water activity below 0.91 would not have most of bacteria growth (Syamaladevi *et al.*, 2016). The a_w changes illustrated the relationship of shelf-life with moisture content, and the reduction of both moisture content and a_w of dried kaffir lime leaves would extend the shelf life due to the inhibition of the growth of microorganisms. Similar trends in a_w reduction were also stated by Speckhahn *et al.* (2010). Furthermore, water activity also plays a pivotal role in vitamin identification and enzyme activity in foods, and both these factors do impart the taste, aroma and color of products. However, in the case of dried foods if the $a_w \leq 0.1$ then the rate of auto-oxidation increases whereas at the a_w of 0.3, the auto-oxidation rate is the slowest and increases again if the a_w surges from 0.55-0.85. This may be due to the fact of oxygen or catalyst mobilization (Nawar *et al.*, 1996). In this case, the lower value of HTST-treated samples may indicate the higher chances of products having more off-flavor from auto-oxidation during shelf-life than that of LTLT-treated samples even though they contained relatively similar moisture content.

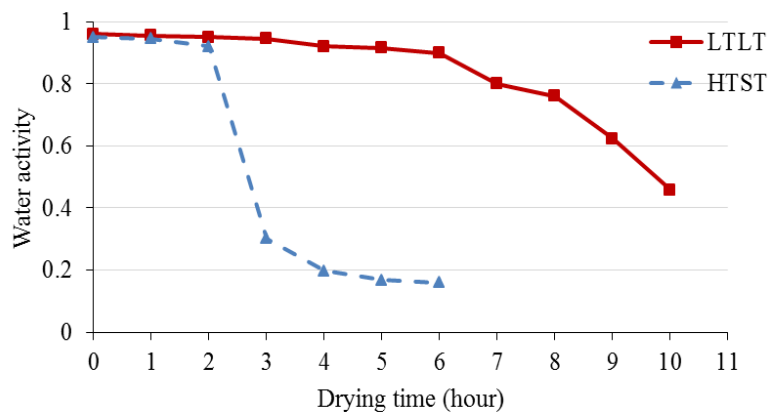


Figure 2. Reduction of the water activity of treated kaffir lime (KL) leaves during drying at various drying conditions.

Effect of drying conditions on color retention of dried kaffir lime leaves

The color of raw and dried food has an excessive effect on the product acceptability by consumer based on visual impression (Perera, 2005) thus it is crucial to choose the drying condition with less impact of color changes of dried products especially culinary or medicinal herbs. Fresh KL leaves were usually dark green with shiny surface on one side with pale colored on the other. For hot-air drying oven, drying conditions greatly affected the color changes of dried kaffir lime (KL) leaves as illustrated in Table 1. All color parameters for final products of both LTLT- and HTST-treated KL samples were significantly different from fresh KL ($p < 0.05$).

The degree of color changes was greatly affected by the drying temperature and time. The high drying temperature greatly affected the color by causing more biochemical damage in samples during drying. Therefore, despite the higher process efficiency with higher drying rate of HTST, it did not have good impact in

terms of preserving color retention for samples. Besides the brightness and yellowness, the other color parameters of LTLT-treated samples were significantly higher than those of HTST-treated samples, and were significantly closer to fresh samples ($p < 0.05$). As overall color, the LTLT-treated samples were brighter (L^*) and greener (a^*) with more color saturation (chroma) than those of HTST-treated samples. This indicated that the LTLT condition as the low impact drying, had higher color retention than HTST with brighter and more-intense-green leaves. This was supported by the value of total color difference (ΔE) shown in Table 1. The difference in color values (ΔE) is widely used to characterize the changes in color of food materials during processing, and is determined from the values from L^* , a^* , b^* parameters. LTLT-treated KL leave had much lower ΔE (40.49) than that of HTST samples (70.04), indicating that the changes of overall color in kaffir lime leaves during LTLT drying condition was much lower. The color changes in the kaffir lime leaves may take place because of non-enzymatic browning or because of the demolition of color pigments present in leaves during the heating process (Raksakantong *et al.*, 2012; Wanyo *et al.*, 2011). Therefore, despite its lower drying rate, using lower temperature in the LTLT drying resulted in a low-impact drying condition which helped preserve desirable color better than HTST drying. Similar pattern of color changes was also reported that using relatively lower drying temperature in hot-air drying resulted in kaffir lime leaves with significantly different ΔE value from those of steamed- or blanched-and-boiled kaffir lime leaves (Ratsewo *et al.*, 2016).

