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Effect of Drying on the Kinetics, Stability of Active Ingredients and Non-Destructive Testing of *Backhousia citriodora* (Lemon Myrtle) Dehydrated Leaves

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ABSTRACT

The dehydration process of lemon myrtle leaves (LML) in Malaysia is still conducted by using conventional method of drying that takes long duration thus decreasing the quality of the dried LML. To prolong the shelf life, LML has to be dried to reduce its moisture content. The present research highlighted the drying kinetics, application of heat pump drying on LML and the application of non-destructive testing on moisture content analysis of dried LML. In the present study, LML were dried by OD (40 to 60°C), VD (40 to 60°C at 50 mbar) and HPD (45°C). Both engineering properties (drying kinetics, effective moisture diffusivity and activation energy) and quality properties (colour, biochemical content and volatile content) of all dried LML were assessed and evaluated.

All drying methods only exhibited falling rate period, indicating that the drying was governed by the movement of internal moisture to the surface for evaporation. The moisture diffusivity (D_{eff}) was found to vary in range of 8.07 x 10^{-10} to 4.53 x 10^{-9} m^2/s for all drying methods and conditions. The activation energy (E_a) was obtained and the values were 13.42 kJ/mol, 45.41 kJ/mol and 72.85 kJ/mol for HPD, VD and OD, respectively. It was found that the drying air velocity of 2.0 m/s (FR2.0) in the HPD was the suitable drying condition as it gave minimum colour changes and the highest

retention of volatiles which was recorded at 89.5%. The total phenolic content (TPC) and antioxidant activities of samples drying at 2.0 m/s also showed high retention, recorded at 74%, 95% and 80% for TPC, 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging activity and ferric reducing antioxidant potential (FRAP), respectively. It was found that HPD sample gave the highest value of greenness (a^*) with the lowest value of total colour difference and browning index. For biochemical content, VD samples showed high retention of TPC, DPPH and FRAP assay followed by HPD samples, while OD samples showed the lowest biochemical content for all drying conditions. The essential oil of the dehydrated LML subjected to OD50, VD50 and HPD was extracted by using simultaneous distillation and extraction (SDE) method and analysed for its volatile compounds by using Gas Chromatography-Mass Spectrometer (GC-MS). HPD samples showed the highest retention of volatiles compounds especially cis- and trans-citral with a total concentration of 89.5%.

Dehydrated LML were packed under four different conditions, normal and vacuum packaging (N and V), each of the packaging was placed at room temperature (25°C) or chilled condition (4°C) (RT and CH) stored for a period of 6 months. It was found that the vacuum packaging yielded higher retention of colour and biochemical content than the non-vacuum packaged LML. This approach combined with

storage at lower temperature (4°C) resulted in a better retention of green colour and higher percentages of TPC, DPPH and FRAP (49%, 72% and 56% respectively) for HPD samples.

Partial least squares regression (PLSR) and cross-validation modelling were used to explore the feasibility of these spectroscopic techniques over three types of prediction models which were full spectral ranges of near infrared (NIR) and dielectric measurement (DM), selected spectral range of NIR and fusion of both non-destructive methods. NIR model gave the highest coefficient of determination, *r* value of more than 0.99 and ratio performance to deviation (RPD) value of 3.16 that indicated excellent prediction of moisture content in the dried LML. The selected spectra analysis of NIR spectra showed no improvement in PLSR results. Whereas, fusion of the non-destructive models showed improvement of the r, root mean square error and RPD values especially for DM method.

The effect of drying on the stability of active ingredients of *Backhousia citridora* (lemon myrtle) dehydrated leaves was investigated and discussed in this study. HPD was found to be a suitable method for LML dehydration, as it resulted in the highest retention of volatiles compound, greenness and antioxidant values after drying and after 6 months storage. The high quality of dehydrated LML subjected to HPD ensures the market acceptability

as well as its functionality. Therefore, this study suggested that HPD with drying air velocity of 2.0 m/s could be used as a proper dehydration process for LML that preserve its functionality for the applications of nutraceuticals, cosmeceuticals and pharmaceuticals.

PUBLICATIONS

Part of the studies in this dissertation have been submitted for publications:

International Journals

i) **Abdul Kahar, A.A.,** Ong, S.P., Watson, N.J. and Law, C.L. Effect of Different Drying Methods and Conditions on Drying Performance and Volatile Retention of *Backhousia citriodora* (Lemon Myrtle) Leaves. *International Food Research Journal*. [Under review, submitted on 1 December 2019].

ii) **Ainaa Abdul Kahar;** Ong Sze Peng; Nicholas J. Watson; Law Chung Lim, Effect of Packaging Conditions and Temperature during Storage on Dried Lemon Myrtle Leaves Quality. *International Journal of Postharvest Technology and Innovation.* [Under review, submitted on 28 April 2020].

Conference Papers

i) **Kahar, A.A.,** Ong, S.P. and Law, C.L., 2018. Effects of Drying on the Colour and Bioactive Compound Retention of *Backhousia citriodora* (Lemon Myrtle) Leaves. In The 4th International Conference of Chemical Engineering and Industrial Biotechnology (ICCEIB 2018), Kuala Lumpur, Malaysia, August 1-2, 2018, pp. 137-139.

ii) **Abdul Kahar, A.A.,** Ong, S.P., Watson, N.J. and Law, C.L., 2019. Effect of Different Drying Methods and Conditions on Drying Performance and Volatile Retention of *Backhousia citriodora* (Lemon Myrtle) Leaves. In The 2nd International Food Research Conference (IFRC 2019), Putrajaya, Malaysia, August 28-29, 2019, pp. 91.

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LIST OF NOTATIONS AND ABBREVIATIONS

Notations

a^*	CIE redness value
b^*	CIE yellowness value
cv	Cross validation
db	Dry basis based moisture content
D_{eff}	Effective moisture diffusivity (m^2/s)
E_a	Activation energy (kJ/mol)
L	half of the thickness of the sample, mm
L^*	CIE lightness value
M	Moisture content (g water/g dry solid)
M_{db}	Moisture content (% dry basis)
M_{wb}	Moisture content (% wet basis)
N	Positive integer
R	Universal gas constant (J/mol.K)
r	Coefficient of correlation (validation model)
$r_$test	Coefficient of correlation (test model)
R^2 or r^2	Coefficient of multiple correlation
T	Absolute temperature (K)
t	Duration of the drying process (min)
W	Watt
% wb	Wet basis-based moisture content (%)
w_b	Mass of bone dry solid (kg)
w_c	Critical moisture content
w_i	Initial sample's weight (kg)
w_t	Sample's weight at t time (kg)
x	Distance from the centerline of a symmetrical specimen in the direction of moisture flow

Abbreviations

AAE	Ascorbic acid equivalent
BI	Browning index
DM	Dielectric measurement at 920 to 950 MHz
DPPH	2,2-diphenyl-1-pycrylhydrazyl free radical scavenging
DR	Drying rate
FRAP	Ferric reducing antioxidant power assay
GAE	Gallic acid equivalent
HPD	Heat pump drying
LML	Lemon myrtle leaves
MR	Moisture ratio
NCH	Normal packaging stored in chiller
NIR	Near-infrared
NIR+DM	Combination of near-infrared and dielectric measurement
NIR1600-1700	Near-infrared spectra at wavelength of 1600 to 1700 nm
NIR1600-1700+DM	Combination of near-infrared at 1600 to 1700 nm and dielectric measurement
NIR1650-1800	Near-infrared spectra at wavelength of 1650 to 1800 nm
NIR1650-1800+DM	Combination of near-infrared at 1650 to 1800 nm and dielectric measurement
NRT	Normal packaging stored at room temperature
OD	Oven drying
OD40	Oven drying at 40°C

OD50	Oven drying at 50°C
OD60	Oven drying at 60°C
RMSE	Root mean square error
RMSEP	Root mean square error for prediction
RPD	Ratio performance to deviation
TCD	Total colour difference
TPC	Total phenolic content
VCH	Vacuum packed and stored in chiller
VD	Vacuum drying
VD40	Vacuum drying at 40°C and 50 mbar
VD50	Vacuum drying at 50°C and 50 mbar
VD60	Vacuum drying at 60°C and 50 mbar
VRT	Vacuum packed and stored at room temperature

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CHAPTER 1 INTRODUCTION

1.1 Background of study

Backhousia citriodora or commonly known as lemon myrtle leaves is an Australian native herb. Lemon myrtle is included in the family of Myrtaceae and originated from Queensland, Australia. First introduced in Malaysia since 2009, there are several successful nurseries and plantation of 32,000 cuttings of lemon myrtle in the area of Sekinchan, Kuala Selangor and southern area of Kuala Linggi and Kluang, Johor. Lemon myrtle received good attention from the industry, because of its remarkable versatility in nutraceutical and pharmaceutical application.

Lemon myrtle leaves is well known for its application in culinary as dried herbs. As known to own a distinguished odour of lemony, the taste of lemon myrtle is described as similar to blend of lemon, lime and lemongrass. Lemon myrtle also has its own demand as herbal tea with refreshing flavor that is potentially an alternative to regular tea. The key odoriferous principle of lemon scented oils is citral that is distributed in two different forms (isomers) which are neral and geranial, thus it is potentially exploited in perfumery (Svoboda and Greenway, 2003).

Lemon myrtle products such as tea is not just refreshing but it is also owning excellent functionality. As for the essential oils, it is famous for its antimicrobial, antifungal and antibacterial properties against pathogens. Lemon myrtle leaves are usually extracted for its essential oil. Its essential oil own antimicrobial properties inhibiting *Escherichia coli* and *Saphyloccocus aureus* at 0.313% and 0.156%, respectively (Sultanbawa, 2016). Citral is the major component of the essential oil which mainly consists of neral and geranial while minor components are ocimere, p-cymene, citronellal, linalool, ß-elemene, ß-caryophyllene, nerol, geraniol, globulol and spathulenol (Svoboda and Greenway, 2003). Therefore, lemon myrtle products with citral as main active component have garnered attention because of its distinguished aroma and possible uses in medicinal, poultry and culinary.

To date, there are only five countries that produce lemon myrtle but only Australia involves in the commercial production. Statistics show that about 1.4 million lemon myrtle trees are commercially planted with production of 2,100 ton of wet leaves and 50 ton of essential oil produced per year. The selling price for each kilogram of wet and dry leaves is approximately in range of AUS$17 to AUS$30 and AUS$35 to AUS$50, respectively. The price of LML essential oil is increased from AUS$100/kg a few years agor to more than AUS$350/kg with the price expected to rise as demand increases

(Chemical identification of lemon myrtle to future proof essential oil, 2021). The estimated price for each plantation is said to be around AUS$6.96 and AUS$22.9 million including dry leaves and essential oil (Sandrang et al., 2014). Table 1.1 shows that comparison on income and production cost for lemon myrtle in comparison to palm oil and rubber. Lemon myrtle has shown its potentialities to be the main crop as it has the highest gross and net income despite the high planting cost that only involved in Year 1.

Table 1.1 Comparison of income and production cost (RM) for lemon myrtle and other crops (Sandrang et al., 2014)

	Crop	Year 1	Year 2	Year 3	Year 4	Year 5
Planting cost until first production	Lemon myrtle	61,318				
	Palm oil			6,070		
	Rubber					7,000
Gross income	Lemon myrtle	27,000	45,000	135,000	270,000	270,000
	Palm oil			2,500	4,000	4,800
	Rubber					4,340
Production cost + processing cost (% gross income)	Lemon myrtle (30%)	8,100	13,500	40,500	81,000	81,000
	Palm oil (40%)			1,000	1,600	1,920
	Rubber (45%)					1,953
Net profit (2 – 3)	Lemon myrtle	18,900	31,500	94,500	189,000	189,000
	Palm oil			1,500	2,400	2,880
	Rubber					2,387

Second category of lemon myrtle processing is postharvest. Lemon myrtle leaves are dried and processed for food, nutraceutical and pharmaceutical applications. Drying of lemon myrtle leaves is important to reduce moisture content of the leaves to facilitate the transportation and storage of the dried product. In Malaysia, lemon myrtle leaves are dried by using withering process as adopted from Australian commercially used for drying lemon myrtle leaves (Figure 1.1). As the same as drying tea leaves, the chamber is made up from wood frame, plywood as chamber's wall and net as the floor. This system is a combination of heat source from ambient air and heated air produced from an installed fan. Hot air is blown into the drying chamber consisting of bed of lemon myrtle leaves. The moisture content of lemon myrtle leaves is dried until it reaches less than 10% wb.

Figure 1.1 Withering process of lemon myrtle leaves in Sekinchan, Selangor adopted from commercial Australian lemon myrtle industry

It is important to find a suitable drying method to preserve it's the physical and biochemical properties of myrtle leaves. An efficient and suitable drying method needs to be determined to ensure the high quality of dried lemon myrtle leaves (LML). The good quality of dehydrated LML is said to have less colour changes (greenish leaves) which is a good indication to the preservation of active ingredients and functionality of the LML. According to Sandrang et al. (2014) a good LML drying process is shown by greenish colour of LML even dried, could produce dried LML leaves that contains high citral content, free from any heavy metal traces and pathogens such as *Escherichia coli* and *Salmonella*. Thus, proper selection of drying

method and condition is key in ensuring a successful drying operation and high retention of the dried LML quality.

Types of drying method and condition are important to counter special characteristics on the dried LML leaves, as its active ingredients are heat sensitive. This study tested heat pump assisted dryer, vacuum and oven drying to dry lemon myrtle leaves. Heat pump dryer (HPD), a closed system that recycles back the generated heated air. It has the advantage of less energy consumption, hygienic system and operating at low temperature (Fayose and Huan, 2016). Nevertheless, vacuum pump is also considered as an option to dehydrate the leaves as low temperature drying condition is able to preserve the heat sensitive active ingredients better (Parikh, 2015). The drying temperature cannot be not higher than 60°C, as lemon myrtle major component, citral, is easily vapourised.

Colour is also an important physical quality that is needed to be preserved. It is correlated to the biochemical compounds' retention of processed products. It is also important to the market acceptability which in turn impacts its commercial value. Biochemical content is expressed by its antioxidant activity and total phenolic content. Drying lemon myrtle leaves at temperature that is higher than 45°C may loss its volatile compound, citral. In contrary, drying at lower temperature may prolong the time, thus lower retention on

the biochemical content after exposed the leaves to heat for some time. Therefore, there is a need to identify a drying method for LML that can dry the LML at a suitable temperature within an acceptable duration to preserve the active ingredients.

To date, the report on post harvesting process on drying especially the application of heat pump drying on lemon myrtle leaves is rather scarce. Therefore, this was the main motivation why this study was conducted. This study focused on the application of oven-, vacuum- and heat pump drying (HPD) on drying lemon myrtle leaves as well as the storage stability study and feasibility of non-destructive techniques on dried product analysis. The purpose of conducting a non-destructive technique was to extend the capabilities of dryer in online monitoring for the quality of LML especially the moisture content. It serves as an important information during dehydration process to ensure high quality of the dried leaves.

1.2 Research problems

Lemon myrtle leaves (LML) were harvested and dried before exported to other countries. Conventionally, LML were dried by using withering trough process where hot air (temperature less than 40°C) was supplied into the drying chamber. The leaves were collected and removed from the stalks before drying (Figure 1.2(a) and (b)). Upon the completion of drying process, the leaves were milled before they

were packed as LML products i.e tea blend (Figure 1.2(c)). The duration of drying to reduce moisture content to approximately 10% was around 96 h. The long period of drying decreases the quality of the dried leaves including its colour properties and hence increases the deterioration of biochemical content and loss of the volatiles compound. Therefore, it is important to determine the efficient drying method and conditions of LML to maintain its quality as well as to obtain maximum retention of biochemical and volatile compounds of the dried leaves. In this study, the potentialities of different drying methods and conditions were explored in order to determine the best dehydration method for LML. In addition, storage stability of each drying product was investigated in order to determine the most stable drying method for a duration of time. This study also studied the feasibility of non-destructive techniques namely near infrared spectroscopy and dielectric spectroscopy on analysis of dried LML

(a)

(b) (c)

Figure 1.2 (a) Process of removing the stalks as preparation for drying; (b) Lemon myrtle leaves ready to be dried; (c) Milling process of dried LM leaves (Bakri, 2015)

Novelty:

There is not much report on drying of LML especially in terms of drying kinetics, application of heat pump drying on LML and the use of non-destructive techniques including near infrared and dielectric spectroscopy on LML analysis. These non-destructive techniques serve as useful information on monitoring the changes of LML during dehydration process . The rapid, manageable and non-destructive measurement is aimed to characterise moisture content in dried LML and reduce the time of analysis. The non-destructive technique also ensures the quality of LML is guaranteed and the market acceptability of dried LML is not compromised. Therefore, this study intended to evaluate the drying kinetics of LML by using various drying methods, the stability of dried product and non-destructive

techniques analysis. The findings of this study are expected to fill the knowledge gap on drying of LML.

1.3 Research objectives

1.3.1 To evaluate the drying performances including drying kinetics, effective moisture diffusivity and activation energy of oven-, vacuum- and heat pump dryer for drying of lemon myrtle leaves (LML).

1.3.2 To analyse the physical qualities (moisture content and colour), phytochemical content (total phenolic content, radical scavenging activity and ferric reducing antioxidant potential) and volatile retention of LML dried with different methods.

1.3.3 To analyse the effect of different drying air velocities of heat pump drying method on colour, phytochemical content and volatile retention of dried LML.

1.3.4 To determine the effect of storage conditions and packaging methods on the quality of LML dried with different methods.

1.3.5 To evaluate the quality (moisture content) of dried LML by using non-destructive techniques of dielectric and near-infrared spectroscopy.

1.4 Research scope

- Determination of drying characteristics

  LML were dried by using three different techniques; namely oven drying (OD), vacuum drying (VD) and heat pump drying (HPD). For OD, the drying temperature was set to 40, 50 and 60°C. VD was also performed at the same temperature range with the pressure set to 50 mbar. HPD was done by setting the relative humidity at 20.85 ± 0.93% RH and different levels of drying air velocities in the range of 15 to 30%. Drying kinetics and drying rates were examined based on the moisture content profile.

- Determination of thermal-physical properties

  Equilibrium moisture content was measured at the end of drying. The effective moisture diffusivity, D_{eff} was determined from the sample moisture based on Fick's second law of diffusion. The activation energy, E_a was determined by using Arrhenius equation.

- Evaluation of colour changes

  Colour assessment was performed to evaluate the colour changes in LML leaves subjected to oven-, vacuum- and heat pump drying, respectively. The values of lightness (L^*), greenness (a^*) and yellowness (b^*) were measured. Chroma,

hue angle, total colour difference (TCD) and browning index were calculated to assess colour changes.

- Evaluation of volatile content

  The extraction of LML essential oil was done by using simultaneous distillation-extraction (SDE) procedure. LML essential oil was analysed for its volatile content by using gas chromatography equipped with mass spectrometry (GC-MS).

- Evaluation of total phenolic content and antioxidant retention

  LML samples were subjected to total phenolic content and antioxidant activity analyses by using Folin-Ciocalteu assay, 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical scavenging assay and ferric reducing antioxidant potential (FRAP) assay, respectively.

- Investigation of the storage stability of dried product

  LML dried products of OD, VD and HPD were stored at different packaging conditions and temperatures. The stability of the LML dried products was assessed in terms of its moisture content, antioxidant activities and colour changes throughout the six-months storage period. Comparisons were made for the its quality changes on normal and vacuum seal (Fullwell DZ-400/2ES) packaging stored at room temperature (25°C) and

chilled (4°C) condition. Analysis on the moisture content, antioxidant activities and colour were performed on monthly basis.

- Evaluation on non-destructive analysis technique on dried products

  Non-destructive technique (NDT) analyses were performed to assess the feasibility of monitoring the moisture content on the sample throughout the drying process. Near-infrared (NIR) and dielectric spectroscopy were performed to estimate the moisture content of the dried LML. The calibration models were done on different dehydration level of moisture to evaluate the accuracy of each NDT analysis.

1.5 Significance of study

This study would result in the discovery of a suitable and energy efficient dryer for the drying of LML. The dryer would be alternatively employed in the agriculture industry by reducing the moisture content and preserving good quality of dried products and its functionality. Furthermore, results of this study could benefit the field of agriculture bioprocessing where efficient processing helps to reduce the cost of operation, ensure market acceptability and supply high quality agriculture products to consumers demand.

1.6 Thesis overview

Chapter 2 discussed the literature review of the samples, drying processes on oven-, vacuum- and heat pump drying. This chapter also discussed the extraction of the essential oil of LML by using simultaneous distillation and extraction with its biochemical antioxidant activities of functional products. At the end of Chapter 2, there is literature on the product stability throughout storage and potential of non-destructive technique of analysis on dried products.

Chapter 3 consists of detailed description of sample and materials used in the experiments. It is also detailing on the procedure for drying methods and conditions. Furthermore, this chapter also discussed the post-drying analyses including physical (moisture content and colour), physicochemical and the product stability. By the end of Chapter 3, the non-destructive techniques namely near-infrared and dielectric spectroscopy were in place to determine the moisture content of the dehydrated LML.

