Effect of different drying methods and conditions on drying performance and volatile retention of *Backhousia citriodora* (lemon myrtle) leaves

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Abstract

The effect of different drying methods *i.e.,* oven (OD), vacuum (VD), and heat pump assisted (HPD) drying on lemon myrtle leaf (LML) as well as the retention of volatiles in the dried product was investigated. Following a constant rate period, each method entered the falling rate period, whereby drying was governed by internal diffusion. The moisture diffusivity (D_{eff}), calculated using Fick's second law, was found to fall in the range of 8.07×10^{-10} to 4.35×10^{-9} m^2/s across the varying drying methods and conditions. The activation energy (E_a), calculated using the Arrhenius equation, was 13.42, 45.41, and 72.85 kJ/mol for HPD, VD, and OD, respectively. Essential oils (EO) were obtained from dried LML through the simultaneous distillation and extraction (SDE) method, and thereafter, the EO was analysed using gas chromatography-mass spectrometry (GC-MS). Citral, an important volatile in LML, was among the EO components identified. Commonly found in its *cis-* and *trans-* forms, citral retention differed between the drying methods. The highest retention of volatiles was observed in the HPD method, followed by VD and OD. As high citral content is indicative of successful post-harvest processing, and given its ability to retain such volatiles in dried LML, HPD is therefore the most suitable drying method for LML drying. HPD can also be applied for the preservation of various agricultural products especially heat-sensitive herbs. The waste heat recovery ability attracts agropreneurs' attention on adapting HPD into their industry. The versatile feature of this controlled dryer not only ensures good quality herb products, but is also economical, efficient, and time-saving.

Introduction

Backhousia citriodora F.Muell. (lemon myrtle) is a flowering plant native to the Australian rainforest (Saifullah *et al.,* 2019). Well known for possessing a distinctive aroma akin to a blend of lemon, lemongrass, and lime, the lemon myrtle leaf (LML) is used to flavour a myriad of foodstuff and beverages (Konczak *et al.,* 2010; Saifullah *et al.,* 2019). In its dried form, LML is also a herb, and could be found in herbal teas (Sultanbawa, 2016). It is the world's richest natural source of citral, which accounts for approximately 98% of its total essential oil (EO) composition (Buchaillot *et al.,* 2009). These traits potentiate the versatility of LML in both the food production and EO industries, thus leading to its increased commercialisation value (Sandrang *et al.,* 2013). As a consequence, more plantations dedicated to LML cultivation have sprouted since it was first brought into Malaysian fields in 2009 (Sandrang *et al.,* 2013). These plantations serve to fulfil the export demand for LML-based goods (Chen, 1997).

The first critical process preceding the production of any such products is LML drying which is an essential step in preserving the leaves (Rayaguru and Routray, 2010). Drying eases the transportation and handling of herbs and spices (Shaw *et al.,* 2005). Moreover, with its low water content, dried LML is less susceptible to microbial contamination, which prolongs its shelf-life. Consequently, this also reduces the risk of economic loss to farmers (Ajayi *et al.,* 2017). The most commonly used method of preservation by drying in food industries is oven drying (OD). It is widely applied in the processing of foods including mint leaf (Therdthai and Zhou, 2009), Mediterranean herbs (Rababah *et al.,* 2015), bay leaf (Diaz-Maroto *et al.,* 2002), and grape leaf (Doymaz, 2011). Meanwhile, more succulent fresh vegetables

including shiitake mushrooms, Jinda chilli (Artnaseaw *et al.,* 2009), and green peas (Nair *et al.,* 2017) are often dried via heat pump assisted drying (HPD). The pump used in this technique extracts and releases heat from circulating air in a closed cycle whilst also obtaining latent energy from the water vapour produced during the drying process (Brushlyanova *et al.,* 2013). Finally, vacuum drying (VD) is the preferred method in processing heat-sensitive materials. Collard leaves (Alibas, 2009) and lemon balm (Argyropoulos and Muller, 2014) are some of the examples of those dried by VD. Regardless, the drying performance of any applied method hinges on its conferred moisture diffusivity and activation energy, as determined by Fick's law and Arrhenius equation, respectively.