Table 1. Comparison of color parameters of treated kaffir lime leaves at various drying conditions to fresh samples.

Sample	Color Parameter					ΔE
	L^*	a^*	b^*	Chroma (C)	Hue angle (h)	
Fresh	33.61 \pm 0.26 ^b	-11.87 \pm 0.11 ^a	30.88 \pm 0.23 ^a	33.08 \pm 0.23 ^a	111.0 \pm 0.20 ^a	-
LTLT	44.31 \pm 0.89 ^a	-6.220 \pm 1.22 ^b	30.78 \pm 0.48 ^b	31.42 \pm 0.56 ^b	101.4 \pm 2.16 ^b	40.49
HTST	44.00 \pm 0.47 ^a	-5.660 \pm 0.50 ^c	30.31 \pm 0.61 ^b	30.84 \pm 0.65 ^c	100.5 \pm 0.84 ^c	70.04

Values were expressed as means \pm standard deviation.

Means with different letters in the same column were significantly different at $p < 0.05$.

Effect of drying conditions on the microstructure of dried kaffir lime leaves

Besides the physico-chemical properties, color and overall appearance that were significantly affected by HTST- and LTLT-drying conditions, the damage on the microstructure scale of dried KL leaves were evidently observed as well. The overall cross-section microstructures on the side of fresh KL leaves was illustrated in comparison with those of dried LTLT- and HTST-treated KL leaves, respectively (Figure 3a-c) whereas Figure 3A-C focused on the pores and layers of fresh KL leaves in comparison with those of LTLT- and HTST-treated KL samples, consecutively. Fresh KL leaves were relatively thick with wax coated on

the surface. The cross section microstructure of the side of fresh KL leaves showed that there were many pores and layers, acting as the pathway for the volatile compounds and moisture from oil sacs and cells to vaporize which resulted in the odor of KL leaves. After drying in the hot air oven at various drying conditions, it was noticeable that LTLT-treated leaves (Figure 3b) showed less damage in the overall microstructure, and was closer to that of fresh KL leaves (Figure 3a) than that of HTST-treated leaves (Figure 3c). Despite the pore size reduction, many pores and layers with space in-between were evidently observed in LTLT-KL leaves (Figure 3B), which was similar to those shown in fresh leaves (Figure 3a), whereas, in HTST-KL leaves, pore size were greatly reduced, and leave layers were crushed and hardly noticeable (Figure 3C). This helped support that LTLT condition had low impact on qualities of dried KL leaves due to its constant drying rate that minimize the structure damage of the leaves. In contrast, HTST condition with sharp-rising and inconstant drying rate, the leave structure was severely damaged despite its shorter drying time, resulting in less desirable dried KL leaves.

Besides the cross section pictures, the illustration of surface on the microstructure scale of fresh KL leaves (Figure 4a) was depicted in comparison with those of LTLT-treated (Figure 4b) and HTST-treated KL leaves (Figure 4c) with more focus area on the surface of those samples in Figure 4A-C, respectively. The surface of fresh KL leaves was smooth (Figure 4a) and porous with some debris distributed randomly along the minor stems (Figure 4A). Likewise, the LTLT-treated samples showed smooth surface with more debris (Figure 4b) where open pores and the minor stems were still observed (Figure 4B). This was due to milder drying condition in LTLT which help minimize the structure damage in KL leaves. In addition, during drying, liquid diffused to the surface of kaffir lime leaves from the interior and carried some solutes with it (Mujumdar, 2006) so the surface of dried LTLT leaves showed more solutes or debris around the pores of leaves (Figure 5). The LTLT-treated KL samples had slightly decreased in pore size (Figure 5b) when compared to the wider open pore of fresh KL leave (Figure 5a).