Chapter 4 is divided into four main subtopics. Firstly, the subtopic covers for the results and discussion on effect of drying methods and conditions on the drying performances, namely the drying kinetics, moisture ratio, effective moisture diffusivity and activation energy of oven-, vacuum- and heat pump drying on LML. After that, this

subtopic was followed by the results and discussion on the effect of drying methods and conditions on biochemical retention. In this part, results on total phenolic content, antioxidant activities and volatile retention from LML EO were presented and discussed. The second subtopic covers the results and discussion on the effect of different drying air velocities of HPD on drying performances, colour and biochemical and volatile content. Moreover, Chapter 4 included the dried leaves stability study where data on moisture content, colour assessment, total phenolic content and antioxidant activities for duration of 6 months for different storage conditions. Finally, the feasibility of conduction non-destructive techniques to analyse moisture content on dried leaves was reported at the end of Chapter 4.

Chapter 5 is the conclusion on relation to the objectives of the study. The most suitable drying method and condition were proposed to achieve an efficient drying process with the highest retention of biochemical content and its stability during 6 months storage. The sensitivity of near-infrared and dielectric spectroscopy for non-destructive analysis on determining moisture content from dried LML was also concluded in Chapter 5. Finally, a list of recommendation and future works that can be explored as initiated from this study was mentioned to end Chapter 5.

CHAPTER 2 LITERATURE REVIEW

In this study, it focused on the effect of drying on stability of active ingredients of *Backhousia citrodora* (lemon myrtle) dehydrated leaves. The aims of this study were to evaluate the drying performances of oven-, vacuum- and heat pump dryer, analyse the physical quality of dried LML, analyse the effect of different drying air velocities on heat pump drying performances as well as the different storage conditions effect on the dehydrated LML and feasibility of using non-destructive techniques on LML's quality evaluation. To date, there is not much report on postharvest of lemon myrtle leaves especially on the drying kinetics, the use of heat pump dryers and dehydration impact on the leaves' quality.

Chapter 2 consists of three following sections. The first section covers the literature review of lemon myrtle leaves on its nutritional values and uses. The first section is followed by the review on lemon myrtle leaves drying and its product quality. The second section describes the effect of drying methods and conditions on the drying performances as well as the effect on the quality and stability of dried product. The end of Chapter 2 mentions the review about near-infrared and dielectric spectroscopy as non-destructive analyses namely near infrared and dielectric spectroscopy on product's quality determination.

2.1 Lemon myrtle

Backhousia citriodora or commonly known as lemon myrtle is an Australian native rainforest tree. This plant is commercially grown in Queensland and North Coast of New South Wales, Australia. The tree normally reaches a height of 6 to 8 m. This tea tree-like plant was introduced to Malaysian fields in 2009 by an authorized company (Qzen Plantations Sdn. Bhd.). To date, a successful nursery and fields can be found in Sekinchan, Selangor and Kuala Linggi, Melaka.

First introduced from Australian in 2009, locally produced lemon myrtle leaves and oil are traded in the international market. Australia's largest commercial grower, Australian Native Products, claims it is now poised to extend its supply to the global tea market, estimated at more than USD 12.6 billion, following the recent expansion of its Australian plantations on lemon myrtle leaves (Lemon myrtle, the Australian native tea, 2020). This crop has a lot of potential to be explored as industrial crop because of its versatility. Lemon myrtle is reported to have functionality as it has high antioxidant content as well as antibacterial and antimicrobial activities. Lemon myrtle products have market worldwide and to date, the demand is more than its production (Sandrang et al., 2013). Hence, this high demand on lemon myrtle products make it to be potentially a main commodity crop.

Figure 2.1 shows the industrial distillation process of lemon myrtle essential oil in Kuala Linggi, Melaka, Malaysia. The process to obtain the essential oil is called steam distillation. Leaves that have been separated from the stalks are placed into a vessel. The process is followed by feeding steam produced from a boiler through pipes to each vessel. The steam extracts essential oil from the leaves and which is then cooled by a condenser. The yield from the condensation consists of oil and water. Therefore, the next process is the separation of water and oil by using sodium sulphate and a separator. The leaves are extracted at a rate of 60 mL/min. The produced oil is kept in an aluminium canister to protect it from vapourising and deterioration of oil quality.

Figure 2.1 Industrial distillation process to produce Lemon Myrtle essential oil (Source: Bakri, 2015)

2.1.1 Nutritional values

This herb has a distinguished aroma and taste that is similar to a blend of lemon, lemongrass and lime. With 90 to 98% of citral, one of the most important essential oil constituents, lemon myrtle essential oil is the world richest known natural source of citral (Sandrang et al, 2013). The nutritional value of lemon myrtle leaves is listed in Table 2.1.

Table 2.1 Nutritional value of lemon myrtle leaves (Source: Konczak et al. ,2009)

Ingredient	Value (per 100 g dry weight)	Ingredient	Value (per 100 g dry weight)
Energy	18.9 KJ/100 gm	**Mn**	1.28 µg
Moisture	82 g	**Fe**	5.77 µg
Protein	-	**Mo**	5.5 µg
Fat	-	**Mg**	188.4 µg
Carbohydrates	-	**K:Na**	65.5
Total Sugar	-	**P**	114.1 µg
Fibre	-	**Folate**	71.0 µg
Ca	1583.2 µg	**K**	1258.7 µg
Zn	1.055 µg	**Vitamin E**	21.2 mg
Cu	0.474 µg	**Na**	1.92 µg

According to Table 2.1, lemon myrtle is exceptionally rich in calcium with 1583.2 µg/100 g dry weight. Lemon myrtle also is a good source

of magnesium (Mg) and Vitamin E. It also contains folate, phosphorus and potassium with 71.0, 114.1 and 1258.7 µg/100 g dry weight, respectively.

2.1.2 Applications

Under the novel food category approved by European Union, lemon myrtle is used as a dried herb, flavouring agent, and herbal tea in Australia (Sultanbawa, 2016). In Malaysia, lemon myrtle processing is divided into two potential categories which include extraction of essential oil from fresh leaves and drying of leaves for spices and extract products development. From these semi-finished products, many functional products can be developed for cosmeceutical and nutraceutical purposes. Hence, because of its broad application in the local industry, lemon myrtle is regarded as a new potential crop for Malaysia (Sandrang et al., 2013).

Lemon myrtle leaves (LML) are potentially very useful in perfumery as it consists of odoriferous principle of lemon scented oils that contains citral as its major compound (Svoboda and Greenway, 2003). As it is susceptible to oxidation, citral is usually converted into geranyl nitrile, a more stable nitrile that is commonly used in cosmetic and household preparation (Ueno et al., 2002). The leaf and flower are used in tea blends and beverages, dairy, biscuits, breads, confectionery, pasta, syrups, liqueurs, flavoured oils,

packaged fish (salmon), dipping, simmer sauce and highly valued for their strong lemon-flavour (Konczack et al., 2010). LML also serves as alternative for lemon-flavouring from lemon-fruits' acids in milk-based foods, such as cheesecake, lemon flavoured ice-cream and sorbet because of non-curdling properties that are usually associated with lemon-fruit acidity (Konczack et al., 2010).

Backhousia citriodora or lemon myrtle leaves are used as a bush food component aside from its application in toiletries and cosmetics production because it demonstrates antibacterial activity on both gram positive and negative bacteria including *Aeromonas hydrophilia, Alcaligenes faecalis, Enterobacter aerogenes, Escheria coli, Klebsiella pneumoniae, Bacillus cereus and B. subtilis* in the range of 6.0 to 8.3 mm of inhibition zone (Cock, 2013). A compound in LML that influences antimicrobial activity against *Staphylocci spp.* is geraniol (Pattnaik et al., 1996). LML also possesses compounds that can be used as antiviral agent. As reported by Burke et al. (2004), the Australian lemon myrtle's essential oil appears to be moderately efficacious and safe in the treatment of molluscum contagiosum, common contagious viral disease in children. The results reported by Wilkinson et al. (2003) also showed that lemon myrtle oil is an effective antifungal and antiseptic agent that has potential in preservation and prevention of microbial spoilage. Lemon myrtle essential oil is revealed as the most potent fumigant

to control mycelium growth and spore germination in controlling brown rot caused by fungus *Monilinia fructicola* in nectarines (Lazar-Baker et al., 2011).

Citral is a key component of essential oil extracted from several lemon scented herbal plants including lemon myrtle. Citral can be potentially used as an agent for leukemia treatment, as it induces the apoptosis on NB4 cells in association with activation of caspase-3, down regulation of Bcl-2 and NF-kB expression and up-regulation of Bax expression with involvement of mitochondria-mediated pathway (Xia et al., 2013).

Lemon myrtle is also applied in poultry industry. There is a report that LML essential oil can be potentially used as a feed additive to control the gut colonization of pathogenic bacteria, *Clostridium perfringens* (Zrustova et al., 2006). LML are exported in dried form, mostly to the European Union and the United States, where it is used as specialty tea as well as for cooking, and healing purposes (Sultanbawa, 2016). Thus, it is important to adapt a suitable post harvesting technique not just to reduce the risk of spoilage, it is also to ensure dried leaves of high quality with its functionalities.

2.1.3 Drying of lemon myrtle leaves

In Malaysia, LML are dried by using withering process for commercial production (Sandrang, 2014). In terms of LML drying, Buchaillot et al. (2009) reported the application of fluidised bed dryer on the LML drying. The fluidised bed dryer was set to three different temperature (30, 40 and 50°C). The effect of the drying temperature on the retention of volatiles and colour was investigated. The two principle components of lemon myrtle oil, neral and geranial, showed declining effect as the drying temperature decreased. This might be due to prolonged drying leading to thermal degradation of the essential oil components. The shortest time of drying at 50°C resulted the highest retention of the volatiles. Furthermore, drying at higher temperature formed a surface layer sealing that reduced the diffusivity of the volatile components. The reduced mass transfer limits the high-molecular-weight of volatiles compounds such as neral and geranial and resulted in higher retention compared to drying at lower temperature. Another quality aspect of drying result is the colour preservation. As the drying progressed, the green colour parameter, *a** values increased, indicated that the loss of green colour with gradual development of dull yellowness as a result of changes in *a** and yellow colour parameter, *b** values. Colour changes are also affected by the temperature of drying, as the temperature increased, larger colour degradation was analysed (lightness colour parameter, *L** and *a** values increased and *b**

value decreased). The degradation of greenness in dried product was explained by the degradation of chlorophyll during heating. In conclusion, drying at 50°C appeared to be the best compromise with good retention of volatile and tolerable degradation of the colour for the market acceptability.

Another researcher, Saifullah et al. (2019) reported on the effects of different drying methods on the extractable phenolic compounds and antioxidant properties from dried LML subjected to hot air drying, vacuum drying, microwave drying, shade drying and freeze drying. It was found that the preservation of total phenolic content (TPC), total flavonoids (TFC), proanthocyanidins, gallic acid, hesperetin and antioxidant activities of LML were significantly influenced by the drying methods and conditions. Freeze dried samples gave the highest antioxidant capacity among all dried samples. Shorter duration of hot air drying and vacuum drying at high temperatures preserved greater biochemical content and antioxidant properties compared to longer drying times at low temperatures. The best condition for hot air drying and vacuum drying was determined as 90°C for 75 min and 90°C for 120 min, respectively with vacuum pressure of 69 ± 1 kPa for vacuum oven. Phenolic compounds and antioxidant potential of LML were also affected by microwave power and radiation time, offering the best recovery at 960 W for 7 min by microwave drying. Whereas, shade drying and sun drying required

longer period of drying which were 12 and 2 days, respectively with shade drying samples gave the lowest FRAP value compared to samples dried with other methods. The variation in the antioxidant activities of LML was influenced by the drying methods and conditions. Saifullah et al. (2019) also suggested that due to minimum drying time and energy, microwave drying can be used for industrial drying of lemon myrtle leaves.

Though LML owns a lot of functional properties and highly potential commodity, there is not much published information on how the drying methods and conditions influence the quality of the dried leaves especially locally grown LML in terms of biochemical and volatile contents, colour and storage stability. From the literature, the drying application on LML only limited to fluidised bed dryer (Buchaillot et al., 2009), hot air drying, vacuum drying, microwave drying, shade drying and freeze drying (Saifullah et al., 2019). There is not much information regarding the application of designer dryer especially heat pump dryer on maximising retention of dried LML. The exploration of using non-destructive techniques for dried LML characterisation also was prospected as a new knowledge in LML drying. These non-destructive techniques serve as useful information on monitoring changes that the LML experience as the dehydration process take place. The rapid, manageable and non-destructive measurement is aimed to characterise moisture content

in dried LML and reduce the time of analysis. The non-destructive technique also ensures the quality of LML is guaranteed and the market acceptability of dried LML is not compromised. In addition, this new study also intended to the enrichment of LML drying in a way to determine suitable drying method and condition not just for the preservation of this highly potential herb, but also for the market acceptability in a cost-efficient method.

2.2 Drying

Drying is a common method used for food preservation. The process of drying involves the conversion of liquid, solid or semi-solid into a solid product with lowered moisture content. Most drying processes involve evaporation where moisture in the product evaporates with the application of heat. Foods are dried to serve several purposes. Preservation and extension of a product's shelf life is the main reason of drying food. Products including herbs also are dried to change their physical form (for instance powder or flakes); to facilitate transportation of the products and value adding for products. For several cases, pre-treatment of food product is required prior to drying in a way to improve the effectiveness of a drying operation.

2.2.1 Drying performances

2.2.1.1 Drying kinetics

Drying is the process of movement of moisture from a solid through various mechanisms like molecular diffusion, capillary diffusion, surface diffusion or the combinations of the mechanisms. The drying performance can be assessed from the drying curve. Drying curve is defined as a plot of drying rate which is the change of moisture content in a certain time interval versus the remaining water content or the moisture content. The production capacity of a dryer is determined by the drying rate. (Berk, 2018).

A typical drying curve consists of three major periods, namely the constant rate period and two distinctive falling rate periods. As shown in Figure 2.2, drying begins with the phase of rising rate where the rate of drying increases as water is being removed from the solid (point A to B). This phase is usually short and sometimes mentioned as initial adjustment period. Point B represents the equilibrium temperature conditions of product surface and drying air.

From point B to C of the curve, this phase is called constant rate period which represents the removal of the unbound water from the product. The drying rate remains nearly constant as water is removed and drying curve shows a horizontal line. During this

period, the drying is controlled by the rate of external heat and mass transfer to a film of free water that is available on the evaporating surface. This phase is also independent to the moisture movement in the product. However, many food and agricultural products seldom exhibit the constant rate period due to the absence of a layer of water on surface. For this case, since internal heat and mass transfer determine the rate at which water is available at the exposed evaporating surface, the falling rate is applied (Mujumdar and Devahastin, 2008).

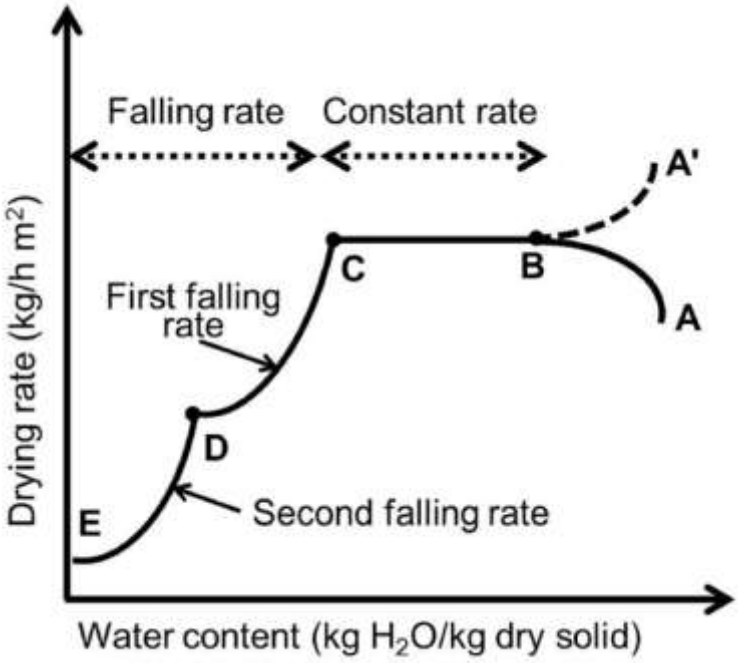


Figure 2.2 Drying rate curve (Silva et al., 2018)

At Point C, the rate is governed by the internal flow of liquid or vapour. The falling rate period is reached, when the drying rate starts to decrease because of internal transport limitation, hence the moisture cannot migrate to the surface easily and below critical

moisture content (w_c). The falling rate period can be divided into two distinguished period, first and second falling rate periods. The first falling drying rate occurs when wetted spots on the surface diminish until the surface is dried or partially unsaturated (Point D). Second falling rate period begins when the surface is completely dry or totally unsaturated. The plane of evaporation recedes from the surface. The mechanism for the transport phenomena varies among the product composition and drying conditions.

Rayaguru and Routray (2010) studied the effect of different heat pump drying conditions on the drying kinetics and quality of aromatic *Pandanus amaryllifolius* leaves. The drying of Pandanus leaves took place mainly under falling-rate period. The mechanism of moisture movement during this period was through diffusion. The initial part of the drying curve was influenced by the low relative humidity in heat pump drying, while later part of drying was influenced by the temperature which acted as the driving force for moisture diffusion.

Premi at al. (2010) also reported the absence of constant rate period in convective drying of drumstick leaves (*Moringa oleifera*). The drying process took place in falling rate period. The drying rate was affected by higher temperature than lower temperature at high moisture content. It was also found that the moisture loss at the beginning was faster than at the end of the drying process which

was influenced by the reduction in moisture content as the drying occur.

Overall, the drying characteristics can be studied through a drying curve where the leaves' moisture content usually decreases over time, with an initial rapid decrease and followed by steady decrease toward equilibrium (Mujaffar and John, 2018). Although drying of food and agriculture products usually takes place in falling rate period, a lot of information can be gained from the drying curve plot. The effect of temperature, drying conditions and other factors such as relative humidity (Rayaguru and Routray, 2010) can be studied to optimise the drying process, which suited the sample material. The effect of drying rate over moisture can be determined to help increase the drying efficiency, as it depends on the materials and drying conditions.

2.2.1.2 Moisture diffusivity and activation energy

According to Taheri-Garavand and Meda (2018), the main mechanism of moisture removal in drying process of food and agriculture products is liquid and/or vapour diffusion. For a simple analysis of the change in moisture content, one dimensional diffusion is considered and Fick's second law of diffusion is used to calculate the flow of moisture within a material that is usually expressed as moisture diffusivity.

The main mechanism involves in moisture transport to the surface to be evaporated in drying process is diffusion. As it allows for an evaluation and comparison of the drying air velocity, the effective moisture diffusivity, D_{eff} become one of the most important indices used to evaluate drying kinetics of agricultural products (Botelho et al., 2018).

Generally, moisture diffusivity in non-food materials is higher than in food materials (Geankoplis, 1993). This is due to the strong binding in water molecules to the cell wall and complicated biopolymers in food structure. Various literatures have been published on the reviews of the experimental determination and compilation of moisture diffusivity for food and agricultural products (Zogzas et al., 1994; Zogzas et al., 1996). The values of moisture diffusivity usually fall between 10^{-12} to 10^{-8} m^2/s for drying of food materials (Zogzas et al., 1996).

Activation energy is the minimum energy required to initiate moisture diffusion from a product (Shahi et al., 2012). Arrhenius's equation has been extensively employed to calculate the energy of activation in way to describe the effect of drying air temperature on the effective moisture diffusion coefficient (Taheri-Garavand and Meda, 2018).

Effective moisture diffusivity and activation energy have been calculated as a requirement for the ideal dryer design. Some of the researchers have studied the effective moisture diffusivity and activation energy on various agricultural products. For instance, Doymaz (2011) reported the characteristics, effective moisture diffusivity and activation energy of grape leaves under hot air at 40, 50 and 60°C in a cabinet dryer. Generally, the effective moisture diffusivity increased with increased drying temperature ranging from 4.13×10^{-10} to 1.83×10^{-9} m^2/s with activation energy estimated as 64.56 kJ/mol.

The effective moisture diffusivity showed dependencies on factors such as drying air temperature and air velocities. Therdthai and Zhou (2009) reported that the variation of effective moisture diffusivity was dependent on the drying conditions of mint leaves (*Mentha cordifolia* Opiz ex Fresen). The effective moisture diffusivity (ranging from 0.9648 to 1.19×10^{-11} m^2/s under hot air drying) increased as the drying temperature increased. Aktas et al. (2017) reported the same trend in heat pump drying of mint leaves where the effective moisture diffusivity was found to increase with an increase in air flow rate and was calculated in range of 3.50 to 5.88×10^{-11} m^2/s. Akpinar and Toraman (2013) varied the drying temperature from 40, 50, 60 to 70°C and set the air velocities at 0.8, 1.5 and 3 m/s. The average

effective moisture diffusivity values were reported from 2.807 x 10^{-10} to 6.977 x 10^{10} m^2/s over the temperature and velocity range. Air temperature and air velocity were proven to affect the moisture diffusivity. The effective moisture diffusivity values were increased with increasing air temperature and velocity. The optimum activation energy of ginger slices drying was found to be 22.72 kJ/mol within 40-70°C temperature range at 0.8 m/s air velocity.