The main constituents of EO extracted from LML are *cis*-citral (32.0 - 40.9%) and *trans*-citral (46.1 - 60.7%) (Sultanbawa, 2016). The quality of an LML batch is dependent on its citral content, as this volatile compound is directly associated with its flavour (Buchaillot *et al.,* 2009). Choosing the optimum drying method for LML processing is therefore crucial, as it is extremely impactful on citral retention. Among the main factors influencing the efficacy of an EO extraction is time. When dried on a fluidised bed, a reduction in drying time resulted in lower losses of volatiles in LML (Buchaillot *et al.,* 2009). Sriwichai *et al.* (2019) reported on the significance of the initial drying temperature and type of heat (*e.g.,* wet or dry) on the aromatic characteristics of *makwhaen* (*Zanthoxylum myriacanthum*) fruit. Diaz-Maroto *et al*. (2002) also asserted the effect of different drying treatments on the volatiles in bay leaf (*Laurus nobilis* L.).

In Malaysia, the typical practice in drying LML is through OD. However, OD requires a longer time to be fully effective, thus rendering the dried LML product unsatisfactory in both appearance and volatile compound retention. Despite these clear disadvantages of OD, the feasibility of using an alternative drying method such as HPD for LML drying has yet to be addressed. The present work, therefore, aimed to evaluate and compare OD to HPD and VD based on their drying performance and volatile retention capability during LML drying.

Materials and methods

Sampling

All LML samples used in the present work were cultivated at the Malaysian Agricultural Research and Development Institute (MARDI) Research Station, Serdang, Selangor, Malaysia (latitude: 2.991597, longitude: 101.702195). The leaves were harvested in 3-month cycles to ensure a uniform maturity across all batches. Each sampling was conducted between 8 to 9 am. Freshly harvested batches were processed on the same day for drying experiments. Before drying, the leaves were separated from the stalks.

Sample preparation

Batches of 500 g of LML were separately dried using OD, VD, or HPD. Leaves were arranged on a tray (L: 45.5 cm × W: 26.5 cm × H: 10 cm). The drying experiment was conducted in triplicates. Data collection for each drying experiment was done by weighing samples at specific time intervals using a digital balance (Mettler Toledo, Switzerland). The samples were dried until three constant consecutive weights were obtained. The dried samples were stored in air-tight plastic containers at room temperature in the dark prior to further analyses.

Drying experiments

OD was performed at 40°C (OD40), 50°C (OD50), and 60°C (OD60) in a hot air convective oven (Memmert, Germany). VD was also performed at 40, 50, and 60°C in a vacuum oven (Model V200; Memmert, Germany) with a set pressure of 50 mbar. The heat pump dryer used for HPD was locally fabricated by I-Lab Sdn. Bhd. (Selangor, Malaysia). The machine is operated by passing hot air (45°C) through its drying chamber; a process which allows moisture to be removed from the sample. The moist air passes through the evaporator coils, and transfers heat to the refrigerant in the heat pump system. As a result, the temperature of the drying air is lowered. The air is cooled to its dew point, eventually leading to the condensation of water. The dehumidified cool air is brought to contact with the condenser of the heat pump system, from which it absorbs heat, thus increasing its temperature. This further lowers the relative humidity of the drying air. Thereafter, the dehumidified drying air (with a relative humidity of 20.85 ± 0.93% at 45°C) is charged back into the drying chamber. This closed-loop system is repeated until the sample has reached a stage of equilibrium moisture.

Moisture content

The moisture content of dried samples was determined through the conventional oven-drying method. Briefly, samples were dried in an oven at 105°C until they reached a constant weight (AOAC, 1990).

Moisture diffusivity and activation energy

Effective moisture diffusivity (D_{eff}) is a function of drying temperature and product moisture content. It is used to model the drying process for various agricultural products. To calculate the D_{eff} of a sample, its moisture diffusivity must first be determined. For this purpose, Fick's second law of diffusion was adopted to fit the experimental data, and expressed in Eq. (1) as stated by Doymaz (2011):

$$\frac{\partial M}{\partial t} = \frac{\partial}{\partial x}\left(D_{eff}\frac{\partial M}{\partial x}\right) \tag{Eq. 1}$$

where, M = moisture content (g water/g dry solid), D_{eff} = effective moisture diffusivity (m^2/s), t = time, and x = distance from the centreline of a symmetrical specimen in the direction of moisture flow.