This phenomena was similar to others that the drying at a low drying rate, the amount of shrinkage bear a direct relationship with the amount of moisture removal from products since vegetable tissues normally encounter some shrinkages during the drying process. (Wang and Brennan, 1995). However, the pore in HTST-treated KL samples was not observed even using enlarge scale ($\times 3500$). This was because of the harsh effect of high drying temperature in HTST drying condition that led to pore and surface shrinkage while the liquid and other compounds were being diffused too fast. It was illustrated that the HTST-treated sample had severe shrinkage (Figure 4c) with no open pores or minor stems being observed (Figure 4C). The severe pore and surface shrinkage in HTST-treated samples resulted in poor rehydration and poor qualities of final product after being rehydrated as compared to that of LTLT-treated sample (data not shown). This agreed with others that high drying temperatures caused quality losses in dried products whereas using low drying temperatures had the tendency to improve the desirable quality of dried products (Beaudry *et al.*, 2004; Nindo *et al.*, 2003).

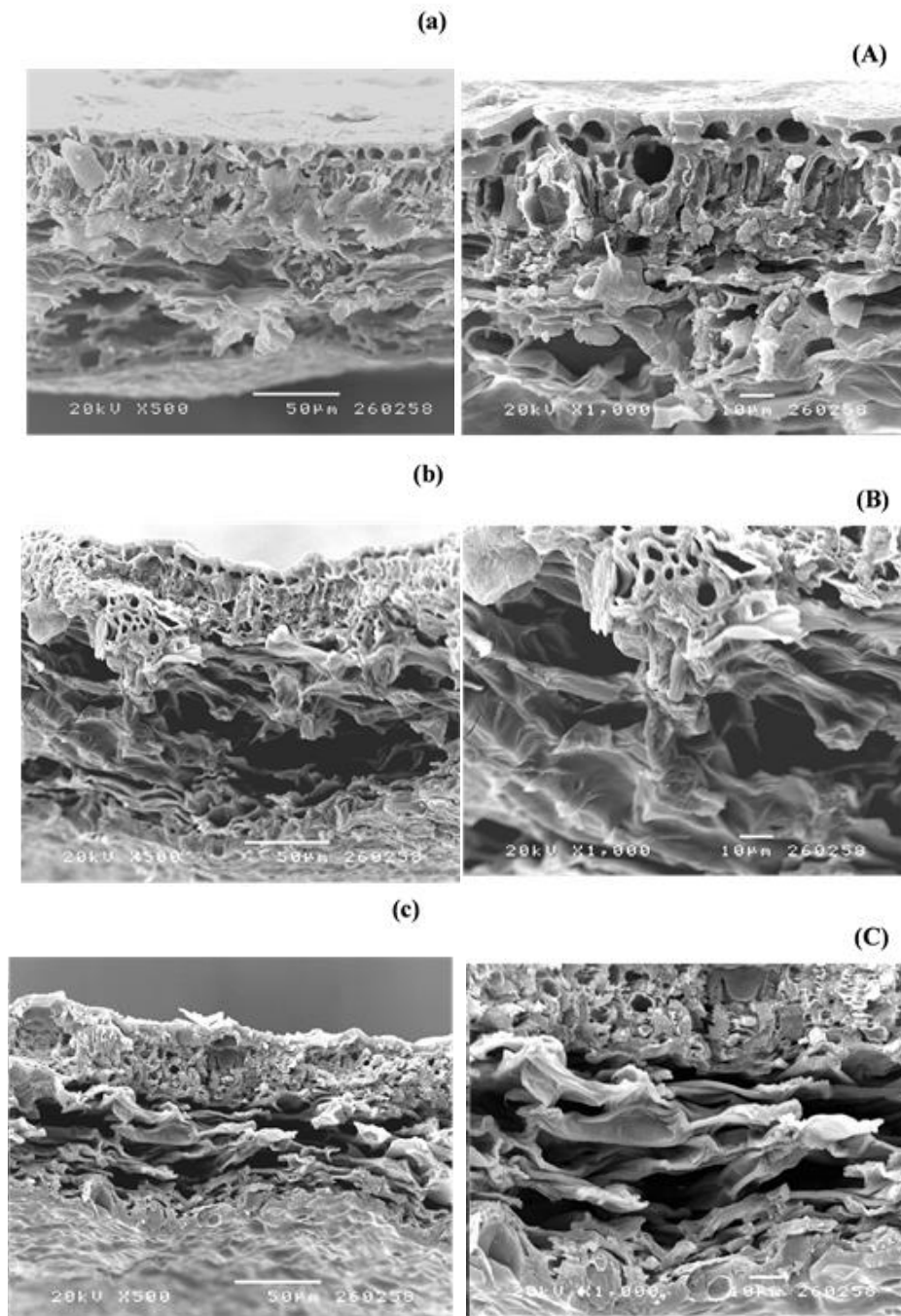


Figure 3. Comparison of the cross-section area of fresh (a), LTLT-treated (b) and HTST-treated (c) KL leaves (x500) and their enlarged pictures (A, B, C)(x1000), respectively, showing how various drying conditions reduced pores and crushed inner layers of KL leaves.