Overall, the effective moisture diffusivity increases with temperature but varies with the moisture content of the product. At higher temperature, the moisture diffusivity is higher. An increase in temperature results in increase in heating energy and causes an increase in water activity which in turn leads to higher moisture diffusivity. Although a lot of information has been given on the effective moisture diffusivity and activation energy for various agricultural products, none of the published literature reported the effective moisture diffusivity and activation energy of LML during drying.

2.2.2 Drying methods

One of the main factors that affects the drying kinetics and the quality of the food product is the drying method. Commonly, hot air drying is used to evaporate water out of a product. The heat treatment also usually causes the deterioration of sensitive

compounds in the product that leads to losses in the bioactive compounds in functional products.

In recent years, there is an increasing interest in antioxidant testing of natural products (Dontha, 2016). Plant phenolic compounds have been frequently reported as bioactive components associated with antioxidant properties and health benefits (Pierson et al., 2012). The properties of antioxidant on neutralising free radicals in vitro through their inherent redox properties explain the association of total phenolic content with health benefits (Vuong et la., 2013). It is expected that the effectiveness of a natural product is proportional to its phenolic content, since antioxidant and antiradical properties are mainly attributed to the presence of phenolic compounds (Dontha, 2016).

In plant materials, 80% of the total antioxidant capacity is the phenolic compounds (Podsędek, 2007). The functionality of a product is evaluated in terms of the phytochemical content of the product. An optimum drying method and condition of a specific sample are important to ensure the phytochemical content retain in the dried samples thus maintaining its functionality. Antioxidants belong to the biologically important compounds in herbs (Adámková and Kouřimská, 2015). Antioxidant compounds in herbs include both dietary one, such as vitamin A, C and E and also reasonable

quantities of non-nutritional antioxidants for example beta-carotene, carotenoids and total phenolic compounds. Total phenolic content (TPC) is one of the antioxidant effects that is measured using the Folin-Ciocalteu reaction. TPC is usually expressed as gallic acid equivalent (GAE) as gallic acid is used as the standard in estimating the TPC. Ferric reducing antioxidant potential (FRAP) assay uses the antioxidants as reductants in a redox-linked colourimetric method to an easily reduced oxidant, Fe(III). The spectrophotometer readings of the assay are related to the reducing potential of electron-donating antioxidant present in the sample. Thus, FRAP assay can rank the reducing potential and the antioxidant potential of the sample . FRAP is expressed as ascorbic acid equivalent (AAE). Free radical scavenging assay (DPPH) activity assay is the most widely used method to determine the primary antioxidant activity of antioxidant compounds in plant and fruits extract (Kumar et al., 2015). DPPH is a test based on the reaction rate between a stable free radical, 2,2-diphenyl-1-pycrylhydrazyl (DPPH) and antioxidants (Mediani et al., 2014). DPPH also uses ascorbic acid as the standard which is the same as FRAP assay and the results are expressed as AAE, ascorbic acid equivalent.

2.2.2.1 Heat pump drying

Heat pump drying (HPD) is an excellent match for thermal applications for both industrial and domestic uses; such as water heating, solar drying or space cooling due to its low temperature thermal requirement. When in used for drying process, this unit of operation is widely known to be energy efficient. Heat pump dryer is more cost effective, as it can extract and re-utilize the heat of a circulating air in a closed cycle and the latent energy of the water vapour is obtained from the drying process, unlike conventional air dryers where no re-utilization of heat is involved in drying process (Brushlyanova et al., 2013). Because of the energy efficiency is improved, the usage of heat pump dryer has attracted significant attention and interest in food processing industry recently. Due to the capabilities of controlling drying conditions, heat pump dryers are applied in drying of heat sensitive materials (Strømmen et al., 2002). This will benefit the drying of LML as to preserve heat sensitive and volatile bioactive content (citral) and thus the phytochemical properties of the leaves are retained throughout drying.

During heat pump drying (HPD), the cooled and dehumidified air is passed over the condenser of the refrigerator to decrease the percentage of relative humidity (Babu et al., 2018). HPD dehumidifies the air, heats and recirculates it through the drying chamber. The circulation of air makes HPD require less energy as

the system can recover the latent heat in a closed loop. This advantage results in higher intact of colour and its active substances. For the extensive review on heat pump drying by Goh et al. (2011), HPD has control over the drying temperature and relative humidity which could improve the drying efficiency to a great extent in terms of preservation of concentrated nutrients. Hence, it is important to investigate the parameters that can optimize the efficiency of HPD for instance the drying air velocity.

In term of drying performances, the HPD drying characteristics are influenced by the physicochemical properties of the sample. Brushlyanova et al. (2013) studied the drying kinetics of different fruit pomaces dried with the heat pump dryer. The fruits that were used in their study were apple, blueberry (wild and cultivated), raspberry, chokeberry and morello pomaces. These fruits were dried at 45°C until equilibrium moisture content was achieved. The drying kinetics of the fruit pomaces (except raspberry) showed two drying periods: constant-rate drying and falling-rate drying. The differences of drying time and the drying rate were most probably due to variation of structures and compositions as well as the differences of the initial moisture content of the fruit pomaces. Nair et al. (2017) also reported the influence of the nature of the material in determining the optimum operating temperature in green peas drying by using a heat pump assisted fluidized bed dryer. As the inlet

temperature depends on the properties of the product to be dried, the increase in the inlet temperature resulted in decrease of the drying time. The increase in drying temperature led to an increase in moisture diffusivity and thus increased the drying rate and reduced drying time.

Heat pump dryer has been reported as a dehydration machine for preservation of the functionalities. Heat pump dryer has been applied to dehydrate and preserve the phytochemical and physical characteristics of *Spirulina* sp. (Costa, 2015). Samples of dried *Spirulina* sp. in the heat pump dryer showed a loss of phycocyanin content and total antioxidant activity (TAA) from 15 to 83% and from 11 to 87%, respectively. The total colour difference (TCD) values were in the range from 4.22 to 13.51. Potisate et al. (2015) studied the changes of phytochemicals, antioxidant capacity and colour in *Moringa oleifera* leaves after heat-pump assisted dehumidified air drying. The phytochemicals of the leaves were retained during drying process. Under optimum storage conditions, the total flavanoids, total phenolics and antioxidant activity of dried *M. oleifera* leaves maintained at reasonable levels.

The drying temperature in vacuum heat pump drying of Shiitake mushroom and Jinda chilli were significantly affected the colour of the dried product (Artnaseaw et al., 2009). Vacuum HPD is an HPD

that operates at vacuum condition. The HPD is designed with 1.2 kW liquid ring vacuum pump to control the drying pressure at 0.1, 0.2, 0.3 and 0.4 bar in the drying chamber. For Jinda chili, the colour degradation increased with an increase in the drying temperature or a decrease in the vacuum pressure. Meanwhile, the colour degradation of Shiitake mushroom increased as the drying temperature or vacuum pressure decreased.

Controlled dryers are on demand as it will not only result in good quality but also be economical, efficient and time saving (Patel and Kar, 2012). Fadhel et al.(2014) studied the effect of different meteorological Malaysia conditions on drying characteristics of lemongrass in a solar assisted chemical heat pump dryer. The effect of two air drying speeds with three representative days (sunny, cloudy and semi-cloudy days) were investigated in their study. The moisture content of the lemongrass was reduced from 9.1 db to 0.36 db. The highest total system energy input from the experiment was determined from the sunny day, followed by semi-cloudy and finally the cloudy day. The decrease of the drying efficiency is attributed by the reduction of the energy at the condenser and hence decrease in the solar radiation. In another work, Ceylan and Gürel (2016) studied the mint leaves dehydrated by using the solar-assisted fluidized bed dryer integrated with a heat pump. The combination of solar air collector, a parabolic through collector and heat pump dryer

increased the efficiency of the drying. The integration of the solar energy systems made the temperature control in this dryer feasible.

Overall, the heat pump dryer has been applied in various agricultural products to reduce the moisture content and thus prolong the shelf life of the products. The mechanisms of re-circulating heat into the drying chamber as a controlled closed-system operation did give advantage to the drying as it ensures the hygiene security of the product. The variation of the quality and drying performances varies between product's composition and initial moisture content. Furthermore, both latent and sensible heat can be recovered as the energy efficient heat pump in drying system is utilised (Patel and Kar, 2012). The waste heat recovery ability attracts agropreneurs' attention on adapting heat pump dryer into their industry. Hence, its application on the heat sensitive material is governed for maximising the quality of the product as guarantee of the preservation of its functionality.

2.2.2.2 Oven drying

For bigger scale of food drying process, food industries usually choose hot air convective dryers as their drying option. The drying of herb is crucial in prolonging the shelf life and preserve the functionality of the herb. The microbial growth and forestalls of biochemical changes are inhibited by drying of herbs but the changes

on appearance and aroma can affect the herb quality (Abdul Razak et al., 2016).

Oliveira et al. (2015) studied the quality and bioactive characteristics of galega kale dried by a convective air drying at temperature range of 35 to 85°C. As the temperature of the drying increased, time of drying reduced. The physical properties of dried kale had greater changed with the increase in temperature. The colour changes were indicative of nutritive losses and product worsening. Due to the nutritional parameters, the antioxidant activity was also affected by the drying temperature. The low TPC value analysed from dried galage kale sample demonstrated the quality deterioration and nutritive loss by the convective drying at higher temperature.

Wu et al. (2013) studied the effects of drying temperature of *Angelica sinensis* (AS) leaves on the antioxidant, colour and sensory properties. The drying temperature significantly affected the antioxidant activities of AS leaves. As the temperature of drying increased from 30 to 90°C, it was found that the antioxidant activities including DPPH free radical and total phenolic content decreased with the optimum temperature of 50°C for AS leaves dehydration.

Pin et al. (2009) studied the quality and drying kinetics of betel leaves (*Piper betle* L.) as effect of different oven drying temperature.

The temperature of drying in this study was varied from 40 to 80°C and 70°C was found to be the optimum drying temperature. Drying at 70°C resulted in higher retention of the major compounds including hydroxychavicol and eugenol. However, lower retention of these two major compounds were obtained as AS leaves was dried at 80°C. It was also reported that the increased of drying temperature decreased the total drying time.

Ashtiani et al. (2017) studied the effect of hot-air drying on peppermint leaves. In this drying process, 30, 40, 50°C and 0.5, 1, 1.5 m/s were selected as temperature and air velocities of drying, respectively. At higher temperature and higher air velocities, hot-air drying exhibited larger moisture diffusivity. The activation energy was determined in range of 21.476 to 27.784 kJ/mol and these values fell between the range of 12.7 to 110 kJ/mol reported on various food products (Xiao et al., 2011; Troncoso and Pedreschi, 2007).

As colour regard, drying method has impact on the physical appearance of the dried products. Rababah et al. (2015) studied the effect between oven drying at 40°C and air drying at room temperature of common Mediterranean herbs including mint, sage, lemon balm and thyme on colour changes, as comparison to fresh samples. The colour of fresh and dried samples was measured by

using colourimeter for its *L**, *a** and *b** values and total colour difference and their colour intensity (chroma) values were calculated. Higher chroma was found in air dried samples compared to samples dried by oven. Because of more pigment destruction, oven drying caused more browning reaction.

Mediani et al. (2014) studied the effects of different drying methods, namely freeze drying, air drying and oven drying of *Cosmos caudatus* on free radical scavenging activity and total phenolic content (TPC). The TPC of samples was affected by the rate of moisture loss. As higher moisture loss obtained from air drying, lower TPC retention was determined from the air-dried samples. Thermal degradation was related to heat treatment as resulted in lower TPC content in oven dried samples.

Roshanak et al. (2016) reported the evaluation of seven different drying treatments in in terms of antioxidant activity of green tea (*Camillia sinensis* or *C. assamica*) leaves. The seven drying treatments attempted in the study were sun-, shade-, oven 60°C-, oven 80°C-, oven 100°C-, microwave- and freeze-drying. Among the methods, the highest radical scavenging activity (IC_{50}=167.166 µg/ml) revealed from sample oven dried at 60°C while microwave sample exhibited the lowest value of IC_{50}. These findings showed the effect of drying treatment that reduced the values of radical

scavenging activity specifically at higher drying temperature of 100°C.

It can be concluded that the application of oven drying has been applied to all kinds of agricultural products including plant leaves, vegetables and many more. The drying temperature shows significant effect in reduction of drying time, however, the quality of the dried products is deteriorated. To be beneficial for human consumption, food characteristics such as form, colour, taste and texture, as well as nutritional components such as antioxidants, need to be preserved at high levels (Jin et al., 2014).

2.2.2.3 Vacuum drying

Vacuum drying is the removal of moisture from a substance by means of creating a vacuum. The application of vacuum drying in agricultural products that are hygroscopic and heat sensitive that operates at low temperature and the removal of moisture is facilitated by the reduced pressure (Argyropoulos and Muller, 2014). Therefore, vacuum drying often serves as an option for dehydration of bioactive compounds in the agricultural products.

Alibas (2007) reported the energy consumption and colour characteristics of nettle leaves using microwave, vacuum and

convective drying. Different vacuum drying conditions at temperature of 50 and 75°C and pressure of 20 and 50 mm [Hg] were attempted for nettle leaves drying. Increasing temperature from 50°C to 75°C in vacuum drying resulted in the reduction of drying time from 65 min to 35 min. The greater loss in brightness was determined at temperature of 50°C and 50 mm [Hg] vacuum combinations.

Argyropoulos and Muller (2014) reported the effect of vacuum drying on the sorption behaviour and bioactive compounds of Lemon balm (*Meliisa officinalis* L.). The lemon balm leaves were dried at temperature of 30°C and pressure of 25 mbar. Vacuum drying resulted in the lowest essential oil content in Lemon balm leaves compared to convective and freeze drying. The content of the oil components was also affected by vacuum drying, as it had lower geranial that resulted in strong earthy, hay-like flavour. The colour of dried Lemon balm leaves was deeper green as indicated by the larger negative green colour parameter, *a** value and thus contributing to higher hue angle value.

Therdthai and Zhou (2009) studied the characteristics of mint leaves (*Mentha cordifolia* Opiz ex Fresen) after microwave vacuum drying and hot air drying . Dried mint leaves subjected to vacuum drying were light-green/yellow which were better than dark-brown colour

leaves dried with hot air drying. The vacuum dried mint leaves also showed highly porous microstructure and higher rehydration rate compared than those hot air dried leaves.

Alibas (2009) reported the characteristics of collard leaves subjected to vacuum drying. Collard leaves were dried at temperature of 50 and 75°C and pressure of 0.4, 50 and 100 mmHg. The lowest hue angle was observed in all vacuum dried samples. The retention of ascorbic acid content in vacuum dried samples was comparable to that of microwave dried samples. The ascorbic acid content was in a range of 71.12 to 84.01 mg per 100 g for all vacuum dried samples.

Hossain et al. (2010) studied the effect of drying method of six Larniaceae herbs (rosemary, oregano, marjoram, sage, basil and thyme) on the antioxidant capacity. The samples were dried using air drying, vacuum oven drying and freeze drying. Each drying method exhibited different effect on the antioxidant capacity of the herbs. Air drying resulted in the highest total phenolic content, rosmarinic content and antioxidant capacity compared to the other two methods. However, during storage, vacuum oven drying resulted in higher TPC and FRAP content in rosemary and thyme, which was better than freeze dried samples.

Overall, for the protection of the bioactive compounds in the agricultural product, vacuum drying has been widely applied. The utilisation of no drying medium dryer has potentially been used to preserve colour, heat sensitive active ingredients as well as the microstructure of the products. For the performance of vacuum drying is dependent on the vacuum-temperature combination as well as the materials. Vacuum drying is said to be applicable to high-values crops like herbs on small scale, as it is too costly for large-scale production of a commodity (Bazyma and Kutovoy, 2005).

Table 2.2 shows the summary on heat pump-, hot air- and vacuum drying under different conditions on various leaf drying application. Different drying methods and conditions can be applied on the preservation of plant leaves. When selecting or designing an appropriate drying process, their valuables constituents require consideration of various factors such as the nature of the food material, energy efficiency and cost of the process. Conservation of critical bioactive compounds requires a system that minimises exposure to light, oxidation and heat.

Table 2.2 Summary on leaf drying on different methods and conditions

Method	Leaves dried	Drying conditions				Reference
		Temperature (°C)	Pressure (mbar)	RH (%)	Air flow (m/s)	
Heat pump	Mint	35	-	-	2, 2.5 and 3	Aktas (2017)
		45 and 50	-	-	-	Ceylan and Gurel (2016)
	Spirulina	30, 40 and 50	-	-	-	Costa (2015)
	Lemon-grass	55	-	-	1 and 3	Fadhel (2014)
	Kaffir lime	40, 50 and 60	-	-	0.4	Poomsa-ad (2011)
		40	-	-	0.5	Phoungchandang (2008)
	Moringa oleifera	50	-	-	0.5	Potisate (2015)
	Pandanus amaryllifolius	35	-	27	-	Rayaguru (2010)
Hot air	Angelica sinensis	30, 50, 70, and 90	-	-	-	Wu (2013)
	Mint	60 and 70	-	-	-	Therdthai and Zhou (2009)
	Collard	50 and 75	-	-	-	Alibas (2007)
	Grape	40, 50 and 60	-	14-45	2.0	Doymaz (2011)
	Mediteranean herbs	40	-	60	-	Rababah (2015)
	Sea Buckthorn	50	-	-	1.0	Kyriakopoulou (2013)
	Cosmos caudatus	44.5	-	-	-	Mediani (2014)
	Bay	45	-	-	-	Diaz-Maroto (2002)
	Betel	40 to 80	-	80-88	-	Pin (2009)
	Galega kale	35 to 85	-	-	1.2	Oliveira (2015)
	Stevia	60 and 80	-	-	-	Halim (2019)
	Carica papaya L.	40, 50, 60, 120	-	-	-	Raja (2019)
	Lemon myrtle	50, 70 and 90	-	-	-	Saifullah (2019)
	Guava	50, 60 and 70	-	-	-	Shravya (2019)
	Lemon thyme	70 and 80	-	-	2.0	Paslawska (2020)

Vacuum	Mint	8, 9.6 and 11.2	133.3	-	-	Therdthai and Zhou (2009)
	Nettle	50 and 75	27 and 67	-	1.0	Alibas (2009)
	Collard	50 and 75	0.5, 67 and 133	-	-	Alibas (2007)
	Lemon balm	30	25	-	-	Argyropoulos and Muller (2014)
	Larniacea herbs	70	600	-	-	Hossain (2010)
	Olive	50, 70 and 90	100	-	-	Elhussein and Sahin (2018)
	Lemongrass	60	-	-	-	Hashim (2019)
	Moringa oleifera	60	100	-	-	Laurence (2019)
	Basil, drumstick and mint	50	-	-	-	Kaur (2019)
	Amaranthus	40 and 50	680, 800 and 853	-	-	Nighitha and Mathew (2019)

2.3.3 Volatile content

In food application, LML are used as fresh or dried herbs, because of its special aroma and flavour. Its essential oil (EO) is used in flavouring. The sensory of the LML as a dried milled leaf has been described as the aroma of lemon candy, perfumed with some menthol notes (Sultanbawa, 2016). LML essential oil (EO) predominantly contains citral (3,7-dimethyl-2,7-octadienal) with two main isomeric aldehydes: neral and geranial (Figure 2.3 (a) and (b)).

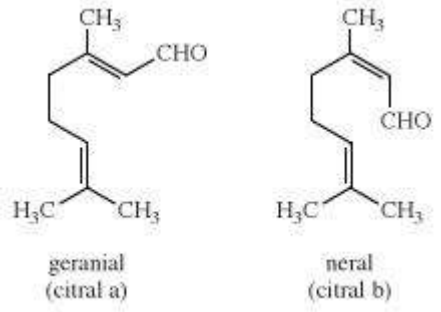


Figure 2.3 Chemical structure of citral aldehydes: (a) geranial (trans-citral) (b) neral (cis-citral) (Source: https://www.britannica.com/science/citral)

Lemon myrtle leaves (LML) essential oil (EO) predominantly contains citral (3,7-dimethyl-2,7-octadienal) with two main isomeric aldehydes: neral and geranial. LML is said to have the highest citral content which is 90-98% compared to lemongrass (75%) and lemon (4%) (Sandrang et al., 2013). Citral is the major chemical component which consist of neral (citral A) and geranial (citral B)

with percentage range of 32.0 to 40.9% and 46.1 to 60.7%, respectively. The volatiles compound can be extracted by using several extraction methods. One of the commonly used methods is simultaneous distillation and extraction (SDE).