Through a slope method, Fick's diffusion equation was then applied in calculating the D_{eff}, which in the context of the present work, encompassed the flat surface of a leaf. LML samples were thus considered to have a slab geometry, with a thickness of less than or equal to 0.3574 mm. The equation was expressed in Eq. (2) as stated by Doymaz (2011):

$$MR = \frac{M_t}{M_o} = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n-1)^2}exp\left[-\frac{(2n-1)^2\pi^2 D_{eff}t}{4L^2}\right] \tag{Eq. 2}$$

where, L = half of the thickness of the sample (mm), n = positive integer, and t = duration of the drying process (min). For cases with longer drying times, Eq. (2) can be shortened to Eq. (3), which includes only the first term of the series (Aktaş *et al.*, 2009).

$$\ln MR = \ln\left(\frac{8}{\pi^2}\right) - \left(\frac{\pi^2 D_e t}{4L^2}\right) \tag{Eq. 3}$$

The D_{eff} could then be extracted from a graph plotted of ln (MR), derived from data obtained throughout the drying experiments against time. From Eq. (3), the ln (MR)-time diagram would present an inclined line with its slope (K) interpreted according to Doymaz (2011), a formula represented as Eq. (4):

$$K = \frac{\pi^2 D_{eff}}{4L^2} \tag{Eq. 4}$$

The D_{eff} could therefore be determined using this equation as well.

Another important factor dictating drying efficacy is the bonding potential of moisture in non-dry materials. At any given moisture content, the energy required to eliminate 1 mol of moisture from the substance is quantified as the activation energy. By using a simple Arrhenius equation (Simal *et al.*, 1996), the activation energy of the hot air technique could be calculated using Eq. (5):

$$D_{eff} = D_o exp\left(-\frac{E_a}{R(T+373.15)}\right) \tag{Eq. 5}$$

where, T = drying air temperature (°C), R = universal gas constant (8.314 × 10^{-3} kJ mol^{-1} K^{-1}), D_o = pre-exponential factor of the Arrhenius equation (m^2/s), and E_a = activation energy (kJ/mol). Eq. (5) can also be rearranged into Eq. (6):

$$\ln D_{eff} = lnD_o - \frac{E_a}{R}\left(\frac{1}{T+273.15}\right) \tag{Eq. 6}$$

From the slope of a straight line when ln (D_e) is plotted against the multiplicative inverse of absolute temperature ($1/(T + 273.15)$), E_a could be derived.

Simultaneous distillation-extraction (SDE)

A simultaneous distillation-extraction (SDE) procedure was performed according to Bidgoli *et al.* (2014). Briefly, 1.5 L round bottom flask was filled with 1 L of distilled water (Milli-Q water purification system; Pall C, Illinois, USA), and into which 100 g of dried LML was added. A multi-exit cap was applied onto the sample flask. One of the exit tubes was connected to the left arm of the SDE apparatus (Likens-Nickerson), which had its right arm attached to 100 mL round bottom flask containing 40 mL of pentane (Sigma-Aldrich, Missouri, United States). A heating mantle was used to heat the sample flask to about 100°C, which is the boiling point of water, and another heating mantle was used to heat the solvent flask to 50°C, which is higher than the boiling point of pentane (36.1°C). Another exit tube of the sample flask was connected to a condenser-equipped distillate receiver. The cooling finger of the distillate receiver was attached to a small pump immersed in ice water. The temperature of the circulating cooling water was set to 10°C.

This systematic flow, termed SDE, was carried out over 4 h at atmospheric pressure. During the process, EO evaporated along with the steam, and was extracted from the condensates collected in the distillate receiver. A few grams of anhydrous sodium sulphate (Sigma-Aldrich, Missouri, United States) were added to the extracted distillate and allowed to stand for 1 h for dehydration. The solution was filtered with filter paper, and concentrated using ultra-high purity nitrogen (99.999%). Extracted EOs were kept in amber glass vials for volatile compound analysis.

Gas chromatography-mass spectrometer (GC-MS)

EOs from LML were analysed by gas chromatography equipped with mass spectrometry (GC-MS-QP2010 Plus-Shimadzu, Japan). Zebron ZB5-ms (Phenomenex, USA) (30 m × 0.25 mm ID × 0.25 µm film thin) columns were used during analysis. Briefly, the temperature of the column was set to 50°C for 1 min, then increased to 130°C at a rate of 7°C/min for 12.6 min. The injector temperature was set to 250°C (split mode, 5:1, injection volume = 0.2 L).

Statistical analysis

All experiments were performed in triplicates in order to verify the repeatability of the results. The data were analysed using One-way analysis of variance and Tukey's honest significance difference (HSD) test at $p < 0.05\%$ using SAS software (Version 9.4, S.A.S. Institute Inc. North Carolina, USA). All data were presented as mean ± standard deviation.