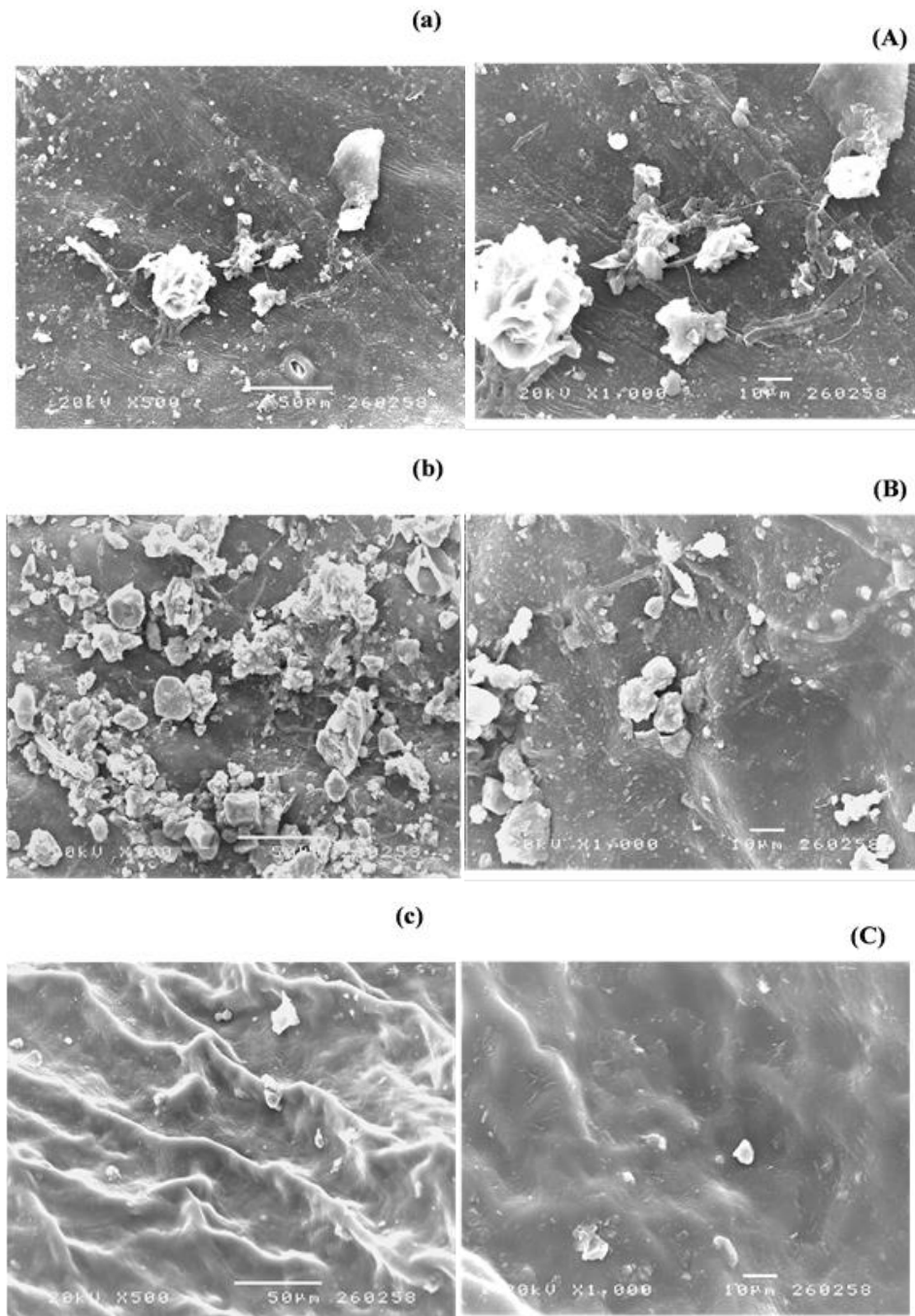


Figure 4. Comparison of the surface area of fresh (a), LTLT-treated (b) and HTST-treated (b) KL leaves (x500) and their enlarged pictures (A, B, C) (x1000), respectively, showing how drying conditions reduced pores and shrunk surface of dried KL leaves.

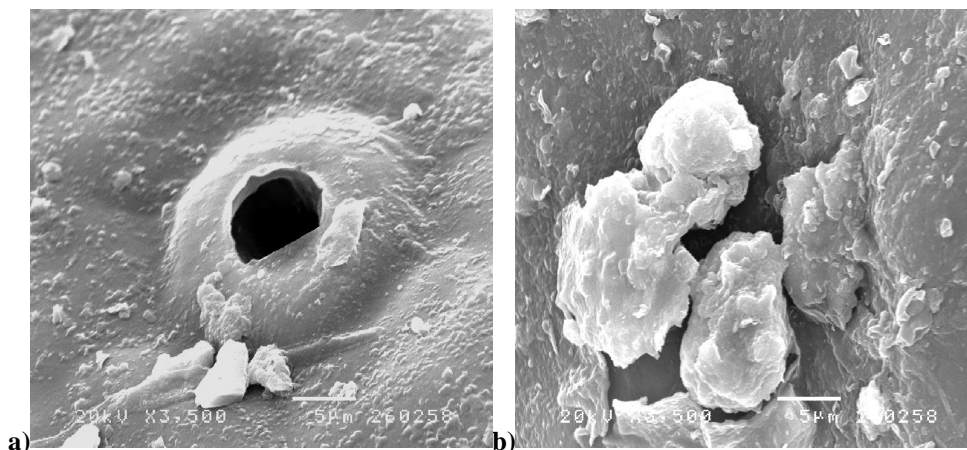


Figure 5. The enlarged illustration of pores on surface of fresh (a) and dried LTLT-treated KL leaves (b) (x3500).

Effect of drying conditions on flavor retention of kaffir lime leaves

In order to determine the flavor retention in fresh and dried kaffir lime leaves from various drying conditions, volatile compounds of kaffir lime leaf were extracted by Simultaneous Distillation Extraction method followed by the separation of the compound using gas chromatography mass spectrophotometer. For compound Identification, volatile compounds were positively identified by comparing their mass spectra and retention index (RI) on HP5-MS columns to those of authentic reference compounds analyzed under identical conditions. Then, the volatile compounds were by calculating the concentration of an aroma component by internal standard methodology by GC-MS using HP5 column. Not all of the compounds identified by GC-MS contributed to the aroma in kaffir lime leaf because of their low relative concentration or their high threshold. Twenty-nine volatile compounds with their odor descriptions that contributed to the major aromas of kaffir lime leaves as well as their threshold were identified and selected based on their concentration (Table 2).

The key volatile compounds such as citronella, sabinene, β -citronellol, geraniol, citronellyl acetate, linalool, Trans- β -caryophyllene or nerolidol were selected based on their concentrations.

The concentration (dry basis) and Odor Activity Values (OAVs) of these selected twenty-nine volatile components of LTLT-treated and HTST-treated KL samples were calculated and compared with those of fresh KL (control) (Table 3). Significant differences were found among the amounts of volatile compounds in both dried KL samples and fresh KL ($p < 0.05$). Citronella was the most abundant compound followed by β -citronellol and l-linalool. The result agreed with others that similar volatile compounds were found with the highest concentration in kaffir lime leaves (Raksakantong *et al.* (2012). Despite the high concentration amount of

these compound, their contributions to the overall odor that consumer could perceive were not as high.