Major chemical compound in LML essential oil (EO) is citral. The preservation of the volatile compound in dried product is an indicator for preservation of its functionality due to the phytochemical retention. By using simultaneous distillation and extraction (SDE), dried LML were extracted for its volatile compound and analysed by using GC-MS. Yang (2013) also reported that the yield of extract of volatile compound can be enhanced by using SDE as the distillation and extraction were done simultaneously.

The chemical composition of the LML EO is shown in Table 2.3. LML has been reported to have the highest citral content which was 90-98% compared to lemongrass (75%) and lemon (4%) (Sandrang et al., 2013). Citral is the major chemical component which consists of neral (citral A) and geranial (citral B) with percentage range of 32.0 to 40.9 and 46.1 to 60.7, respectively.

Table 2.3 Chemical composition of the essential oil of lemon myrtle (Sultanbawa, 2016)

Component	Percentage
β-Myrcene	0.1-0.7
6-methyl-5-Hepten-2-one	0.1-2.5
(±)Linalool	0.3-1.0
Citronellal	0.1-0.9
Iso-neral (*Cis-iso* citral)	0.6-2.7
Iso-geranial (*Trans-iso* citral)	1.0-4.2
Neral (citral A)	32.0-40.9
Geranial (citral B)	46.1-60.7
Trans-geraniol	0.4-0.7

The volatile compounds can be extracted by using several extraction methods. One of the common methods is simultaneous distillation and extraction (SDE). The basic protocol of SDE involves a flask of sample that contains the sample and boiling water. A solvent flask contains organic solvent such as pentane. The sample flask and solvent flask are heated to 100°C and 50°C, respectively. Kawakami (1997) also studied the characterization of SDE for tea aroma and analysis of tea by using gas chromatography-Fourier transform infrared spectrometry-mass spectrometry (GC-FTIR-MS). The author found out that EO extracted by using SDE method and analysed by GC-FTIR-MS was able to characterise the compounds

that contributed to the tea aromas compared to the brewed extraction and stream distillation under reduced pressure method for extraction and single gas chromatography analysis method.

Gas Chromatography – Mass Spectrometer (GC-MS) is one of the most efficient technique for the separation, identification and quantification of volatile compounds (Yang et al., 2013). Generally, polar or mid-polar GC columns were used to investigate volatile compounds. Diaz-Maroto et al. (2002) reported the effect of different drying treatments on the volatiles in bay leaf (*Laurus nobilis* L.). They found that SDE yielded better quantitative GC-MS results with the highest amount of 1,8-cineole and linalool of 33.28 and 24.53 µg/g dry weight, respectively.

2.3 Dried product stability assessment during storage

2.3.1 Storage stability assessment of dried lemon myrtle leaves

Stability of a dried food product is usually influenced by several factors including the packaging material, temperature of storage, relative humidity and etc. Packaging of material involves selection of material types for instance different types of plastics such as polypropylene (PP), polyethylene (PE) and foil type (aluminium foil).

Packaging fulfils numerous purposes that include preventing contamination during transportation and distribution, preserving the product's integrity and maintaining the flavour profile of the product (Chaliha et al., 2013).

As on the suitable storage for lemon myrtle leaves, Chaliha et al. (2013) studied the effect of materials and storage on major volatile compounds of lemon myrtle and two other Australian native herbs, namely anise myrtle and Tasmanian pepper. The effectiveness of alternate high-barrier property packaging materials, polyvinylidene chloride coated polyethylene terephthalate/casted polypropylene (PVDV coated PET/CPP) and polyethylene terephthalate/polyethylene terephthalate/aluminum foil/linear low-density polyethylene (PET/PET/Foil/LLDPE) at ambient temperature for 6 months was investigated and compared. After 6 months, the greatest volatile loss from lemon myrtle was observed in traditional LDPE packaging followed by storage in PVDC coated PET/CPP and PET/PET/Foil/LLDPE with 87, 58 and 23% loss, respectively. However, different results were obtained for anise myrtle and Tasmanian pepper leaf. There was no significant difference between the two high barrier property packagings, as the losses were less than 30%. Therefore, it was suggested that the selection of correct packaging material is important to retain the volatile content of herbs.

Chaliha et al. (2013) reported on the storage conditions effect on lemon myrtle , the leaves were commercially dried and stored at room temperature. No information is reported on retention of the biochemical and volatile content when samples stored at cold-storage temperature. Furthermore, the effect of storage condition on the oven-, vacuum- and heat pump dried LML has not been studied. In this study, it was intended to investigate the effect of storage conditions (packaging condition and temperature of storage) on the quality of LML dried with different methods.

2.3.2 Storage stability assessment of other leafy materials

During storage, migration of water, oxygen and volatile can take place through packaging materials and thus jeopardise the quality of the dried product. Hence, vacuum packaging also provides alternative of preserving and ensuring quality of food products. The intent of vacuum packaging is normally done by removing oxygen from the container to extend the shelf life of food thus limiting the possibilities of bacteria or fungi growth and preventing losses of volatile components (Joubert et al., 2010). Araújo et al. (2017) evaluated the effect of air-drying temperature and storage conditions on the nutritional and sensory properties of dried galega kale after 5 months of storage. The effect of normal packaging and

vacuum packaging on samples air-dried at 40, 55 and 70°C was investigated. The lowest drying temperature (40°C) resulted in the highest retention of vitamin C, total phenolic content, total antioxidant capacity and chlorophyll content with were 62, 38, 92 and 48%, respectively. Generally, packaging without vacuum improved nutritional features of galega kale when compared to packaging with the vacuum. All nutritional compounds decreased after the 5 months of storage, especially vitamin C was the most affected compound. Similar results were obtained in relation to the colour properties concluding the vacuum packaging did not add value to the dried product. It is concluded that the quality of dehydrated galega kale did not get improved by application of vacuum packaging as similar results were obtained from with vacuum and without vacuum packaged samples .

As for the impact of drying methods and storage conditions on quality aspect, Chong and Lim (2012) reported on the drying effects on the antioxidant properties of microwave-, oven-, sun- and freeze-dried herbal tea from selected Vitex species for 30 days storage. Oven- and sun-drying were found to cause more deterioration to the antioxidant properties of the herbal leaves, while microwave and freeze drying maintained the antioxidant properties of the leaves and having least physicochemical properties change during storage. On the other hand, Korus (2011) studied the effect of preliminary

processing, method of drying and storage temperature on the level of antioxidants in air- and freeze-dried kale (*Brassicca oleracea* L. var. *Acephala*) leaves after 12 months of storage. The dried kale was stored in glass jars at two different temperatures: ambient temperature, 18-20°C and cold temperature, 8-10°C. The drying of the kale leaves resulted a decrease in the content of the biochemical components ; loss of the biochemical content that depended on the extent of drying methods. After 12-months storage, freeze dried leaves retained higher levels of total identified polyphenols, vitamin C and Trolox equivalent antioxidant activity (TEAC) which were 36, 15 and 33% more than air-dried samples. In addition, better retention of antioxidants was observed in dried leaves stored at cold-store temperature.

Apart from that, storage conditions also show the impact on the volatile retention of dried herbs. Baritaux et al. (1992) reported the effect of drying and storage on essential oil production of basil, *Ocimum basilicum* L. The basil was dried at 45°C and stored in sealed aluminium polyethylene polyamide bags at 4°C. The losses of total essential oil after drying were 19, 62 and 66% at 3, 6 and 7 months storage, mainly responsible by significant decrease in the content of methylchavicol and eugenol.

The long-term effects of drying conditions on the colour of tarragon leaves during storage were studied by Arabhosseini et al. (2007). The leaves were dried at 45, 60 and 90°C and colour was measured after 15, 30, 60 and 120 days. Colour parameters changed during the storage period. The largest change in colour expressed by hue angle was found in the material dried at 90°C, while sample dried at 45°C exhibited the smallest change. The colour changes were assumed to be affected by the moisture content, as having higher moisture content, more colour changes occur during the storage period. Based on these results, it is recommended to dry tarragon leaves at lower temperature (5°C) to keep the quality closes to the fresh material.

Generally, drying methods, packaging conditions and temperature of surrounding during storage were proven to have impact on the quality of dehydrated leaves. From the aspect of quality preservation, lower storage temperature ensures better preservation than storage at higher temperature. The packaging conditions also play important role to reduce the quality deterioration and thus enhance the target export markets, where longer shelf life is crucial in shipment and transportation of bulk herbal products (Chaliha et la., 2013).

2.4 Non-destructive analysis of plant leaves

2.4.1 Near-infrared analysis

Near-infrared (NIR) spectroscopy measures the absorption of electromagnetic radiation including wavelengths from 750 to 2500 nm and its spectra include broad bands that arise from absorptions in overlapping wavelengths (Prieto et al., 2017). These measurements mode include transmittance, interactance, transflectance and two modes that are applied most, diffuse transmittance and diffuse reflectance (Huang et al., 2008). The diffuse reflectance mode was used in an analysis of caffein content in *Camillia sinensis* (green tea) leaves (Luyapert et al., 2003); prediction of minerals in *Moringa oleifera* leaf powder (Rebufa et al., 2018); nitrogen content in rubber leaves (Tang et al., 2018); and identification of aging stage in coffee leaves (Mees et al., 2018). NIRS was also used in many applications to determine chemical compositions, colouring strength, maturity and dry matter (Zalacain et al., 2005; Xie et al., 2014; Zhang et al., 2017).

The near-infrared (NIR) spectrometry measures the absorption of light by the sample. For solid samples, the light sources rich in NIR radiation illuminate samples in diffuse reflectance modes (Wang and Paliwal, 2007) due to strong scattering characteristics (Alander et al., 2013). In diffuse reflectance, the change of the signal intensity

is attributed by scattering and absorbance of the solid sample (Pasquini, 2003). The NIR spectrum is presented as the relative reflectance that measures the proportional amount of reflected light measured from a sample, relative to the amount of reflected light measured from a reference plate (white reference). NIR measure the absorbance and reflectance of light to the determine the moisture content. The more that light is absorbed, the higher the moisture content in the sample. Generally, the shape and intensity of a NIR spectrum is influenced by the type of sample, distance between the detector and sample and instrument optics (Sundaram et al., 2015).

NIR spectroscopy analysis has been used in many plant analyses, as it is rapid, easy-to-use and non-destructive, which can be carried out in-line or on-line. Furthermore, NIRS has been used for the quantification of quality parameters of green tea (Chen et al., 2012; Lee et al., 2014; Sinija and Mishra, 2011) and black teas (Hall et al., 1998). Zhao et al. (2006) and Chen et al. (2007) demonstrated the application of NIR spectroscopy on green tea, black and Oolong teas to identify the tea measured at spectral range of 11000 to 3800 cm^{-1} in 1.928 cm interval. Many researchers have proven the application of NIR for moisture content determination on leaves including commercial tea samples (Diniz et al., 2015); potted plants of *Epipremnum aureum* (Zhang et al., 2012); and hardwood leaves

(Shimbori and Kurata, 2017). All the authors reported good prediction results were obtained from the full spectra range (1200-2500 nm) compared to short ranges of spectra.

Bonfil et al. (2005) studied the use of NIR transmittance and reflectance spectroscopy to monitor water contents in wheat flag leaves. As the finding of the study, NIR reflectance could be used to monitor the water status as it can give accurate indications in real time. However, NIR transmittance was significantly inferior and might not be appropriate for further application.

For the determination of moisture content in *Artemisia annua* L. dry powder leaves, hand-held near-infrared spectroscopy devise was used in the wavelength range of 1210 to 1797 nm, specifically for moisture content determination (Camps et al., 2011). A model used to determine moisture content was particularly accurate with the value of moisture prediction and strong NIR absorption band was found at around 1400 to 1440 nm.

Neto et al., (2017) used wavelength range of 500 to 1039 nm on Vis/NIR spectroscopy to estimate the water and chlorophyll status in sunflower leaves under moderate to severe water stress. As a result, the chlorophyll and water content in sunflower leaves were

estimated based on spectral signature indicated by the external validation results obtained for the proposed models.

NIR spectroscopy has also been reported as a method to determine the quality of plant leaves on post-drying treatment. In analysis of macro and micro nutrients, the sugarcane leaves were dried, ground and sieved through 1 mm prior to NIRS analysis (Galvez-Sola et al., 2015). They found that scanning a dried ground sample obtained repetitive spectra, as NIR spectroscopy was verified with a high proficiency in the estimation of nitrogen and calcium as well as acceptable estimation of potassium, magnesium, iron and zinc, but not for boron, copper and manganese content estimation.

2.4.2 Dielectric spectroscopy

Dielectric spectroscopy is the measurement of electric properties when exposing a material to an applied electric field (Jiao, 2019). Sensing the dielectric properties can be used for rapid measurement of their moisture content, since dielectric properties of materials are highly correlated with the amount of water in materials such as agricultural products and food materials (Nelson and Trabelsi, 2012). Measurement of food electric properties has been explored extensively that includes detecting moisture content, sugar content, maturity, freshness and etc. There are several factors that have been

found to influence on dielectric properties, including composition of materials, frequency of the alternating fields, temperature of the material, and bulk density of particulate materials in addition of moisture content of the sample (Nelson and Trabelsi, 2012).

Hashmi et al. (2014) used dielectric spectroscopy to determine the dielectric properties of fresh leaves of *Ficus benghalensis*, *F. elastica*, *F. religiosa* and *Morus nigra* (Moraceae) and *Hibiscus rosa-sinensis* and *Gossypium hirsutum* (Malvaceae) and found that the dielectric responses of different species belonging to the same family were qualitatively similar, whereas different families were substantially different. Reduction rate of time conductance varied due to anatomical and phytochemical properties.

Jördens at al. (2009) reported the application of terahertz time-domain spectroscopy to evaluate of leaf water content on dried-compressed leaf disc of coffee leaves (*Coffee arabica* L.). By manipulating the frequency from 0.3 to 1.8 THz, the influence of scattering became important at higher frequencies and was modelled by a Rayleigh roughness factor.

Afzal and Mousavi (2008) reported the use of dielectric spectroscopy to estimate the moisture content in maize leaf. As the leaf moisture increased, the dielectric capacitance increased too. Capacitance

measured at 100 kHz was more accurate compared to higher frequency (1 MHz), but desirable model was obtained from 1 MHz frequency.

Mahani et al. (2016) studied dielectric spectroscopy on water hyacinth plant collected from agriculture drainage. High-resolution impedance analyzer spectrometer (Schlumberger Solarton 1260) was used in the measurement of pressed disc on its permittivity (ε') and dielectric loss (ε'') was calculated from the output parameters i.e capacitance C (ω) resistance R (ω), loss factor (tanδ) and dielectric properties were found to depend on the applied electric field frequency, magnitude of heating power as well as concentrations of pollutants.

For the comparison of water stress and unstressed condition on tomato plant leaves, Emmerik et al. (2015) used dielectric spectroscopy to determine its moisture content. The measurement of magnitude of the reflection coefficient was done at 1201 frequencies from 2.1 to 4.1 GHz and depended on the dielectric constant. As the tomato leaves dried out (water stress condition), the difference in frequency between the leaf and the Teflon block decreased corresponding to a low dielectric constant.

All of the reports covered wide application of non-destructive techniques in plant leaves application but none of them has been applied to LML. From the literature, the NIR spectroscopy has been successfully applied to determine the moisture content on various teas and plant leaves with its spectral data performed. Dielectric spectroscopy also has been utilised on various leaves species to assess their moisture content at different frequencies. From this study, the application of NDT on LML will provide information on the characterising LML dehydrated leaves and thus increase the diversity of NDT application on agricultural products especially plant leaves.

It is important to use NDT for characterisation of dried LML, as rapid measurement of the dried LML moisture content can be done and reduce the time of analysis. Hence, this effort will also reduce the cost and losses of sample for analysis. On the other hand, the NDT will also ensure the quality of the dried LML and help to produce dried LML with high quality for local and global markets.

2.5 Research Gap

According to the literature review, only two papers reported on the drying on LML using fluidized bed dryer (Buchaillot et al., 2009), hot air-, freeze-, microwave-, shade-, sun- and vacuum drying (Saifullah et al., 2019) and its impact on the appearance, biochemical and volatile content. None of the reports mentioned the use of heat pump

dryer as the dehydration process of LML. Furthermore, the intention to gain more knowledge on the drying performances, namely the drying kinetics, effective moisture diffusivity and activation energy is important to determine the most suitable and efficient drying method for LML in way to preserve the physical and physicochemical content of LML. This study was intended to fulfil the research gap in LML drying especially in exploring alternative methods suitable for the heat sensitive materials in LML and their stability throughout storage under different storage conditions. It was also proposed to evaluate the feasibility of non-destructive techniques of analysis which was never applied on the characterisation of dried LML that might be useful as rapid and on-line monitoring of the mass and heat transfer of the drying processes.

CHAPTER 3 METHODOLOGY

In this chapter, it will mention the details of sample, materials, each drying method and condition parameters involve in this study. Next, this chapter is mentioning the procedures to analyse the physical characteristics of different dried products including moisture content, colour assessment, biochemical and volatile content. This chapter is followed by describing the arrangement for LML stability during storage and lastly, the non-destructive techniques analysis of near-infrared and dielectric spectroscopy with its parameter's settings involved in this study.

3.1 Samples

Lemon myrtle leaves (variety: Linpinwood B.) were freshly harvested from MARDI Research Station, Serdang, Selangor, Malaysia (latitude: 2.991597, longitude: 101.702195) and used for the drying experiment on the same day. The leaves were separated from the stalks before drying. The leaves were cleaned and screened for insect bites before the drying process. The sampling time was also uniformed between 8 to 9 am every sampling to obtain similar initial moisture content of the leaves. Figure 3.1 shows the plantation plot of LML in MARDI Research Station.

Figure 3.1 Lemon myrtle plantation plot in MARDI Research Station

3.2 Drying of lemon myrtle Leaves

Samples of 500 g of LML (90 mm x 30 mm x 1 mm) were dried using three different drying methods, namely oven drying, vacuum drying and heat pump drying. Samples were arranged on a perforated wire mesh tray (L: 45.5cm x W: 26.5cm x H: 10cm) (Figure 3.2). All drying experiments were conducted in triplicates. Data collection for each drying treatment was done by weighing samples at specific time intervals at every 15 min for the first 3 hours, every 30 min for the next 3 hours and every 2 hours using a digital balance (Mettler Toledo, Switzerland). The samples were dried until constant weight was obtained in three consecutive readings. The dried products were

determined for its bone dry weight and stored in air-tight plastic containers in dark at room temperature for further analysis.

Figure 3.2 The lemon myrtle leaves on a perforated wire mesh **tray ready for drying**

3.2.1 Heat pump drying

The heat pump dryer (HPD) was fabricated by i-Lab Sdn. Bhd (Selangor, Malaysia). Heat pump dryer is operated by passing hot air (45°C) through the drying chamber and allowing moisture to be removed from the sample. The moist air passes through the evaporator coils and transfers the 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 and further cooling results in the condensation of water. As a result, the cooled air is dehumidified. Then, the dehumidified cool air is brought to contact with the condenser of the heat pump system. At the condenser, the cooled air absorbs the heat from condenser and hence its

temperature is increased. Increasing the temperature of the dehumidified air further reduces the relative humidity of the drying air. Thereafter, the dehumidified drying air (at 45.94±2.31°C with relative humidity of 20.85 ± 0.93%) was charged into the drying chamber. The closed loop system repeats its cycle until equilibrium moisture of sample is achieved. The dimension of the dryer is 2.3 x 1.0 x 2.1 m (length x width x height) and comes with two drying chambers measured 330 x 330 x 1000 mm each. The working power of the HPD is 700 W. A probe (LOGPROBE 60, E + E Elektronik Ges. M.b.H, Austria) was used to measure the air velocity, the frequency of the fan power was adjusted until the desired air velocity was obtained.

Figure 3.3 Heat pump dryer unit

3.2.2 Oven drying

Oven drying (Model: UF750, Memmert, Germany) involved drying at temperature of 40, 50 and 60°C (OD40, OD50 and OD60). The temperature of the oven ranges from 20 to 300°C. The drying air is supplied by forced air circulation by air turbines that is adjustable in 10% and set as 100% for every experiment. The drying chamber of the oven has a dimension of 1224 x 1720 x 784 mm and its working power is 7000 W.

Figure 3.4 Oven dryer

3.2.3 Vacuum drying

The drying process was carried out by using a vacuum oven (Model V200; Memmert, Germany) at temperature of 40, 50 and 60°C and vacuum pressure of 50 mbar. The temperature and pressure of the vacuum oven range from 20 to 200°C and 5 to 1100 mbar, respectively. The material was heated by using heating plate. The drying chamber of the vacuum oven has a dimension of 550 x 600 x 400 mm and its working power is 1200 W. A digital pressure display is mounted on the vacuum oven to indicate the vacuum pressure in mbar.