Results and discussion

Drying duration and final moisture content

Figure 1 shows the effect of the drying methods on the drying times and moisture contents of dried LML. Collectively, whereas total drying time fell between 12 and 76 h, the applied methods reduced LML moisture content from $63.27 \pm 4.66\%$ to a range of 5.38 to $10.36 \pm 0.94\%$. OD40 took the longest drying time of 76 h. By comparison, OD60 dramatically reduced this to 12 h. Across the varied treatment parameters, OD60 samples yielded the lowest final moisture content, whilst OD40 and HPD yielded the highest. For temperature-dependent methods, namely OD and VD, manipulating the drying temperatures affected both time of drying and final moisture content significantly. The higher the drying temperature, the shorter the total drying time, and the lower the final moisture content of the dried LML. This could be due to the vaporisation of surface moisture from the material which became more efficient upon contact with more heat (Doymaz, 2011). Alibas (2007) has reported a similar observation with nettle leaves dried using VD, where total drying time decreased with increasing VD temperature. Conversely, although HPD operated at a lower temperature of 40°C, the reduction of the moisture content was comparable to that of OD50. This might be due to low relative humidity (RH) of drying air in HPD though the drying air temperature was lower (Rayaguru and Routray, 2010).
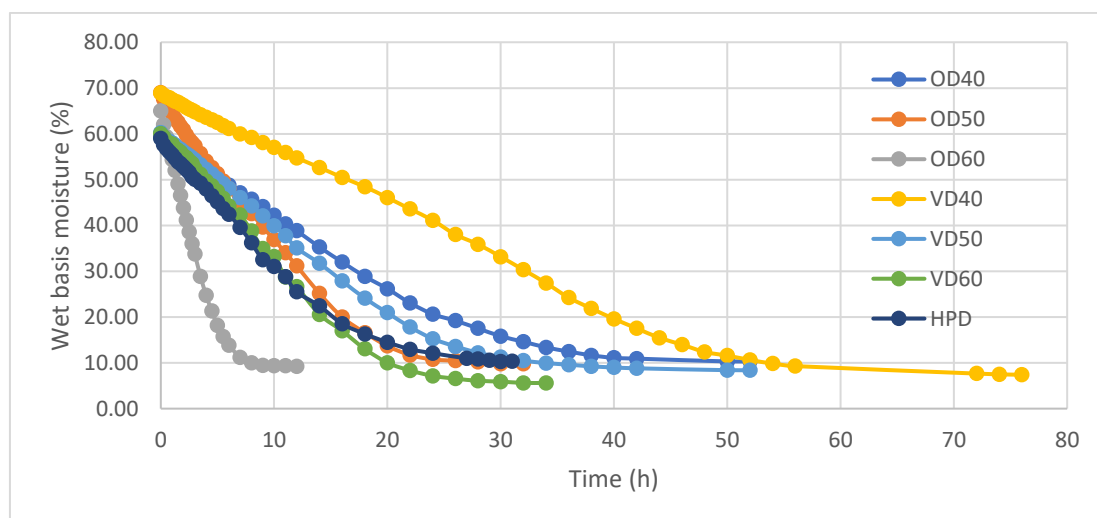

Figure 1. Impact of drying methods on drying time (h) and moisture content (%) of dried lemon myrtle leaves (LML).