Table 2. The odor description and their threshold values of major aroma compounds of found in kaffir lime leave

RI _{DB} -	Compound name	Perceived odor	Threshold	In
802	Hexanal	Fresh green leaf*	0.0045 ¹	
854	Trans-2-hexenal	Green Grassy*	1.125 ²	
925	α -thujene	Green Spice*	n.a	
932	α -pinene	Pine Woody*	5.0 ³	
941	Butanoic Acid	Sweaty rancid**	6800**	
978	Sabinene	Woody terpinic citrus**	n.a	
982	β -pinene	Pine Lemon Woody*	140 ⁴	
988	B-myrcene	Spice Kaffir Lime leaf citrus*	13.0 ⁵	
1013	α -terpinene	Lemon Kaffir Lime leaf citrus*	n.a	
1026	dl-Limonene	Citrus like**	10.0 ¹	
1037	trans β -ocimene	spice sweet*	40.0 ⁶	
1110	Rose Oxide	Flowery rose like**	0.1**	
1114	l-linalool	Floral Sweet*	6.0 ⁷	
1163	Citronella	Strong citrus, kaffir lime leaf, green*	25.0 ⁸	
1209	Decanal	kaffir lime leaf citrus*	1.97 ⁹	
1238	β -Citronellol	fresh kaffir lime leaf citrus*	40.0 ⁸	
1258	Geraniol	Rose like**	0.04 ¹⁰	
1359	Citronellyl acetate	Lemon Kaffir Lime leaf sweet*	250.0 ⁸	
1368	Neryl acetate	kaffir lime leaf citrus*	10.0 ⁶	
1382	α -copaene	Spice*	0.006 ¹¹	
1411	Dodecanal	Fatty Citrus Like**	0.9**	
1417	Trans- β -caryophyllene	kaffir lime leaf citrus spice*	64.0 ¹	
1429	β -cubebene	kaffir lime leaf citrus*	n.a	
1437	Aromadendrene	Dried Kaffir Lime leaf woody*	n.a	
1456	α -humulene	Dried Kaffir Lime leaf woody*	0.16 ¹²	
1497	Bicyclogermacrene	Very little lemon like woody*	n.a	
1503	γ -cadinene	Dried Kaffir Lime leaf woody*	0.12 ¹³	
1518	Farnesene	Dried Kaffir Lime leaf woody*	n.a	
1564	Nerolidol	Dried Kaffir Lime leaf woody*	0.25 ¹³	

Note: RI=retention index from capillary column DB-5 (thickness 0.25 μ m, length 30.0m and

In order to evaluate the qualities of KL leaves based on the volatile compound, the OAV values were considered as well as the concentration of those compounds. The OAV value was calculated by dividing the concentration with the threshold level of that compound in water. The relative contribution to overall flavor of each volatile

compound was determined using the odor activity values. The higher the OAV value of a compound, the more the contribution in the leaf odor which would be perceived. Therefore, the volatile compound with high concentration amount may not be the major contribution to the overall perceived odor if their thresholds are high. This was the case for citronella, β -citronellol, citronellyl acetate, linalool or Trans- β -caryophyllene, which were the top five volatile compounds most abundant and positive notes in fresh and treated KL leaves. Their high threshold resulted in lower OAV values, indicating that consumers need more concentration from these compounds in order to perceive their odors. On the other hand, some positive notes such as geraniol and rose oxides or relatively negative notes for KL leaves such as α -copaene, Nerolidol or γ -cadinene had higher OAV values due to their low threshold, indicating that consumers did not require high amounts in order to perceive the odors of these compounds. According to the OAV values of fresh and dried kaffir lime leaves for both drying conditions, α -copaene had the highest value followed by hexanal, γ -cadinene, nerolidol and geraniol. However, hexanal was not found in dried KL leaves from either LTLT- or HTST-drying condition.