Figure 3.5 Vacuum Dryer

3.3 Drying performances evaluation

3.3.1 Moisture ratio and drying rate

The moisture content of the sample can be calculated by using Eq. (1) (Aktas et al., 2009):

$$M_{db} = \frac{w_i - w_b}{w_b} \quad \text{Eq. (1)}$$

or;

$$M_{wb} = \frac{w_i - w_b}{w_i} \quad \text{Eq. (2)}$$

where;

M is the moisture content (kg/kg)

w_t is the weight of sample at a time t (kg)

w_i is the initial weight of sample (kg)

w_b is the mass of bone-dry solid (kg)

The moisture content at any given time to the initial moisture content (both relative to the equilibrium moisture content) is the moisture ratio. It can be calculated for each time interval using Eq. (2) (Aktas et al., 2017):

$$MR = \frac{M - M_e}{M_o - M_e} \quad \text{Eq. (3)}$$

where;

M is the moisture content at specific time (g water/g dry basis)

M_e is the equilibrium moisture content (g water/g dry basis)

M_o is the initial moisture content (g water/g dry basis)

The moisture ratio (MR) is the moisture content of sample at any given time to the initial moisture content (both relative to the equilibrium moisture content). It can be calculated for each time interval using Eq. (3). The MR was simplified to M/M_o because the relative humidity of the drying air continuously fluctuated during drying.

Drying rate indicates the rate of moisture loss of the sample due to drying, there is a loss of moisture with time. The drying rate (DR) for each drying method was calculated using Eq. (4) as stated by Doymaz (2011):

$$DR = \frac{M_{t+dt} - M_t}{dt} \qquad \text{Eq. (4)}$$

where;

M_{t+dt} is the moisture content at $t + dt$ (g water/g dry basis)

M_t is the moisture content at specific time (g water/g dry basis)

t is the drying time (min)

3.3.2 Moisture diffusivity and activation energy

The effective moisture diffusivity is a function of drying temperature and product moisture content to model the drying process for various agricultural resources. To determine the moisture diffusivity, the Fick's second law of diffusion was adopted to fit the experimental data and expressed as according to (Shi, 2006; Doymaz, 2011) as Eq. (5):

$$\frac{\partial M}{\partial t} = \frac{\partial}{\partial x}\left(D_{eff}\frac{\partial M}{\partial x}\right) \qquad \text{Eq. (5)}$$

where; M stands for moisture content (g water/g dry solid), D_{eff} is effective moisture diffusivity (m^2/s), t is time and x is the length of the moisture flow from center or the distance from center in the direction of moisture flow. By using method of slopes, Fick's diffusion equation was applied to determine the effective moisture diffusivity, in this study for a flat surface. The LML samples were considered having a slab geometry due to their flat surface and less than or equal to 0.3574 mm thickness. The equation was as Eq. (6) as stated by Doymaz (2011):

$$MR = \frac{M_t}{M_o} = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n-1)^2}exp\left[-\frac{(2n-1)^2\pi^2 D_{eff}t}{4L^2}\right] \qquad \text{Eq. (6)}$$

where L is half of the thickness of the sample (mm), n is a positive integer, and t shows the duration of the drying process (min). For cases with longer drying time, Eq. (6) can be simplified to Eq. (7) which includes only the first term of the series (Aktas et al., 2009).

$$\ln MR = \ln\left(\frac{8}{\pi^2}\right) - \left(\frac{\pi^2 D_{eff} t}{4L^2}\right) \quad \text{Eq. (7)}$$

The effective moisture diffusivity can be extracted from a graph plotted the drying data from the experiments as ln (*MR*) against time. From Eq. (7), the ln (M*R*)-time diagram would show an inclined line with its slope (K) according to Doymaz (2011) that is given by the Eq. (8):

$$\mathrm{K} = \frac{\pi^2 D_{eff}}{4L^2} \quad \text{Eq. (8)}$$

The effective moisture diffusivity is also determined using this equation.

The following assumptions for Fick's diffusion model were made for infinite slab body of lemon myrtle leaves:

- Initially, moisture is distributed uniformly within the mass of the sample.
- Mass transfer is uniform with regard to the center
- The surface moisture content achieves equilibrium with the surrounding air's condition.
- Negligible resistance to mass transfer at surface compared to internal resistance of sample.
- The mechanism of the sample's mass transfer is described by the diffusion process.

- Shrinkage is negligible and constant value of diffusion coefficient which could be due to the structural rigidity of LML cellular tissues preventing the shrinkage (Prothon et al., 2003).

To determine the drying characteristics, another important factor is the bonding potential of moisture in non-dry materials. At given moisture content, the energy required to eliminate 1 mole 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 is as given in Eq. (9):

$$D_{eff} = D_o exp\left(-\frac{E_a}{R(T + 373.15)}\right) \qquad \text{Eq. (9)}$$

where T is the drying air temperature (°C), R is the universal gas constant (8.314 × 10^{-3} kJ /mol.K), D_o is the pre-exponential factor of the Arrhenius equation (m^2/s), and E_a is the activation energy (kJ/mol). Eq. (9) can be rearranged into the form of Eq. (10):

$$\ln D_{eff} = lnD_o - \frac{E_a}{R}\left(\frac{1}{T + 273.15}\right) \qquad \text{Eq. (10)}$$

From the slope of a straight line when ln (D_{eff}) was plotted against the multiplicative inverse of absolute temperature (1/(T + 273.15)), E_a was derived.

3.4 Determination of quality parameters

3.4.1 Pre-treatment of lemon myrtle leaves

Pre-treatment was done to ensure the homogeneity of sample. For grinding, the whole LML leaves were put in the ultra-centrifugal mill (Model ZM 200; Retsch, Germany). The powder was sieved with a mesh width of 1.0 mm and this sieved powder was used for further analysis. Dried ground lemon myrtle powder (about 1 g) was put in petri dish (diameter: 25 mm) (Figure 3.6). The petri dish must be fully covered with the ground sample. The sample layer should be at least 0.25 cm thick.

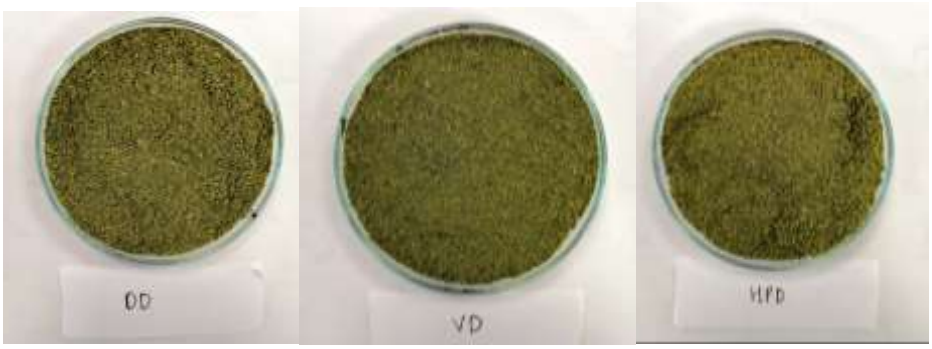


Figure 3.6 Milled dried lemon myrtle leaves of (a) oven dried; (b) vacuum dried and (c) heat pump dried

3.4.2 Moisture content

The bone-dry weight (w_b) was determined by drying the sample in an oven at 105°C for 24 hours (AOAC, 1990).

3.4.3 Colour assessment

The colour of dried samples was determined using a colourimeter (HunterLab ColorFlex EZ) (Figure 3.7). The samples (ground LML) were placed in a transparent glass container (Figure 3.7(b)) and an opaque cover (Figure 3.7(c)) was placed to cover the sample's cup for colour measurement. The *L**, *a** and *b** values of the dried samples were recorded. The *L** value was evaluated from 0 to 100 that expresses darkness to lightness; the positive or negative *a** value relates to the redness or greenness of the sample respectively; while *b** value shows the colour of sample ranging from yellow (positive *b** value) to blue (negative *b** value). Chroma, hue angle and browning index were calculated according to the formulas (Eq.11 to Eq.14) as stated by Oliveira et al. (2015).

$$Chroma = \sqrt{a^{*2} + b^{*2}} \quad \text{Eq. (11)}$$

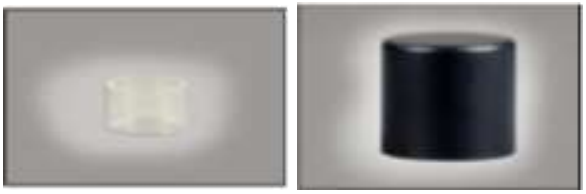
$$Hue\ angle = \tan^{-1}\frac{b^*}{a^*} \quad \text{Eq. (12)}$$

$$Browning\ index = \frac{100\ (X - 0.31)}{0.17} \quad \text{Eq. (13)}$$

where;

$$X = \frac{a^* + 1.75L^*}{5.645L^* + a^* - 3.012b^*} \quad \text{Eq. (14)}$$

(a)



(b) **(c)**

Figure 3.7 (a) Hunter ColorFlex EZ colorimeter unit; (b) glass sample cup and (c) opaque cover (Source: CFEZ User's Manual)

3.4.4 Biochemical analysis

3.4.4.1 Extraction for biochemical activities

Extraction procedure of the dried sample was done by following the method described by Abd. Razak et al. (2015), with slight modifications. The extraction was done by mixing 1.0 g of dried LML with 10 mL of deionised water (Milli-Q Water purification system, Pall C, Illinois, United States) and placed in water bath at 55°C for 10

min. The solution was cooled before it was centrifuged (Model: Centrifuge 5810 R, Eppendorf, Germany) at 10,000 rpm for 10 min. The supernatant was filtered using filter paper (Whatman No.1) and the sample was extracted prior to analysis.

3.4.4.2 Total phenolic content

The total phenolic content estimation was done by following a method of Folin–Ciocalteu as described by Abd Razak et al. (2015), with some modifications. Briefly, 5 mL of the diluted Folin–Ciocalteau reagent (Sigma-Aldrich; Missouri, United States of America) was allowed to react with 1 mL of extract for 3 to 8 min before reacted with 4 mL of 7.5% (w/v) sodium carbonate (Sigma-Aldrich; Missouri, United States of America) at room temperature for 2 h. The mixture's absorbance was measured by using a spectrophotometer (Model: Cary50 UV-Vis, Varian, United States of America) at 765 nm. Based on the absorbance, the TPC value of the LML extract was determined based on the calibration graph (absorbance versus concentration as shown in Figure C1, Appendix C) of gallic acid constructed using 0-200 ppm gallic acid. The results were expressed as mg of Gallic Acid Equivalent (GAE)/g sample.

3.4.4.3 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging activity

The radical scavenging activity of the extract was determined by using a method described by Thaipong et al. (2006), with some modifications. The DPPH stock was prepared by dissolving 24 mg DPPH with 100 mL ofmethanol and placed in the amber bottle. The working solution was obtained by mixing 10 mL of stock solution with 45 mL methanol to obtain an absorbance of 1.1±0.02 units at 515 nm using the spectrophotometer (Model: Cary50 UV-Vis, Varian, United States of America). Approximately 2,850 µL of 2-2-diphenyl-1-picrylhydrazyl, DPPH (Sigma-Aldrich, analytical grade: Missouri, United States) working solution was reacted with 150 µL of extract in dark for 30 min. The absorbance was read at 515 nm using the spectrophotometer. A standard curve was constructed by using DPPH stock solution with concentration in the range of 0-140 ppm of ascorbic acid as shown in Figure C2 (Appendix C). The scavenging activity was determined by using Eq. (15):

DPPH radical scavenging activity (%) Eq. (15)

$$= [(A_{blank} - A_{sample}) / A_{blank}] \times 100$$

where;

A_{blank} = Absorbance of blank sample (Absorbance without extract)

A_{sample}= Absorbance of sample

3.4.4.4 Ferric reducing antioxidant potential (FRAP) assay

Another antioxidant activity that was analysed in LML powder was ferric reducing antioxidant potential (FRAP) values. The FRAP assay was performed according to Benzie and Strain (1996) method, with some modifications. FRAP stock solution was prepared by mixing 300 mM acetate buffer (3.1g sodium acetate trihydrate, $C_2H_3NaO_2.3H_2O$ ((Sigma-Aldrich; Missouri, USA) and 16 mL of acetic acid (Sigma-Aldrich; Missouri, USA)) at pH 3.6, 10 mM TPTZ (2,4,6-tripyridyl-s-triazine, Sigma-Aldrich; Missouri, USA) solution in 40 mM hydrochloric acid, HCl (Merck & Co, New Jersey, USA) and 20 mM Iron (III) Chloride Hexahydrate, $FeCl_3.6H_2O$ (Sigma-Aldrich, Missouri, USA) solution. The preparation of the working solution of this assay was done by mixing 25 mL of acetate buffer, 2.5 mL of TPTZ solution and $FeCl_3.6H_2O$ solution, respectively. Prior to the FRAP analysis, the working solution mixture was warmed to 37°C. Then, in the dark , 150 µl of extract was reacted with 2,850 µl of FRAP solution. After 30 min, the mixture's absorbance was measured by using the spectrophotometer at 593 nm. A standard curve was plotted by calibrating with different ascorbic acid (Sigma-Aldrich, Missouri, United States) concentration ranging from 0 to 200 ppm of ascorbic acid as shown in Figure C3 (Appendix C). The absorbance of FRAP solution at different concentration of ascorbic acid over 30 min was measured and plotted. The value of FRAP were expressed as mg ascorbic acid equivalent (AAE)/g sample.

3.4.5 Determination of volatile content

3.4.5.1 Simultaneous Distillation-Extraction (SDE)

Simultaneous distillation-extraction procedure was performed according to Bidgoli et al. (2014). SDE procedure was done by placing hundred (100) grams of dried LML into a 1.5-L round bottom flask containing 1 L of distilled water. The sample bottle was then connected to the left arm of Likens-Nickerson SDE apparatus (Figure 3.8), while its right arm was connected to a 100- mL round bottom flask with 40 mL of pentane. The sample bottle was heated by a heating mantle (MTops, MS-E102, Korea) to boiling point of water around 100°C, and the solvent was simultaneously heated by another heating mantle (MTops, MS-E102, Korea) to 50°C, higher than boiling point of pentane of 36.1°C.

Then the flask was connected to a distilling receiver with a condenser. Distillation was carried out for 4 h at atmospheric pressure. The essential oil evaporated with steam during the distillation process and was separated from the condensates and collected in the distillate receiver.

The cooling finger was connected to a small pump (Hiblow, HP-80, Japan) which was placed into a chilled water bath. The temperature of circulating cooling water was operated at 10°C. A few grams of

anhydrous sodium sulfate (US Phatmacopeia; Sigma-Aldrich; USA) were added to the extracted distillate and allowed to stand for 1 h for dehydration. The solution was filtered using filter paper. The filtered solution was concentrated by placing the vial under nitrogen purge apparatus with ultra-high purity nitrogen (99.999%) (N_2 Micro, Claind; Italy) until the volume of solution remain unchanged. The essential oil was placed in an amber glass vial for further volatile compound analysis.

Figure 3.8 Simultaneous distillation and extraction (SDE) unit with refrigerated water circulator

3.5.5.2 Gas Chromatography-Mass Spectrometer (GC-MS)

Gas Chromatography – Mass Spectrometer (GC-MS) is one of the most efficient techniques for the separation, identification and quantification of volatile compounds (Yang et al., 2013). LML essential oil was analysed by gas chromatography equipped with mass spectrometry (GC-MS-QP2010 Plus-Shimadzu (Figure 3.9)). The column used in this analysis was Zebron ZB5-ms (30 m x 0.25 mm ID x 0.25 μm film thin). The column temperature was set to 50°C for 1 min, then increased to 130°C at the rate of 7°C/min, and then held for 12.6 min. The injector temperature was set at 250°C (split mode with the ratio adjusted to 5:1, injection volume = 0.2 μL).

Figure 3.9 Gas Chromatography-Mass Spectrometer (GC-MS)

3.5 Storage conditions for product stability

After drying of LML using the three drying methods, cooling was performed to the dried samples before packaging in order to avoid condensation after packaging. Approximately 2 g of dried samples obtained from each drying technique (OD, VD and HPD) was packaged in polyethylene bags (thickness of 0.04 mm). Dried leaves were packed into non-vacuum (N) and vacuum (V) packaging conditions. For vacuum packed, the bags were subjected to vacuum packaging at 1 mbar for 10 s, using vacuum sealer machine (Model: Fullwell DZ-400/2ES, Malaysia) as shown in Figure 3.10. Both packaging conditions were placed in two different temperature conditions, at room temperature (RT) in the range of 22-25°C, 45-58% RH and chilled condition (CH) at an average temperature between 4 to 6°C, 62-75% RH. The arrangement of the storage study conditions is shown in Figure 3.11.

Figure 3.10 Vacuum sealer machine for vacuum packaging

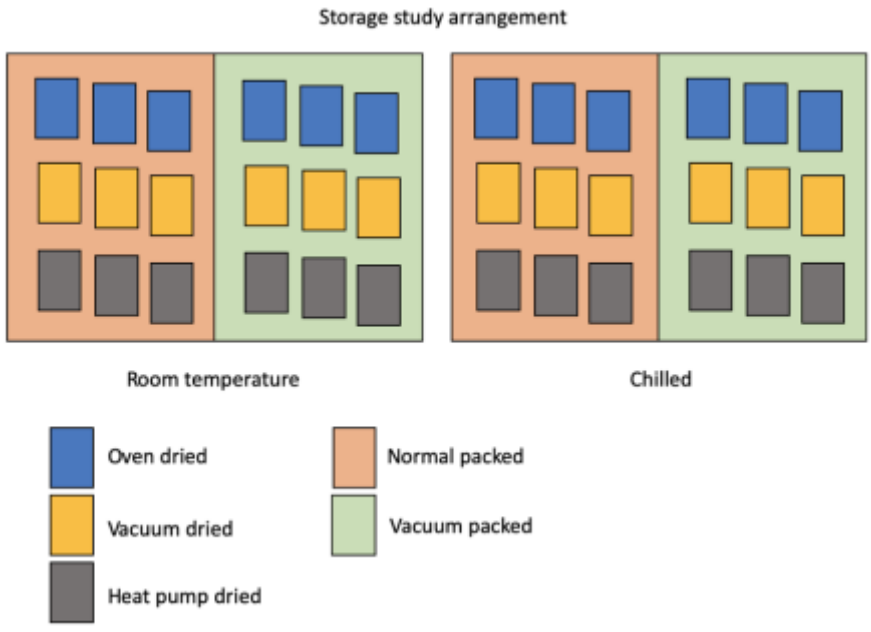


Figure 3.11 Storage conditions arrangement for dehydrated lemon myrtle leaves by using heat pump-, oven and vacuum dried

3.6 Statistical analysis

All data are expressed as mean ± standard deviation. The data were analysed using one- and two-way ANOVA with SAS software (Version 9.4, S.A.S. Institute Inc. Cary, North Carolina, U.S.A.) and differences between means were compared using Tukey's Honest Significance Difference (HSD) at $p<0.05\%$.

3.7 Non-invasive techniques for physical properties analysis of dried lemon myrtle leaves

3.7.1 Near-infrared spectra collection

The experimental set up followed the device configuration as mentioned by Rady et al. (2019) as shown in Figure 3.12. The system comprised a three-dimensional (3-D) printed plastic enclosure especially designed for the measurement. This included the slotted groves at different vertical levels with 1.5 cm apart. The NIR sensor used was a NIRONE S2.0 spectrometer (Spectral Engines, Oulu, Finland). This sensor had a compact shape and light weight (25 x 25 x 17.55 mm, 15 g) suitable for integration with processing technologies, such as dehydrators. The sensor contained two tungsten vacuum lamps with a peak power of 1 W and a single element extended InGaAs detector. The signal-to-ratio (SNR) of the

sensor was 38.75 dB. The sensor has a detection range of 1550 to 1950 nm.