Moisture diffusivity and activation energy

Moisture diffusivity is the most important transport property for calculating moisture transfer within agricultural food products during drying (Akpinar and Toraman, 2013). It is important not only to explain drying kinetics and for the interpretation of experimental results, but also to effectively simulate the drying process. Moisture diffusivity (D_{eff}) was calculated and applied to derive activation energy (E_a) in accordance to Fick's second law and Arrhenius equation, respectively. Figure 2 shows the ln (*MR*) with drying times at different air temperatures for OD, VD, and HPD.
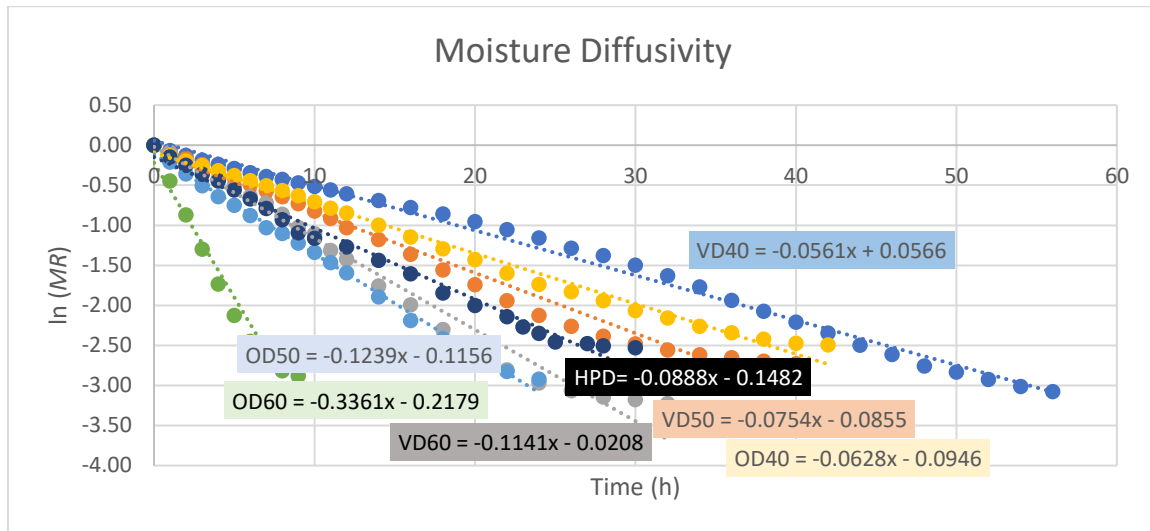

Figure 2. Variation of ln (*MR*) with drying times at different air temperatures (*T*) and methods. Incline lines represent slope (*K*) for moisture diffusivity determination (Eq. 4) for each drying method and condition.

As shown in Table 1, the coefficient of R^2 for the different drying methods and conditions were in the range of 0.9645 to 0.9955. The D_{eff} of LML was calculated and found to be between 8.07×10^{-10} and 4.35×10^{-9} m^2/s for OD, whilst ranging between 4.167×10^{-10} and 1.188×10^{-9} m^2/s for VD. The D_{eff} of HPD at its set temperature was 1.31×10^{-9} m^2/s. The D_{eff} of each drying method was then shown to increase with increasing air temperatures for OD and VD, coinciding with previously published results by Akpinar and Toraman (2013) and Komolate *et al.* (2018), which respectively demonstrated that temperature had a major effect on D_{eff} during the process of drying ginger slices and fish. The D_{eff} values obtained in the present work are also in agreement with the range of 10^{-12} to 10^{-8} m^2/s, reported as D_{eff} of drying of food materials by Zogzas *et al.* (1996).

Table 1. Effective moisture diffusivity (D_{eff}) and coefficient of determination (R^2) for different drying methods and conditions.

Drying method	Drying temperature (°C)	D_{eff} (m^2/s)	R^2
OD	40	8.07×10^{-10}	0.9907
	50	1.54×10^{-9}	0.9955
	60	4.35×10^{-9}	0.9645
VD	40	4.17×10^{-10}	0.9938
	50	6.98×10^{-10}	0.9787
	60	1.19×10^{-9}	0.9781
HPD	45	1.093×10^{-9}	0.9859

The minimum energy needed to initiate moisture diffusion from a substance is known as its activation energy or E_a (Shahi *et al.*, 2012). Here, it was calculated by plotting ln D_{eff} versus the temperature of $1/T(K)$ (Figure 3a). The results are presented in Figure 3b. A linear correlation as represented by the straight bisecting line on the graph of ln D_{eff} against $1/T(K)$ indicated an Arrhenius dependence. The E_a value determined from this sloping line was found to be 72.85 and 45.41 kJ/mol for OD and VD, respectively. Meanwhile, HPD required only 13.42 kJ mol of energy to initiate moisture diffusion in LML. These E_a values therefore