Most of the compounds concentration decreased during the heat treatment as compared with the fresh sample. Hexanal and trans-2-hexenal were significantly lost in both the LTLT- and HTST-treated samples which indicated that freshness and the grassy odor in dried kaffir lime leaves would be lost. Hexanal was reported to be easily lost from kaffir lime leaves during the heating process (Jirapakkul *et al.*, 2013). However, this was not applicable for other compounds such as γ -cadinene, Bicyclogermacrene, α -humulene, Trans- β -caryophyllene, α -copaene, citronellyl acetate and neryl acetate, which showed an opposite trend, that their concentrations increased when subjected to the heating process.

It has been previously reported in herbs that, due to the heat treatment, certain volatile compounds increased such as increasing of thymol in dried thyme (Venskutonis, 1997), eugenol in dried bay leaves (Díaz-Maroto *et al.*, 2002) and the sesquiterpenes in some herbs because of heating (Baritoux *et al.*, 1992). The increase in concentration of some compounds may cause by the loss of water and the increasing of acidity in the leaves during the drying process that triggers the hydrolysis of glycosides, resulting in the release of aglycones which increasing the concentration of some volatile compounds (De Torres *et al.*, 2010). Among twenty nine compounds, fourteen compounds in HTST-treated KL samples had higher concentrations than those of LTLT-treated KL leaves. This may be due to the oxidation reactions or the releasing of substances cell wall when it ruptures during the heating process (De Torres *et al.*, 2010). Despite the high concentration in HTST-treated samples, either almost all of those compounds had relatively negative notes (less desirable) such as spice-like, dried kaffir lime leaf or woody or they were positive notes with low OAV values. On the other hand, the LTLT condition preserved more odorants with desirable notes including dl-limonene, β -citronellol, geraniol, citronellyl acetate, B-myrcene and decanal in dried LTLT-treated KL leaves with significantly higher amount than that of HTST-treated KL leaves ($p < 0.05$).

Table 3. The concentrations and odor-active values of aroma compounds in fresh and dried KL leaves treated at various drying conditions.

Compound name	Concentration (ppm)			OAV		
	Fresh	LTLT	HTST	Fresh	LTLT	HTST
Hexanal	95.44 ^a	n.d	5.4 ^b	21161.86	n.a	n.a
Trans-2-hexenal	874.33	n.d	n.d	777.04	n.a	n.a
α -thujene	193.66 ^a	41.76 ^c	86.47 ^b	n.a	n.a	n.a
α -pinene	66.55 ^a	35.41 ^b	26.24 ^c	13.20	7.03	5.21
Butanoic Acid	416.78 ^a	9.88 ^c	10.75 ^b	0.06	0.00	0.00
Sabinene	1293.66 ^a	661.56 ^b	534.40 ^c	n.a	n.a	n.a
β -pinene	126.66 ^a	52.49 ^b	46.44 ^c	0.09	0.04	0.03
B-myrcene	856.59 ^a	368.17 ^b	316.76 ^b	65.54	28.17	24.24
α -terpinene	72.46 ^a	13.57 ^b	17.33 ^b	n.a	n.a	n.a
dl-Limonene	154.40 ^a	59.70 ^b	57.63 ^b	15.42	5.96	5.76
trans β -ocimene	153.93 ^a	60.73 ^b	56.73 ^b	3.84	1.51	1.42
Rose Oxide	85.40 ^a	9.80 ^c	13.03 ^b	854.00	98.00	130.30
l-linalool	4243.7 ^a	974.33 ^c	1172.33 ^b	705.99	162.09	195.03
Citronella	96310.47 ^a	37370.65 ^c	42691.93 ^b	3850.57	1494.11	1706.86
Decanal	12.43 ^a	12.80 ^a	11.06 ^b	6.31	6.49	5.61
β -Citronellol	8166.21 ^a	2275.31 ^c	2343.42 ^b	204.09	56.87	58.57
Geraniol	388.50 ^a	29.83 ^b	13.66 ^c	9384.06	720.53	329.95
Citronellyl acetate	2639.66 ^c	4157.42 ^a	3934.36 ^b	10.56	16.63	15.74
Neryl acetate	73.45 ^c	116.33 ^b	145.94 ^a	7.28	11.53	14.46
α -copaene	705.0 ^c	765.59 ^b	1106.35 ^a	114634.15	124486.18	179894.31
Dodecanal	14.09 ^a	7.48 ^b	13.11 ^a	15.66	8.31	14.57
Trans- β -caryophyllene	4145.33 ^b	4004.33 ^c	5438.0 ^a	64.76	62.56	84.96
β -cubebene	47.69 ^c	56.95 ^b	77.00 ^a	n.a	n.a	n.a
Aromadendrene	169.00 ^a	63.4 ^c	119.2 ^b	n.a	n.a	n.a
α -humulene	679.10 ^c	611.00 ^b	852.00 ^a	4202.35	3780.94	5272.28
Bicyclogermacrene	1243.30 ^c	1647.70 ^b	1873.00 ^a	n.a	n.a	n.a
γ -cadinene	1336.20 ^b	1338.0 ^b	1860.8 ^a	10979.46	10994.25	15290.06
Farnesene	779.60 ^b	788.20 ^a	480.40 ^c	n.a	n.a	n.a
Nerolidol	2033.00 ^a	1442.20 ^c	1760.50 ^b	8077.08	5729.84	6994.44