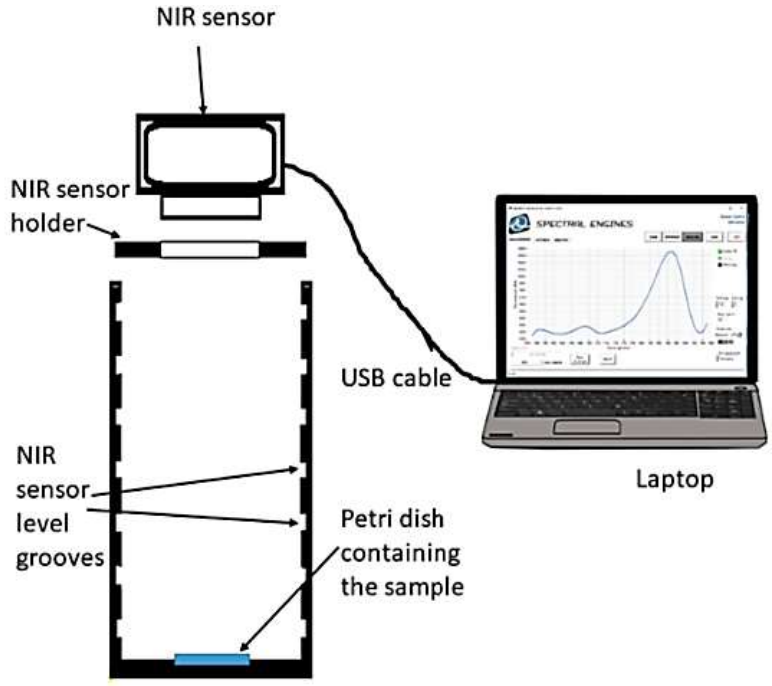


Figure 3.12 Device set up for near-infrared (NIR) analysis

The sensor configuration applied in this study was using a spectral resolution of 1 nm from 1550 to 1950 nm which made 401 data points for each sample measured. Each sample was placed on the holder closest distance to the sensor (1.5 cm). The light intensity ratio used in this study was 100% of the peak power. There were 5 replicated scans for each moisture level and for each scanned sample, there were 3 acquired spectra, with 1 s between each spectrum, and the average of the three spectra was recorded. To obtain the relative reflectance, a reference spectrum was recorded using a standard white disk at the same height and light intensity

(100%), in addition to a dark or background spectrum at 0% light intensity and the same height. In NIR spectra collection near-infrared spectrophotometers are usually sensitive to the change of outer environment conditions such as temperature and humidity. Therefore, the room temperature was kept in 25°C, and the humidity was kept at an ambient level (60% RH) in the laboratory. Moreover, the sensor was turned on at the desirable settings for 10 min to obtain uniform absorbance values.

3.7.2 Dielectric spectroscopy

The dielectric measurement set up was set according to Yuan et al. (2019). Quartz tubes (dimension: 25 mm (internal diameter) x 57 mm (height)) were used to hold the samples. The microwave resonator was used in the measurement system by detecting changes in resonant patterns introduced by different moisture content in dried LML. These changes can be correlated to the total complex permittivity of high (water) and low (LML) phases. When LML of different moisture level was placed at the center of the resonant cavity, the variation of the water volume fraction could be observed by the change of the complex permittivity (dielectric constant and loss), which is indicated by the frequency shift in the resonant pattern inside the cavity.

A resonant cavity method was used to measure the dielectric properties of dehydrated LML. It consisted of a cylindrical copper cavity with hole at the center to hold the quartz sample holder. The resonant was coupled to an external microwave source, Hewlett-Packard 8753C network analyser utilising a 85047A S-parameter test set (300 KHz-6 GHz) (Hewlett-Packard, Palo Alto, California, USA), was achieved with two loop antennas. The diameters of cavity and central hole were 245 mm and 45 mm, respectively and height of the cavity was 24.7 mm. The cavity and Vector Network Analyser (VNA) are shown in Figure 3.13.

Figure 3.13 Benchtop measurement system of a cylindrical resonant cavity

A non-destructive techniques (NDT) namely Near-infrared (NIR) and dielectric (DM) spectroscopy were used to determine the moisture content of dried LML NIR spectroscopy were performed for the spectra range of 1550 nm to 1950 nm. Whereas, dielectric

spectroscopy was done by using the benchtop measurement system of a cylindrical resonant cavity in the frequency range of 912 MHz to 960 MHz.

Figure 3.14 shows a diagram of the apparatus set up. The beginning of the measurement, the empty quartz sample holders' (with the sealer) properties were measured. Then, the sample was placed in the holder and placed at the center of the cavity for measurement. Measurements were made by utilising the resonant mode of TM010 at resonant frequency of 937 MHz at room temperature and SPAN of 50 MHz. The frequency range measured was from 912 MHz to 962 MHz. The number of points was set to 201 and the power of the microwave source to 10 dB m.

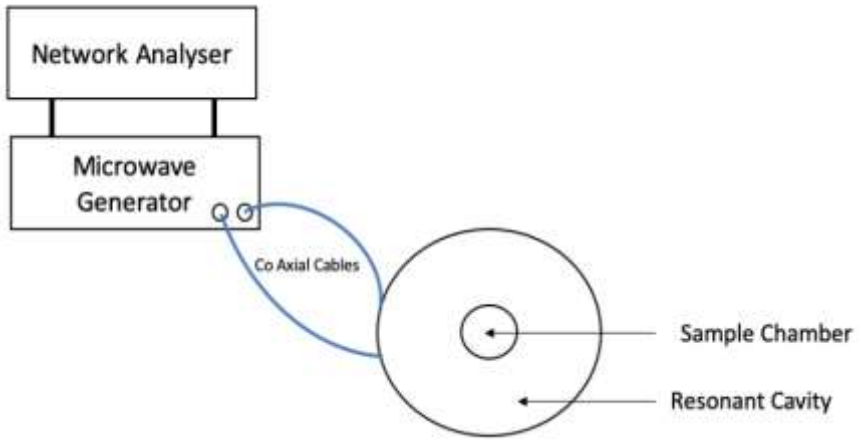


Figure 3.14 A schematic view of design for dielectric spectroscopy

The IF bandwidth and averaging factor were adjusted to 100 Hz and 4 respectively. Frequency shift (FS) as a function of the empty and the loaded sample holders was calculated as stated by Yuan et al. (2019) by using the Eq. (16):

$$Frequency\ shift, FS = \frac{f_o - f}{f_o} \qquad \text{Eq. (16)}$$

Where;

f_o = resonant frequency of empty sample holder

f = resonant frequency of sample

To analyse the dielectric measurement, the frequency shift was plotted against the moisture content. The coefficient of determination was determined. As plants are non-homogenous material with variety of compositions, moisture and other environmental conditions. Therefore, it is important that the measurement involve certain non-controllable uncertainties (Navarrete et al., 2011). The dehydrated leaves were ground and sieved to 1.0 mm mesh for uniformity before dielectric measurement.

3.7.3 Data analysis

For NIR, the relative reflectance for each sample was calculated from light intensities of the sample (I_s), the reference target (I_r) and the dark (I_d) as stated by Rady et al. (2020) by using Eq. (17):

$$Relative\ reflectance, RR = \frac{I_s - I_d}{I_r - I_d} \qquad \text{Eq. (17)}$$

The samples of LML subjected to oven-, vacuum- and heat pump drying were prepared with different levels of moisture and used for developing a model for moisture content prediction. A number of 195 samples were prepared for the calibration, and cross-validation method was used for validation of the developed partial least squares (PLS) model since the number of samples was limited. The spectrum of one sample was deleted from the validation set and was left out for cross-validation. A PLS model was built with the remaining spectra of the validation set. The left-out sample was predicted with the PLS model of the remaining set, and the procedure was repeated with leaving out each of the samples of the validation set. When the optimal model with minimum prediction error was obtained, it was used with the test data set for prediction model evaluation. The models were evaluated for its correlation coefficient (*r*) value, root mean square error (RMSE) and ratio of performance to deviation (RPD) for the validation and test model.

For NIR and DM spectroscopy, correlation coefficient (*r*) values of 0.81 – 0.9 can be used for screening and approximate calibration, values of 0.91 – 0.95 can be used for most applications including research, values of 0.96 – 0.98 are workable for most industrial applications and finally, when *r* is more than 0.99, it is suitable for any application (Williams, 2007). Another predictive ability to be

evaluated for good calibration model is the value of ratio between performance to deviation (RPD). RPD is the ratio of the standard deviation of reference data to the standard error of predicted data for population tested (Sinija and Mishra, 2011). Values of 1.5 to 2.0 refers to model capability to differentiate between high and low constituent values. Whereas values of RPD in the range of 2.0 to 2.5 means a possibility of coarse prediction of reference values. Values of RPD of 2.5 to 3.0 or higher can be used for good and excellence prediction (Nicolai et al, 2007).

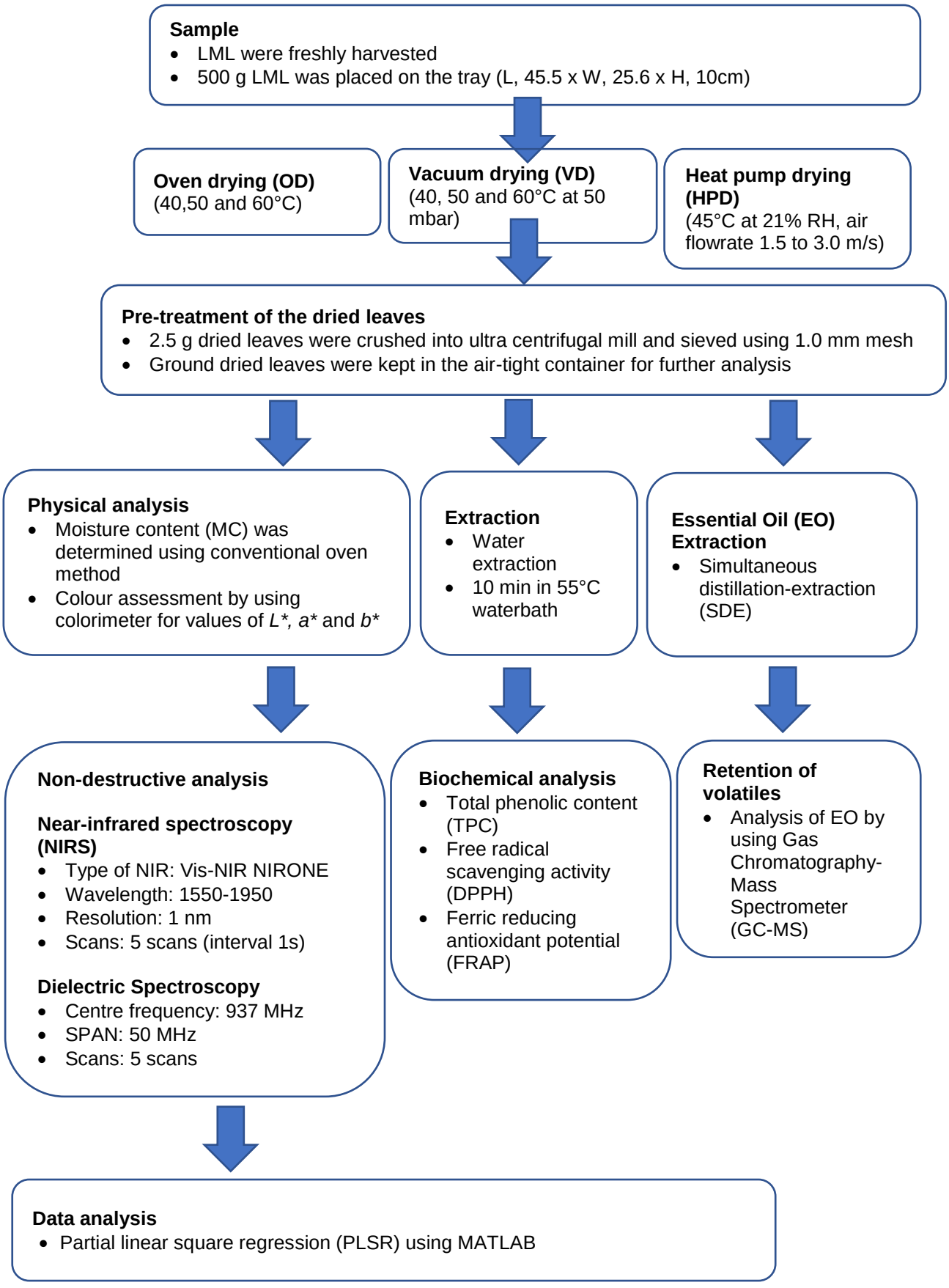
The PLSR technique depends on the applying the partial least square regression (PLSR) on the performances of validation and test models for NIR and DM. The NIR spectral data were fully analysed based on the full spectra and dielectric measurement data points and no pre-processing applied for data analysis. The number of folds that the data were divided to or k value was chosen in this study to be 4 which indicated that the data were divided into 4 parts. Hence, for all models, data were divided into a training set (75%) and a test set (25%). To increase the robustness of the deduced prediction models, a four-fold cross validation technique was applied only on the training set of data and the optimal prediction model obtained was the one that produced the minimum prediction error. This optimal model was then used with the test data set. Cross validation is a common technique used, when number of predictors is larger

than the number of objects. Thus, the parameters of the obtained models were such that the number of partial least square (PLS) components were optimized (Varmuza and Filzmoser, 2016). A number of 195 samples were prepared for the calibration, and cross-validation method was used for validation of the developed partial least square model since the number of samples is limited. PLSR was performed using MATLAB_R2019a software to calculate the value of correlation coefficient (*r*), root mean square error using cross-validation (RMSE) and ratio between performance to deviation (RPD) for both validation and test set.

The performance of the validation and test models for moisture content determination was evaluated according to three different categories. The first category involved models on full features of NIR and dielectric measurement (DM). The full variables models involved the analysis from 1550 to 1950 nm (401 data points) and 912 – 962 MHz (201 data points) for NIR and DM, respectively. The second model evaluation was on the NIR selected spectral range of analysis. There were two selected ranges involved in the PLSR which were from the range of spectra of 1600 to 1700 nm and 1650 to 1800 nm. The selection of the ranges of data analysis was determined from the plot spectral data which showed the distinguished plots of different drying methods in this study, namely oven-, vacuum- and heat pump drying for dehydration of LML. Finally, the PLSR was done

to evaluate the performance of fusion models of the NDT on both full and selected spectral range of NIR with DM. From this category, three analyses were done on full features of NIR and DM fusion (NIR+DM), selected NIR spectral range of 1600 to 1700 nm and DM fusion (NIR1600-1700+DM) and lastly the fusion of selected NIR spectral range of 1650 to 1800 nm and DM (NIR1650-1800+DM). The PLSR was determined for its regression coefficient (*r*), RMSE_cv and RPD_cv for the calibration model and *r*_test, RMSEP and RPD for the prediction model.

Figure 3.15 Flowchart of LML drying and analysis methodology



CHAPTER 4 RESULTS AND DISCUSSIONS

This chapter is divided into four main subtopics which firstly focused on the effect of the drying methods on drying performances, colour assessment, biochemical and volatile retention of lemon myrtle leaves (LML). Secondly, the effect of drying air velocities on the heat pump drying performances, quality parameters and volatile compounds retention was presented. Next, the stability study of different dried LML which focused on the effect of packaging conditions and temperature of surrounding during storage was discussed. Finally, the feasibility of non-destructive techniques like near-infrared and dielectric measurement for analysis of dried LML quality are included in this chapter.

4.1 Effect of drying on drying performance, biochemical and volatile retention of lemon myrtle leaves

Lemon myrtle leaves (LML) were dried to reduce the water content in order to reduce the risk of microbial contamination and thus prolong its shelf life. Three different drying methods were carried out for the drying of LML in order to compare the effect of the drying methods towards the drying kinetics and physicochemical content of the dried lemon myrtle leaves (LML). The methods chosen were oven drying (OD), vacuum drying (VD) and heat pump drying (HPD). The

temperature range for OD and VD used in this study were from 40 to 60°C and pressure were set to 50 mbar for VD method.

Drying time and final moisture content for each drying method are shown in Table 4.1. In general, the moisture content of LML was reduced from the initial moisture content of 63.27 ± 4.66 g H_2O/gDS to a final moisture content of around 5.38 to 10.36 ± 0.94 gH_2O/gDS. VD40 gave the longest drying time which was recorded as 76 h while higher temperature drying condition such as OD60 shows significant reduction in total drying time which was recorded as 12 h. It is expected that the higher temperature of drying air would result in shorter drying time with lower final moisture content as compared to other drying methods where its operating temperature is relatively lower. Among all drying methods tested in this study, OD60 gave the lowest final moisture content while OD40 and HPD resulted in the highest final moisture content of 10.36 ± 0.94 and 10.36 ± 0.61 % w.b, respectively. For temperature dependent drying method namely OD and VD, different operating temperature may thus result in different final moisture content and drying time.

Table 4.1: Drying time and final moisture content of dried LML

Drying method (condition)	Drying time (h)	Final moisture content (w.b) (%)
Fresh	-	63.27 ± 4.66[a]
OD40	52	10.36 ± 0.94[b]
OD50	32	8.57 ± 0.98[b]
OD60	12	5.38 ± 0.01[b]
VD40	76	8.42 ± 0.62[b]
VD50	52	7.39 ± 0.52[b]
VD60	34	5.62 ± 0.35[b]
HPD	30	10.36 ± 0.61[b]

Mean values ± standard deviation (n=3 replications) within the same column with the same letter are not significantly different (p>0.05)

Drying carried out at higher temperature tend to promote the vaporisation of surface moisture of the drying material. As the drying temperature increases, the drying rate also increases and thus reducing the drying time. At higher temperature, the drying rate is enhanced due to the fact that higher drying forces for heat transfer because of the higher drying temperature. Doymaz et al. (2011) reported that the drying time that required to reduce the moisture content in grape leaves was dependent on the drying condition, as the shortest time of drying was obtained from the highest drying temperature (60°C) whereas the lowest temperature of drying (40°C) resulted in longer time of drying. The same observation was also reported by Alibas (2007) that during Nettle leaves drying, as the temperature of vacuum drying increases, the total drying time decreases.

It can be seen that the increase in drying temperature increases the drying rate. As the drying temperature increases, the difference of temperature between drying air and material resulted in higher gradient of temperature. The temperature gradient is proportional to the drying temperature. As the drying temperature increases, the temperature gradient also increases and thus facilitate the higher degree of heat transfer and the material absorbs more heat. A part of the heat increases the temperature of the material, whereas a part of the heat enhances the vaporisation of the water at the surface of the sample. During internal moisture vaporisation, the water vapour migrates through the pores within the sold matrix. Faster moisture reduction contributes to the higher drying rate. Hence this explains the influence of higher drying temperature to higher drying rates in the OD and VD of LML.

Although HPD operated at lower temperature (approximately 45°C), the reduction of the moisture content was comparable to OD50. This is due to low relative humidity (RH) of drying air in the HPD though the drying air temperature was lower (Phoungchandang et al., 2009). Heat pump drying process has advantage on drying time (shortest) with the final moisture content resulted in acceptance range of dried samples. In this case, this advantage will be beneficial in terms of energy consumption as the drying time is relatively short.

Relative humidity is the amount of moisture in the air compared to what the air can hold at that temperature. Low relative humidity of drying air in a heat pump dryer makes it easier for the moisture to evaporate and thus shorter the drying time. Rayaguru and Routray (2010) reported that due to low RH of drying air in a heat pump dryer, the moisture reduced 150% d.b though the temperature was lower. The low relative humidity in drying air gave a higher evaporation rate of moisture from the material and consequently faster reduction in the moisture content and hence the total drying time was reduced in drying of *Pandanus* leaves.

For HPD drying, although it operates at lower temperature, the drying rate is still relatively faster than hot air drying at higher temperature. The key parameter in HPD operation is the low RH. As the HPD took place in low RH, it creates a big moisture gradient between the drying medium and the surface moisture. This contributes to the higher degree of moisture transfer from material surface to the drying air. The final moisture content is at a higher level because the equilibrium moisture content (EMC) is a function of temperature. Since the operating temperature in HPD is low, thus the final EMC of 10.36 $\pm$ 0.61 % w.b is relatively higher.

4.1.1 Drying performances

4.1.1.1 Drying kinetics and drying rate

Figure 4.1 shows the effect of drying methods on the drying time and moisture content of dried LML. From the plot of moisture content (% w.b) against drying time, it is clearly shown that drying time decreases with increase in drying temperature for both oven and vacuum drying method. The steepest slope of drying slope is given by OD60 followed by OD50, HPD, VD60, VD50, OD40 and finally VD40. For both oven drying and vacuum drying, higher temperature resulted in higher drying rate, hence shortened the drying time.

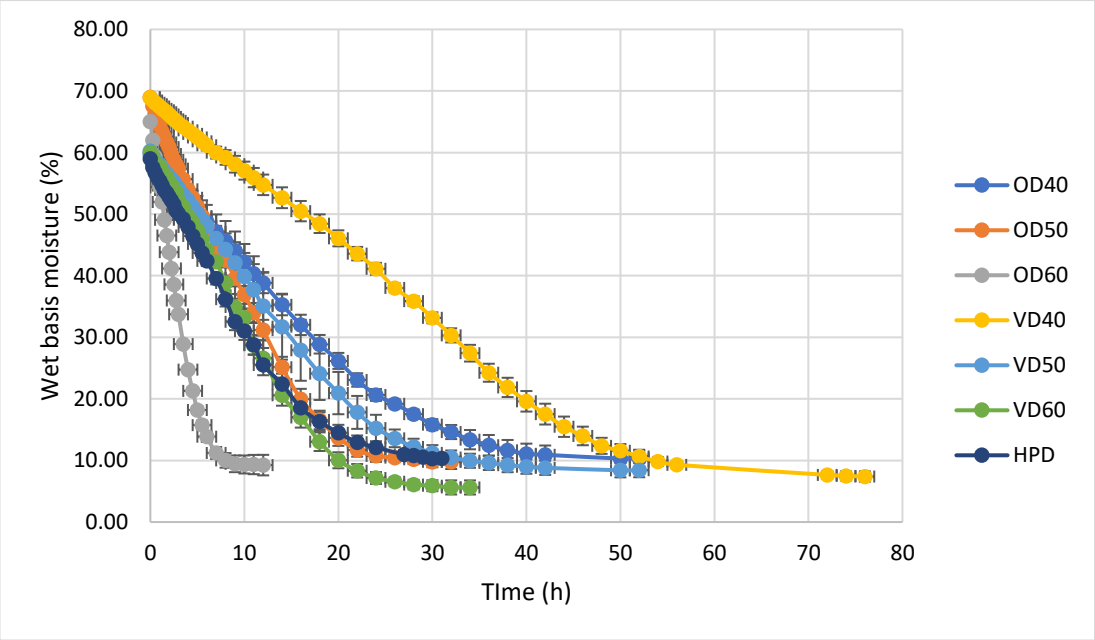


Figure 4.1 Impact of drying methods on drying time and moisture content of dried lemon myrtle leaves.