complied with a previously reported range for a variation of food products by Xiao *et al.* (2012), which fell between 12.7 and 110 kJ/mol. Additionally, D_{eff} and E_a values obtained in the present work were all in reasonable agreement with the consensus D_{eff} of 6.76143×10^{12} to 1.57018×10^{-10} m²/s and consensus E_a of 42.07 to 44.74 kJ/mol for savoury leaves; 4.54×10^{-10} to 1.08×10^{-9} m²/s and 20.32 to 38.54 kJ/mol for basil leaves; 4.13×10^{-10} to 1.83×10^{-9} m²/s and 64.56 kJ/mol for grape leaves; and 3.067×10^{-9} to 1.941×10^{-8} m²/s and 62.96 kJ/mol for mint leaves (Doymaz, 2006; 2011; Shahi *et al.*, 2012; Taheri-Garavand and Meda, 2018).
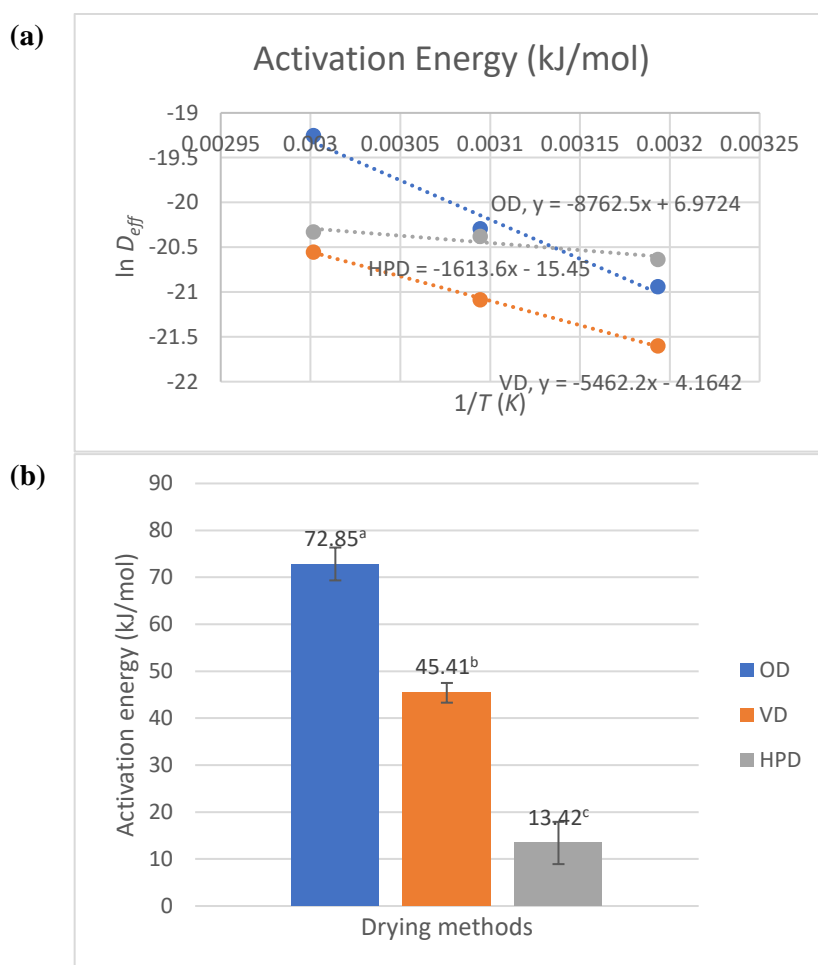

Figure 3. (a) Arrhenius-type relationship between effective moisture diffusivity and 1/absolute temperature, $T(K)$ (blue dots: oven-dried sample; red dots: vacuum-dried sample; green dots: heat pump-dried sample); and **(b)** activation energy (E_a) calculated based on moisture diffusivity. Vertical bars indicate standard deviation, and values marked by the different letter are significantly different ($p < 0.05$).

Retention of volatiles

LML EO predominantly contained citral (3,7-dimethyl-2,7-octadienal), a volatile compound bearing two main isomeric aldehydes *i.e.,* neral and geranial with retention index (RI) in the range of 1211 - 1240 and 1236 - 1260, respectively (Babushok *et al.*,

2011). LML is said to have the highest citral content (90 - 98%) of all citrus or citrus-like genus of plants. By contrast, lemongrass has an estimated citral content of 75%, whereas lemon has merely 4% (Sandrang *et al.*, 2013). LML EO isolated from OD50, VD50, and HPD samples through the SDE