Values with different letters in the same row were significantly different from each other at $p < 0.05$.

“n.d.” referred to “not detected” whereas “n.a” referred to “not available”

Therefore, for flavor retention with OAV taken in consideration, the LTLT condition preserved more key odorants with desirable notes such as geraniol or citronellyl acetate than the HTST condition which gave high OAV in less-desirable notes such as α -copaene, γ -cadinene and nerolidol. This result was supported by

the sensory evaluation that consumer preferred the LTLT-KL leaves over the HTST-KL leaves for overall flavor. Thus, it could be concluded that a low impact drying condition (LTLT) helped preserve high flavor retention in dried kaffir lime leaves in a more effective way than the HTST condition. In addition, the data of these volatile compounds could be used for optimizing the drying conditions (selection of suitable drying temperature during the hot-air drying method) of kaffir lime leaves.

Table 1. Consumer acceptances on dried KL leaves treated at various conditions.

Sample	Sensory		Attribute	
	color	appearance	overall flavor	overall acceptance
LTLT-KL	7.35 ^a ±0.38	7.11 ^a ±0.98	7.52 ^a ±0.59	7.85 ^a ±0.46
HTST-KL	6.45 ^b ±0.50	7.05 ^a ±1.21	6.65 ^b ±0.22	6.79 ^b ±0.53

Values were illustrated as means ± standard deviation. Means with different letters in the same row were significantly different from each other at $p < 0.05$.

Effect of drying conditions on consumer perception of dried kaffir lime leaves

Dried KL leaves from both drying conditions were assessed by forty screened panelists for their acceptances in the organoleptic properties of dried KL leaves using 9-point hedonic scale. The result showed that besides the appearance, there were significant differences between LTLT- and HTST-treated KL samples in all attributes ($p < 0.05$). Panelists preferred dried LTLT-treated KL leaves over HTST-treated KL samples based on the color, overall flavor and overall acceptances. Thus, this consumer perception data helped support the claim that low impact drying in LTLT condition did improve and retain the physico-chemical properties, color retention, microstructure appearance and flavor retention of dried KL leaves whereas those of HTST KL samples were greatly damaged from harsh drying condition.

Conclusions

This study demonstrated that using different drying conditions for kaffir lime leaves would produce individually different results based on the physico-chemical properties, color and appearance and organoleptic properties of dried leaves as well as the odorous volatile compounds. The low impact drying condition (LTLT treatment) using relatively milder condition was more effective drying method in terms of preserving fresh-like qualities of dried KL leaves with less damage, more flavor retention and more desirable products with high overall acceptance. The positive fresh-note compounds dominating the overall odor of KL leaves were also preserved with high flavor retention in LTLT-treated KL leaves. Despite higher process efficiency based on drying rate, the HTST drying condition caused severe damage in internal and external structures of dried KL leaves, resulting in less desirable products. Therefore, the low impact drying condition of LTLT was more

promising than HTST since it could enhance the appearance and flavor retention in dried KL leaves which were the key desirable indicators for superior dried culinary herb products.

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