The moisture reduction profile of various drying methods is shown in Figure 4.2. From Figure 4.2, the reduction of moisture until equilibrium moisture achieved in the sample was dependent on the method and condition of drying. All the OD conditions gave shorter time of drying, whereas all VD conditions gave longer time of drying. Higher operating temperature (60°C) also gave shorter total drying time for OD and VD. This could be due to the convection of air that was in contact with the LML that created higher temperature gradient and thus facilitated faster removal of moisture in OD especially at higher temperature of operation. Pal et al. (2008) also reported the same trend for the drying of green sweet pepper, that as temperature increased, the drying curve exhibited steeper slope thus exhibiting an increase in drying rate.

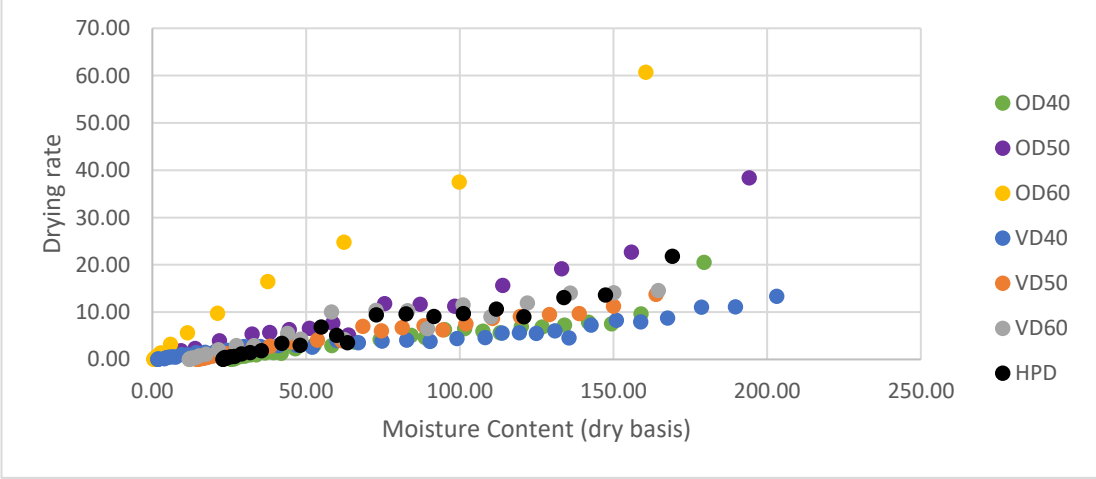


Figure 4.2: Drying rate with respective moisture content for each drying method

Figure 4.2 shows the drying rate with respect to moisture content for each drying method. The highest rate was obtained from OD60 followed by OD50, HPD, VD60, VD50, OD40 and finally VD40. Drying of LML took place mainly under falling-rate period. During this period, the migration of moisture occurred through the mechanism of diffusion (Rayaguru and Routray, 2010). At the beginning of the OD and HPD, the drying rate was very high and the drying rate continued to decrease as the moisture content approached equilibrium. All the vacuum drying conditions gave lower drying rate that effected the total drying time especially for VD40. The difference in drying rate resulted from vacuum drying might be due to the absence of convection that leads to significantly slower heat transfer to solid phase. Therefore, a longer time required for the moisture of the product to achieve equilibrium moisture content in VD. The temperature in vacuum drying also has an important effect on the drying time. Increase in temperature in vacuum drying resulted in reduction of drying time and thus increase the drying rate. It can be seen that the drying rate of VD40 was the lowest in comparison to other drying methods and conditions.

The peak drying rate for LML was found to be 60.72 g water/g solid.h for OD60 while the lowest rate was VD40 with only 13.35 g water/ g solid.h. Drying in oven reduced the moisture content to range of 37 to 142 % d.b, while vacuum drying at 40 to 60°C only reduced the

moisture content up to 122 % d.b. This was reported by Babu et al. (2018) that the heat transfer to solid phase in VD is slowed down due to the absence of convection.

From Figure 4.2, as the drying temperature increases, the removal of moisture also increases due to more heat is provided to the leaves and thus increases the drying rate. Although the HPD was operated at lower temperature (approximately 45°C), the reduction of the moisture content was comparable to OD50. This is due to low RH of drying air in the HPD albeit the drying air temperature was lower (Rayaguru and Routray, 2010). At low RH, the mass transfer is high resulting in higher loss of water. This is due to the higher partial pressure of water between the air and sample. Thus, moisture evaporation rate increases as the RH reduced. Taheri-Garavand and Meda (2018) also reported that as for the effect of relative humidity in drying air to the drying rate and drying time, as the relative humidity decreases, the drying rate increases and change accordingly to the drying time on savory leaves drying

It can be seen from Figure 4.2 that the drying rate varies with drying method and conditions. The drying rate for each drying condition is influenced by the drying temperature. For oven and vacuum drying, OD, drying at high temperature (60°C) tends to give higher drying rate while at low drying temperature (40°C) tends to give lower

drying rate. The drying air temperature influences the rate of drying for OD and VD. As the temperature increase, it influences the heat transfer and makes it faster, thus results in rapid evaporation of moisture from the sample. As the sample is dried, the evaporation occurs from inside of the solid and vapour reach the surface by molecular diffusion through the material. The diffusion rate of moisture from the sample also increases and resulting in shorter drying time. As for the higher drying rate for high drying temperature, it is important to observe the changes in product quality as it may deteriorate by high temperature exposure during dehydration process that will affect the functionality i.e the active compound of the product.

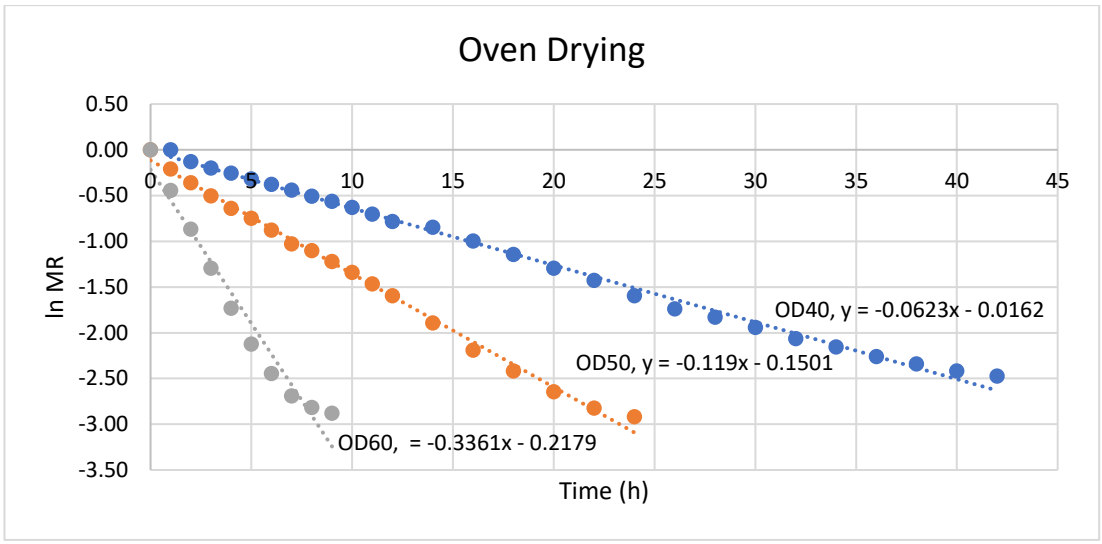
4.1.1.2 Effective moisture diffusivity

During the drying process of agricultural food products, moisture diffusivity is the most crucial transport property for the study of moisture transfer inside the product. The knowledge of the effective diffusivity of a material is necessary not only to describe the drying kinetics and interpret experimental observations but also for simulation of the drying process.

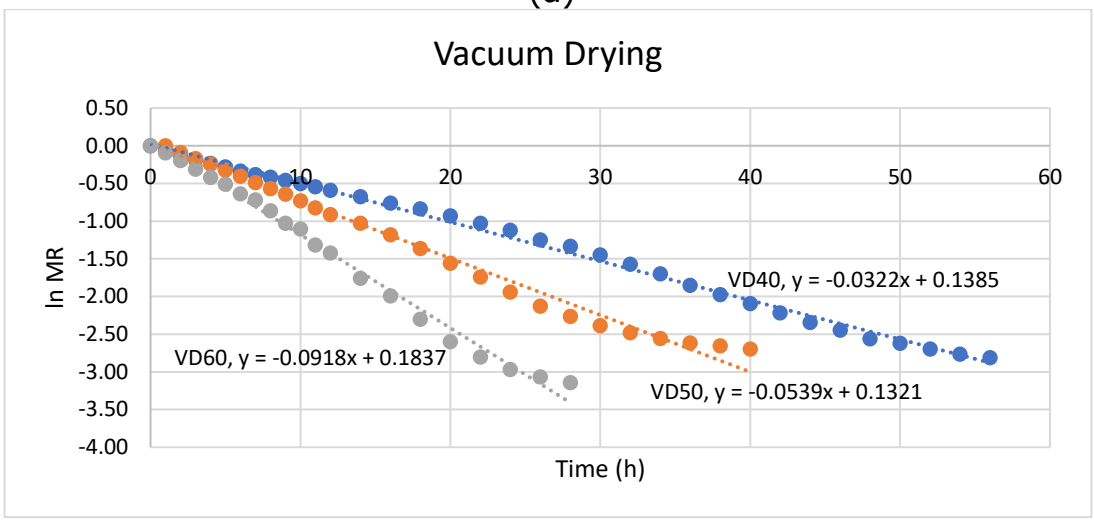
The effective moisture diffusivity (D_{eff}) values of the as the dehydration process were long with moisture ratio, MR<0.6 (Altay et al., 2019).

Figure 4.3 shows the plot of ln (MR) against time of lemon myrtle leaves for different drying methods and conditions. According to Figure 4.3, the slope of the line, *k*-value, ranged from 0.0623 to 0.3361 and 0.0322 to 0.0918 for OD and VD as the temperature of drying increment from 40 to 60°C. The *k*-value for HPD resulted as 0.0888. From Figure 4.3, it can be seen that the k-value decrease proportionally to the temperature of drying. As the drying temperature increase, the *k*-value increased and thus resulted higher D_{eff} values. As comparison of different drying method, OD ranged in higher *k*-value than VD method for the same variation of drying temperature from 40 to 60°C. For instance, the *k*-values for OD60 was 0.3361 and only 0.0918 for VD. This variation is influenced by the mechanism of dehydration process. OD involve in drying using air as medium, thus the available drying air, increase the moisture diffusion from the internal solid to the surface of the product. As the moisture accumulated at the surface of theproduct, the saturated moisture creates the moisture gradient that facilitate the mass transfer and thus increase the drying rate of the product. On the other hand, VD involve drying in absence of convection, took longer time for the moisture transfer from the internal to the surface

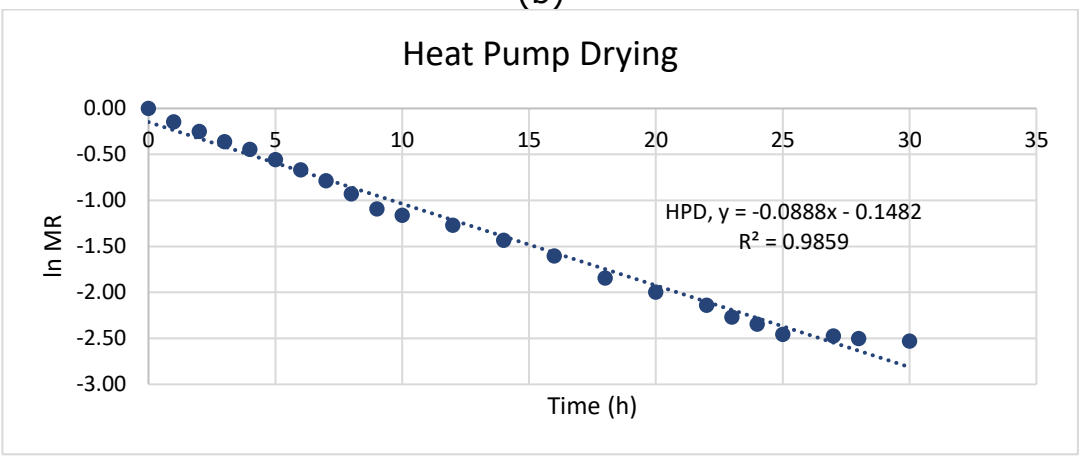
of the solid. At the same drying temperature, OD took a less drying time and bigger D_{eff} then VD. Another possible reason may be that compared with OD, the vacuum in VD reduce the heat capacity of the air at the same drying temperature, causing a decreased heat transfer rate from air to material. Then drying rate is reduced and more drying time is needed. Thus, the D_{eff} is lower in VD and explains the lower rate of drying with longer time for the moisture to achieve equilibrium.



(a)



(b)



(c)

Figure 4.3 Variation of ln (*MR*) with drying time at different air temperature (*T*) and method.

The values of D_{eff} values of all samples are shown in Table 4.2. As shown in Table 4.2, the coefficient correlation, R^2 for different drying methods and conditions are in the range of 0.9645 to 0.9955. This means that the plots are closely distributed to the regression line as indication of a good correlation of ln *MR* with time for all drying conditions. The calculated moisture diffusivity, D_{eff} of LML were found to be as 8.07 x 10^{-10} to 4.35 x 10^{-9} m^2s^{-1} for OD, while 4.167 x 10^{-10} to 1.188 x 10^{-9} for VD and 1.31 x 10^{-9} for HPD. The values of D_{eff} obtained from this study also are within the range of 10^{-12} to 10^{-8} m^2/s for drying of food materials (Zogzas et al., 1996). Therefore, the calculated D_{eff} for LML in the present study is within the range reported by the authors mentioned previously.

Table 4.2: Effective moisture diffusivity, D_{eff} and coefficient of determination, R^2 for different drying methods and conditions

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

Effective moisture diffusivity, D_{eff} values increases with increment in drying air temperature. As the temperature increases, the level of vibration in water molecules increases and thus reducing its viscosity that leads to alterations in water diffusion through the capillaries of agricultural products. Meanwhile, during the drying process, the increment in temperature of drying also increases the intensity of water vibration that results in a faster diffusion of water.

The D_{eff} value for HPD was ranked as the fourth highest. The D_{eff} value for HPD took place after OD60, OD50 and VD60. D_{eff} value for HPD was not the lowest and this might due to the low RH while the dehydration took place in HPD. Taheri-Garavand and Meda (2018) mentioned that at higher RH, the D_{eff} has weaker linear relation with ln *MR* which mean lower R^2 values. This might be explained by a more saturated medium of drying air at higher RH and hence the moisture was not be able to transfer out easily. This result is in agreement with findings by Aktas et al. (2017) where the lower RH in HPD for mint drying resulted in increase in moisture diffusivity compared to solar drying. Thus, it can be said that the mechanism of drying, temperature of drying air and RH have certain impact on the D_{eff}.

Larger effective diffusivity was obtained in HPD, this is because HPD operates at low RH, it increases the moisture migration rate from

internal of the sample to the surface of the material and facilitate the moisture removal from the sample. The decrease in the air humidity also increases mass transfer which in turn creates a high partial pressure of water and the sample, thus the tremendous loss of water. This causes the higher moisture evaporation rate and increases the drying rate. The higher D_{eff} also corresponded to the rapid decline of the moisture content of the product. As at higher drying temperature, it expedited the water molecules and resulted in rapid moisture loss compared to drying at lower temperature.

The effective moisture diffusivity increases with increasing of air temperature for OD and VD, which is in accordance with the report published in Akpinar and Toraman (2013) and Komolate (2018) that the major effect of temperature in which the moisture diffusivity increased with increase of air temperature found out in ginger slice and fish drying. As reported by Goneli et al. (2014) on drying of *Schinus terebinthifolius* leaves for temperature range of 40 to 70°C, the D_{eff} values varied from 0.1476×10^{-11} to 1.5811×10^{-11} m^2/s. Martins et al. (2015) reported that the D_{eff} for *Serjania marginata* leaves was within 0.6630×10^{-11} to 12.0712×10^{-11} m^2/s for the same temperature range. With reference to both cases, the range of D_{eff} is higher than the D_{eff} LML investigated in present study. This may be due to the morphological characteristics of the leaves and the leaves' thickness as well. The average LML thickness is 0.3574

mm whereas the thickness of *Schinus terebinthifolius* and *Serjania marginata* leaves are 0.3631 mm and 0.5922 mm, respectively. As mentioned by Silva et al. (2017), the leaf thickness is a factor to be considered on the analysis of D_{eff} of leaves during drying. Quequeto et al. (2019) reported that the average thickness of *Piper aduncum* L. leaves is 0.1687 mm because the thickness is smaller contributing to higher D_{eff}.

The values of D_{eff} obtained from this study also are within the range of 10^{-12} to 10^{-8} m^2/s for drying of food materials (Zogzas et al., 1996). The value of D_{eff} for LML drying via OD, VD and HPD are slightly higher than D_{eff} reported by oven drying of rosemary leaves for 2.55 x 10^{-11} to 1.51 x 10^{-10} (Mghazli et al., 2017), mint leaves for 0.91 x 10^{-11} to 10.41 x 10^{-11} (Motevali et al., 2016), forced convective drying on savory leaves for 6.76 x 10^{-12} to 1.57 x 10^{-11} (Taheri-Garavand and Meda, 2018), tray dryer on lemongrass for 0.97 to 1.33 x 10^{-10}, HPD on *C.asiatica* leaves for 3.91 x10^{-11} to 1.82 x 10^{-10} (Trirattanapikul and Phoungchandang, 2012).

The temperature is the key parameter that significantly influences the effective moisture diffusivity. An increase in the effective diffusivity is an indicator of lower resistance to mass transfer in the material during drying (Mujaffar and John, 2018). Increasing the temperature of drying gave rise in effective moisture diffusivity

owing to the accelerated mass transfer. Further, the higher the temperature of drying, the thermal energy is increased and thus increase the activity of water molecules that lead to higher moisture diffusivity. In addition, an increase in temperature decreases the resistance of the fluid to flow and hence lead to the diffusion of water molecules in the product capillaries and the moisture diffusivity value increase (Torki-Harchegani et al., 2016).

4.1.1.3 Activation Energy

The activation energy was calculated by plotting ln D_{eff} versus the temperature of, $1/T$ (K^{-1}) and is shown in Figure 4.4. The straight line from the plot ln D_{eff} versus $1/T$ (K^{-1}) indicates that the drying data fits very well the Arrhenius dependence, and the slope of the straight line is activation energy, E_a/R (8.314 $JK^{-1}mol^{-1}$).

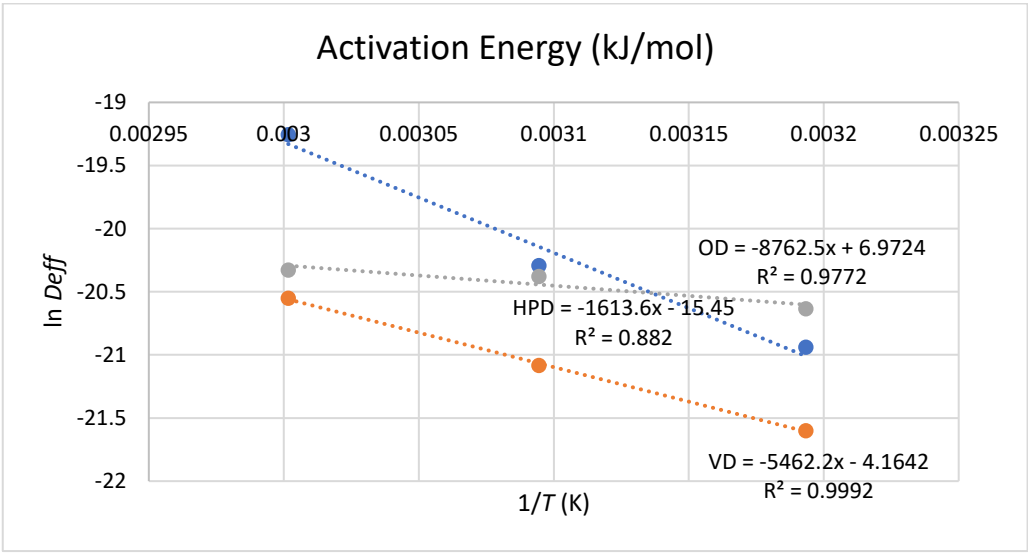


Figure 4.4 Ln effective moisture diffusivity, D_{eff} versus 1/absolute temperature, $T(K^{-1})$

Table 4.3 shows the activation energy of lemon myrtle drying subjected to oven-, vacuum- and heat pump drying. As shown in Table 4.3, the lowest value of E_a was calculated from HPD followed by VD and OD. The activation energy, E_a were found to be 72.85 and 45.41 for OD and VD, respectively, while only 13.42 kJ/mol for HPD. The E_a for all types of dryer fall between the range of 12.7 to 110 kJ/mol reported on various food products for all type drying methods (Xiao et al., 2011; Troncoso and Pedreschi, 2007).