method manifested as a pale-yellow oil with a strong lemony odour. Extracts for each drying method was analysed for its volatile compound retention by using GC-MS. Chromatograms are shown in Figure 4. Two distinguished peaks were detected from each extract regardless of the drying method. However, peak characteristics were dissimilar, exhibiting different drying strategies which will selectively retain specific compounds more efficiently over others.
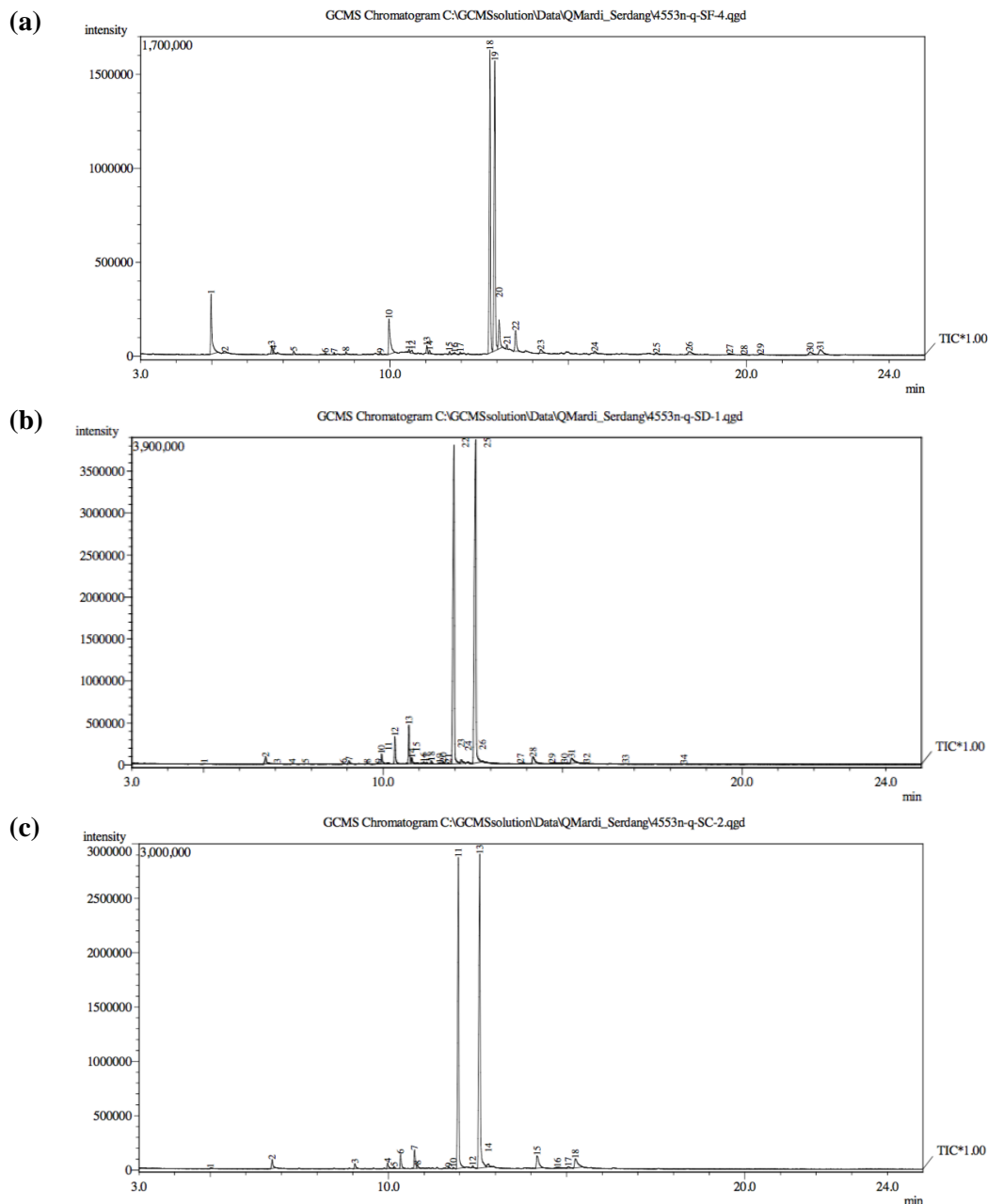

Figure 4. Chromatograms of **(a)** OD50, **(b)** VD50, and **(c)** HPD.

Further details on the identified volatile compounds are presented in Table 2. VD50 and HPD samples showed peaks at the retention time (RT) of 12 and 12.5 min, respectively, each representative of *cis-* and *trans*-citral. The VD50 EO was composed of 39.63% *cis*-citral and 39.63% *trans*-citral. In comparison, HPD immensely improved citral retention, as its EO comprised 45.67 and 45.08% of *cis-* and *trans*-citral, respectively. The two dominant peaks produced from the EO of OD50 deviated completely from those of VD50 and HPD peaks. They were detected at the RTs of 12.804 and 12.939, and were identified to be epoxy-linalool oxide (RI = 1410 - 1478; Babushok *et al.,* 2011). Low levels of this compound were also detected in the HPD EO (0.67% total), but was not at all found in VD50 EO.

Based on Table 2, the identified volatile compounds varied between drying methods. For

instance, linalool and 3,7-dimethyl-6-octenal (citronellal) were identified only in VD50 and HPD samples. Linalool (RI = 1074 - 1098; Babushok *et al.,* 2011), a terpenoid with a floral and citrus-like odour, improves food quality as its addition enhances flavour as well as fragrance (Yang *et al.,* 2013). Citronellal (RI = 1216 - 1143; Babushok *et al.,* 2011) detected at an RT of 10.150 min from VD50, is a compound described as having a sweet, floral, and rosy smell, distinguishable from others by its waxy quality, and citrus green colour in LML extract (Sultanbawa, 2016). Apart from these desirable aromatic properties, linalool and citronellal, alongside the more ubiquitous compounds such as epoxy-linalool, neric acid, and citral, are all believed to be responsible for the insecticidal activity of LML (Garba, 2016). Therefore, the successful recovery of such volatiles is important as it is an indication that the employed drying methods did not negatively affect the integrity of EO contained in LML.

From Table 2, the maximum retention of volatile compounds was achieved through HPD, followed by VD50, and finally OD50. This may largely be due to drying time. LML required increasingly more time to enter its falling rate period as the applied drying method changed from HPD to VD50 and OD50. Such an observation is in line with findings presented by Phoungchandang *et al.* (2008), whereby the HPD method resulted in both shorter drying times and higher levels of remaining citronellal. Buchaillot *et al.* (2009) also reported that by reducing drying time, the amount of volatiles lost would also be decreased. It is postulated that the technique involved in HPD is most efficient at inducing the formation of a crust layer at the surface of the partially dried LML, which acts as a protective barrier against the mass transfer of water, thus limiting the diffusion and loss of high-molecular weight volatiles.

Table 2. Identified volatile compounds for OD50, VD50, and HPD.

Identified volatile compound	OD50		VD50		HPD	
	Retention time (min)	(%area)	Retention time (min)	(%area)	Retention time (min)	(%area)
Linalool (3,7-dimethyl-1,6-octadien-3-ol)	nd	nd	9.052	0.36	9.06	0.72
Citronellal (3,7-dimethyl-6-octenal)	nd	nd	10.15	0.17	10.16	0.25
Citranellol	nd	nd	11.68	0.25	11.69	0.13
cis-citral (3,7-dimethyl-2,6-octadienal)	11.966	0.18	11.975	39.63	11.963	38.88
trans-citral (3,7-dimethyl-2,6-octadienal)	nd	nd	12.578	45.67	12.562	45.08
3,7-dimethyl-2,6-octadien-1-ol	nd	nd	12.175	0.61	nd	nd
Epoxy-linalool oxide	12.804	33.59	nd	nd	12.808	0.67
	12.939	34.4				
	15.743	0.36				
	18.397	0.97				
3,7-dimethyl-2,6-octadienoic acid	nd	nd	13.83	0.16	14.745	0.75
			14.71	0.27		
2,7-dimethyl-2,7-octanediol	nd	nd	14.18	1.41	14.17	3.02
Citronellal hydrate (3,7-dimethyl-7-hydroxy-octanal)	14.23	0.78	nd	nd	nd	nd
	19.929	0.3				
TOTAL		70.58		88.53		89.50

Conclusion

Three methods were chosen in this comparative analysis between different drying techniques on LML, namely OD, VD, and HPD. Drying temperatures were also varied at 40, 50, and 60°C for both OD and VD. All drying methods vastly expelled water from LML, where it was reduced from a range of 58 - 70% of total leaf mass, to just 8 - 10%.

These significant reductions demonstrated that each method was indeed useful as an industrial application in prolonging the shelf-life of fresh produce. However, considerable variations in the final moisture content of the dried LML product were observed across drying methods. Moreover, analysis of LML EO showed obvious disparities in their volatile compound retention capability. EO extracted from samples dried via HPD retained the highest

percentages of LML EO volatiles, followed closely by samples from VD, and finally from OD. Additionally, not only would HPD require the shortest drying time, it is also a method that incurs low capital and operating costs, particularly as compared to VD. To conclude, HPD is the recommended drying method of practice for LML, and would likely be just as robust for other high EO-containing materials.

Acknowledgement

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