From Figure 4.4, the straight line indicates the effect of temperature range on the moisture diffusivity in expression of Arrhenius dependence. The steepest slope is shown by OD that indicates the highest E_a value compared to other drying methods. The reduction of the ln D_{eff} was also bigger for each of the drying temperature. For HPD method, the gradient of the slope was the smallest, and the reduction of the ln D_{eff} was only minimal within the same range of drying temperature. From this result, it can be seen that the reduction ln D_{eff} not so depending on the reciprocal temperature. Thus, the gradient or the activation energy is dependent on the reduction of ln D_{eff} for each drying temperature investigated.

Table 4.3 Activation energy of lemon myrtle drying subjected to OD, VD and HPD

Sample	Oven Drying (OD)	Vacuum Drying (VD)	Heat Pump Drying (HPD)
Activation Energy, E_a (kJ/mol)	72.85	45.41	13.42

The dependency of E_a value on moisture diffusivity is presented by the plot of ln D_{eff} versus 1/T(K). The higher the slope, the higher the E_a value of the drying process. Results indicate that the E_a values for LML vary between 13.42 and 72.85 kJ/mol for HPD, OD and VD. In agricultural materials, there are two forms of moisture, namely free and bound moisture. Most of water in LML is in the form of bound moisture. As mentioned earlier in 4.1.1, OD resulted in the highest drying rate compared to other the two drying methods. Therefore, the E_a of OD is also highest. Higher E_a indicates that more energy is required and therefore OD needs to be carried out at higher temperature compared to the other two drying methods. Compared with OD, the VD and HPD have a lower E_a. A possible explanation was that compared with OD, VD decreases the water evaporation temperature due to vacuum; HPD decreases the air relative humidity. Both the factors help to facilitate the water evaporation and lower E_a is expected. As a result, high drying temperature may affect physical and chemical properties of the sample. Due to the

structure of leaves, drying rate of LML occurred in one falling rate period.

The effectiveness of drying process varies according to its operation. Some of the dryers are effectively operated at low temperature, while other are good as high temperature dryer. For example, convective dryer involves in higher operating temperature whereas freeze drying works effectively at lower operating temperature. In this study, it is aimed to dehydrate the LML at lower temperature in a way to preserve the volatile and biochemical content of the dried LML. Dryer that operates below than 60°C is considered as lower temperature operating dryer. E_a value is used to represent the effectiveness of a particular drying process related to its operating temperature.

The lower E_a value for a particular drying process is related to the effectiveness of the drying operation at lower temperature. As shown in Table 4.3, the lowest value of E_a was calculated from HPD followed by VD and OD. Drying LML in HPD required low E_a value indicates that the method could be conducted at lower temperature effectively. For VD, the value of E_a showed that the operation required medium temperature. On the other hand, OD gave the highest E_a value, that shows good representation of oven to operate at relatively high temperature for effective drying operation.

The findings in Table 4.3 is in an agreement with a report by Elhussein and Sahin (2018) where the activation energy for a dryer depended on the drying method along with the temperature as oven drying had more activation energy whilst vacuum drying on olive leaves yielded less value. The value of E_a in HPD also was lower than others reports on mint leaves, 84 kJ/mol (Ardestani et al., 2016) and *Orthosiphon aritatus* leaves, 32.4619 kJ/mol (Klungboonkrong et al., 2018). With the same range of operating temperature (40 to 60°C) in HPD, the E_a values for mint leaves and *O. aritatus* leaves were higher than E_a for LML drying. This might be due to the initial moisture content of the sample. The initial moisture content of the mint leaves and *O. aritatus* leaves were 88.5 and 87.19%, respectively, while the range of moisture content for fresh LML is in range of 60 to 70%. The higher the initial moisture content, the higher the activation energy is required in the drying process. As relation to high initial moisture content in mint leaves and *O.aritatus*, more energy required to facilitate the moisture diffusion until the moisture content of the samples achieved equilibrium. This concluded that with lower initial moisture content of LML responsible for lower activation energy required in HPD for LML drying.

4.1.2 Colour changes

Colour is an important quality that needs to be maintained, especially for their commercial acceptability. The changes in colour might result in quality deterioration including its functionality. The *L** value shows the lightness (positive values) and darkness (negative values) of the sample while *a** and *b** indicate the greenness and yellowness of a sample, respectively.

Table 4.4 shows the values of colour parameters of *L**, *a** and *b** of fresh and dehydrated LML for each drying condition. The *L** value is useful to reveal the brownness and darkness of leaves after drying (Arabhosseini et al., 2007). As shown in Table 4.4, drying resulted in the *L** values in range of 38.43 to 45.51 and 46.38 as *L** value for fresh LML. The higher *L** value indicates the lighter the colour of the sample, while lower *L** value represents the darker colour of the sample. The highest *L** value was obtained in HPD sample. *L** value from OD60 was the lowest as from the observation of visual colour degradation, the final color of the sample was green-brownish colour. OD60 gave the lowest *L** value might be due to the higher temperature of operation that tends to oxidise chlorophyll in the leaves that resulted in change of colour from bright green to darker green. This result is in line with the finding by Argyropoulos et al. (2009) who reported that remarkable loss of lightness (*L**) was

detected as the drying temperature increased to over 50°C in lemon balm (*Melissa officinalis* L.) drying process.

Table 4.4: Values of *L, *a** and *b** obtained from fresh and dehydrated LML after each drying condition**

Sample		*L**	*a**	*b**
Fresh		46.38 ± 0.08[a]	-5.87 ± 0.11[a]	28.98 ± 0.15[a]
	40	42.88 ± 1.04[b]	-2.9 ± 0.04[b]	26.43 ± 0.13[b]
OD	50	41.95 ± 0.79[b]	-2.58 ± 0.1[b]	26.32 ± 0.31[b]
	60	38.43 ± 0.91[b]	0.48 ± 0.34[b]	26.07 ± 0.54[b]
	40	41.6 ± 0.25[b]	-2.49 ± 0.66[b]	23.49 ± 0.57[b]
VD	50	43.18 ± 0.07[b]	-3.37 ± 0.03[b]	27.45 ± 0.38[b]
	60	42.2 ± 0.81[b]	-2.97 ± 0.1[b]	27.65 ± 0.52[b]
HPD	45	45.51 ± 0.41[b]	-4.00 ± 0.15[b]	27.94 ± 0.37[b]

Mean values ± standard deviation (n = 3 replications) within the same column with the same letter are not significantly different (p>0.05)

In comparison between the drying methods, VD40 also resulted in significant change of *L** value. VD samples gave low value of *L** as indication of darker sample. This is because VD tends to take longer time for drying to be completed. Therefore, longer exposure to drying air results in significant colour change in LML and causes VD40 sample's colour to be darker.

The *a** value represents the greenness of the sample, which in range of negative value (greenness) to positive value (redness). Lower *a** value (more negative) indicates the greener the samples' colour

while higher *a** value (more positive) indicates the samples' colour is closer to redness. From Table 4.4, the *a** value increased with drying temperature. According to Table 4.4, the highest *a** value was obtained from OD60 sample. This result might be due to the chlorophyll degradation by oxidation at higher drying temperature and thus change the sample's colour from bright green (more negative *a** value) to darker green-brownish (more positive *a** value). Oliveira et al. (2015) reported that the increase of air drying temperature reduced the brightness, the intensity of green colour and the yellow intensification of *galage kale*. From Table 4.4, the greenest sample with the lowest *a** value after drying was obtained from HPD sample. This might be due to better preservation of cell structure in HPD samples that lead to less chlorophyll degradation (Buchaillot et al., 2009) and thus resulted in only slight change of *a** value in comparison to the *a** value of fresh LML.

The yellowness or blueness of a sample is indicated by its *b** value. The higher the b* value represents the yellowness while lower *b** value represents the blueness of the sample's colour. Table 4.4 shows the value of *b** for fresh, OD and VD from temperature of 40 to 60°C and HPD samples. The *b** value for dehydrated samples were in range of 23.49 to 27.94. The lowest *b** value was obtained from VD40 samples, while HPD samples gave the highest *b** value. The higher *b** value indicates the yellowish colour that leads to brighter

colour sample. This result is correlated with *L** values as HPD resulted in highest *L** value as indication of the lightest dried sample's colour. The lower *b** value for VD40 was likely caused by the longer drying time, as it reduced the *b** value and thus resulted in darker coloured sample.

The changes of colour parameters of *L**, *a** and *b** were influenced by the drying methods and conditions. Higher temperature and time of drying resulted in significant changes in colour as higher *a**, lower *L** and *b** value. This change in appearance (colour) is caused by the degradation of chlorophyll. As drying occurs at higher temperature, the chlorophyll degrades and causes oxidation. Oxidation of chloropyll causes the loss of two magnesium atoms, which is replaced by two hydrogen atoms. The replacement contributes to the changes of colour of LML after drying from bright (low *a** value, high *L** and *b** values) to darker (high *a** value, low *L** and *b** values) colour of the dried samples. Drying time also affects the changes, as longer exposure to drying air causes remarkable colour changes to the dried LML. Therefore, as consideration to shorter drying time and lower operating temperature, HPD is a suitable drying method in minimising colour changes especially the change of greenness (*a**) value of the dried LML.

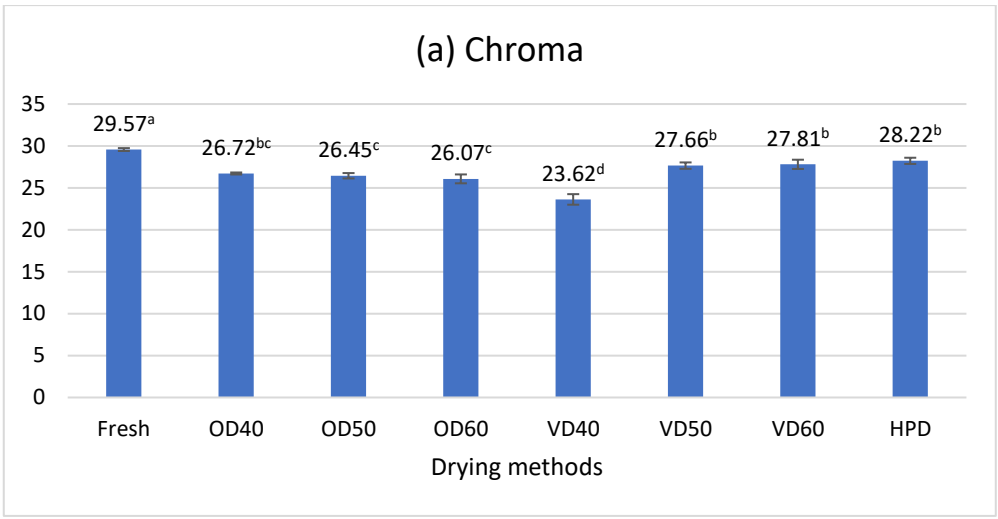
The colour saturation or chroma parameter permits the determination of the strength of response to the hue of a colour in a qualitative manner, through interpretation of its intensity and depth (Rubinskiené, 2006). Figure 4.5(a) depicts the chroma of fresh and dried LML for HPD and different drying conditions of OD and VD. The values of chroma were calculated by using Eq. (4) which is 29.57 for fresh leaves and dehydrated leaves are in the range of 23.62 to 28.22. The chroma values were affected by different drying methods and conditions. Higher chroma value indicates the more intense (pure) colour, while lower chroma value indicates the less intense of the samples' colour. According to Figure 4.5(a), the highest chroma value is shown by HPD and lowest chroma value is exhibited by VD40 samples. Generally, the values of chroma decrease with an increase in drying temperature and drying time. VD40 has the longest drying time resulted in the lowest chroma value among all drying conditions. The prolonged exposure to drying air caused the colour changes especially in value of a^* and b^* that lead to changes in colour intensity or chroma of the dried samples.

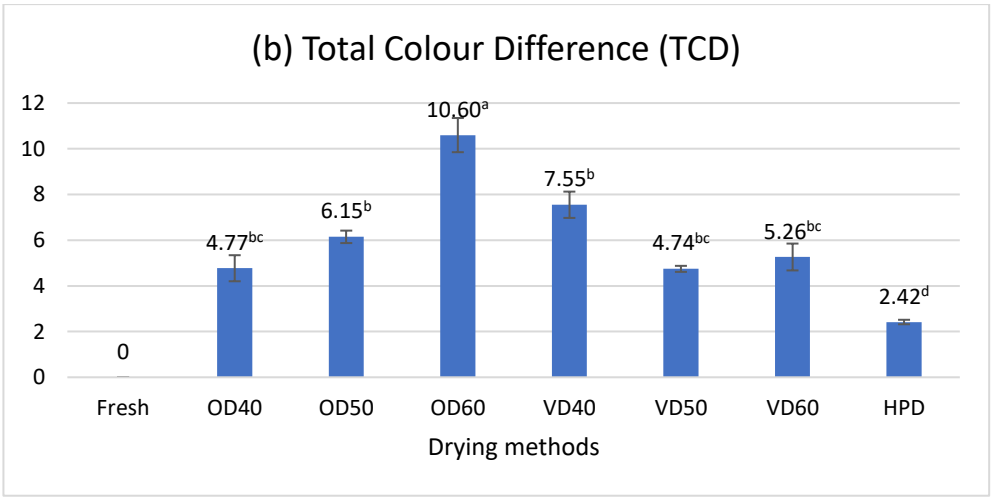
Total colour difference (TCD) is also an important parameter in measuring colour. Figure 4.5(b) shows the TCD value of dehydrated LML subjected to different drying conditions, with reference to the fresh colour parameters. The lower the TCD value indicates the minimal changes, while higher TCD value indicates more changes

the LML experience during drying process. The TCD values were in the order: OD60 > VD40 > OD50 > VD60 > OD40 > VD50 > HPD. From Figure 4.5(b), OD60 shows the highest TCD, whereas HPD shows the lowest TCD value. The highest TCD measured from OD60 sample might be attributed to higher increment in *a** value (less green) and reduction of *L** value (darker colour) compared to fresh sample. The significant changes of these parameters were influenced by the higher temperature of drying that leads to chlorophyll degradation that changed the bright-green colour to green-brownish colour of dried sample. Although the time of drying for HPD is not the shortest, HPD resulted in the lowest TCD value. This finding is supported by the highest *a** value closest to the fresh leaves that represents slight or minimal changes leaves experienced during dehydration process by using HPD.

Browning index (BI) is an important parameter to define browning in the sample. BI represents the purity of brown colour. Figure 4.5(c) shows the value of BI for fresh, HPD and different drying temperature for OD and VD samples. As shown in Figure 4.5(c), BI value for OD and VD LML was affected by the drying temperature. The BI values increased as the drying temperature increased. BI value for HPD was slightly higher than fresh sample indicating that HPD resulted in slight colour change of LML. The highest value of BI for each OD and VD was observed in OD60 and VD60, respectively.

The visual colour degradation (from green to green-brownish) for OD60 was obvious during the drying process, followed by VD60. OD60 resulted in the highest BI value which indicated that higher purity of brown colour in the sample. This result is supported by the significant change in *a** value that changes from -5.87 to 0.48, positive value that represents the closer the colour to redness. These results might be due to change in *L**, *a** and *b** values that thus, affected the BI value. The significant change in *a** value from OD60 samples indicates that the sample experienced significant changes at higher drying temperature.





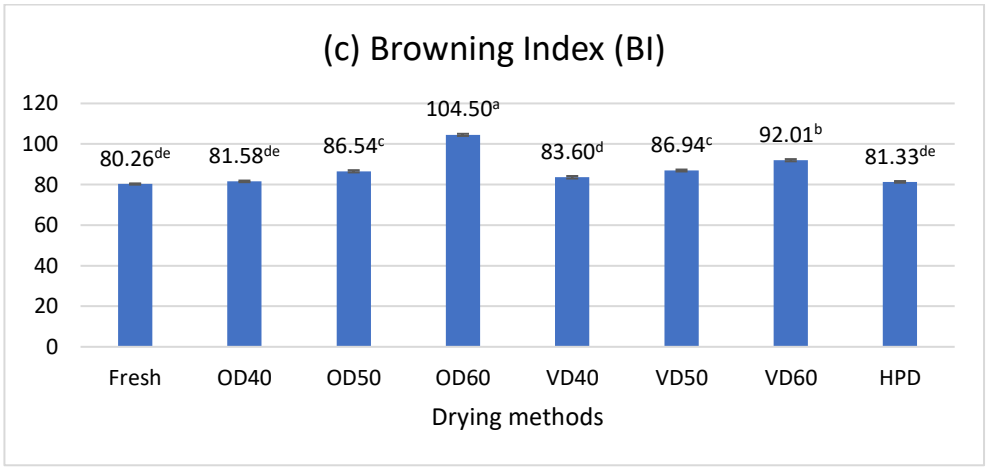


Figure 4.5 Colour properties for fresh and dehydrated LML after each drying condition. Vertical bars indicate standard deviation and values marked by the same letter are not significantly different (p>0.05).

Poomsa-ad et al. (2011) reported that the discolouration of products can be affected by several factors including drying temperature, drying air velocity that lead to pigment degradation during drying process and browning reaction occurring, resulted in colour change especially under air drying. This report was also in line with a study by Buchaillot et al. (2009) where the higher the drying temperature, the greater the colour degradation of sample which led to chlorophyll loss and thus less green colour preservation. The variation of the colour assessment on chroma, TCD and BI values are influenced by the drying methods and conditions. In order to maximise the colour retention during drying (high chroma, low TCD and BI), it is recommended to use HPD as dehydrator for LML. With considerable drying time and lower operating temperature, HPD was proven to reduce the moisture content of LML leaves with better colour preservation of the dried LML.

4.1.3. Biochemical analysis

Total phenolic content (TPC) for fresh, heat pump dried and different conditions of oven- and vacuum drying of lemon myrtle leaves are shown in Figure 4.6(a). TPC was evaluated in both fresh and dried leaves in order to assess the losses which permits the comparison of the effect of the different drying methods and conditions on TPC retention of dried LML. According to Figure 4.6 (a), the TPC content for all drying methods and conditions are in the range of 43.71 to

63.97 mg GAE/g sample and the TPC for fresh LML is 74.18 mg GAE/g sample. The highest TPC value was obtained from VD60, while the lowest TPC value was obtained from OD60. Lower TPC value indicates the greater loss of after LML was subjected to OD.

For OD, an increase in drying temperature resulted in higher reduction of TPC value. This explains the exposure of sample to high temperature caused the degradation of phenolic content. However, the TPC reduction of LML after VD did not follow the same trend as OD. This might be attributed by the duration and temperature of drying. Longer drying time (52 h) and higher drying temperature (50^{o}C) resulted in higher reduction of TPC in VD50. Whereas, as the temperature increased, drying time was decreased and hence resulted in lower TPC reduction in VD60.

Figure 4.6 (a) shows the influence of drying conditions on TPC reduction. The loss of phenolics compounds during drying might be due to the temperature and the duration of drying. During drying, exposure to high temperature may cause disruption of cells and results in the release of oxidative and hydrolytic enzymes that were capable of oxidising phenolic compounds. Moreover, high drying temperature inactivates enzymes that are responsible for polyphenols oxidation such as polyphenoloxidases and peroxidases that are present in plant materials (Lim and Murtijaya, 2007). The

decrease in TPC during drying can also be attributed to the alteration in the chemical structure of polyphenols caused by the binding of polyphenols with other compounds and thus make it unavailable for extraction (Mrad et al., 2012). This result was also reported by Wu et al., (2013) where the total phenolic content of *Angelica sinensis* leaves was significantly affected by the drying temperature in oven. Ben Haj Said et al. (2013) also reported the same findings on the significant loss of TPC from 60 to 69% as a result of increasing drying temperature in *Allium roseum* leaves. Similarly, the reduction of TPC can also be contributed by the longer duration of drying. The prolonged exposure to drying air may cause the degradation of phenolic content and thus higher reduction of TPC in dried leaves.

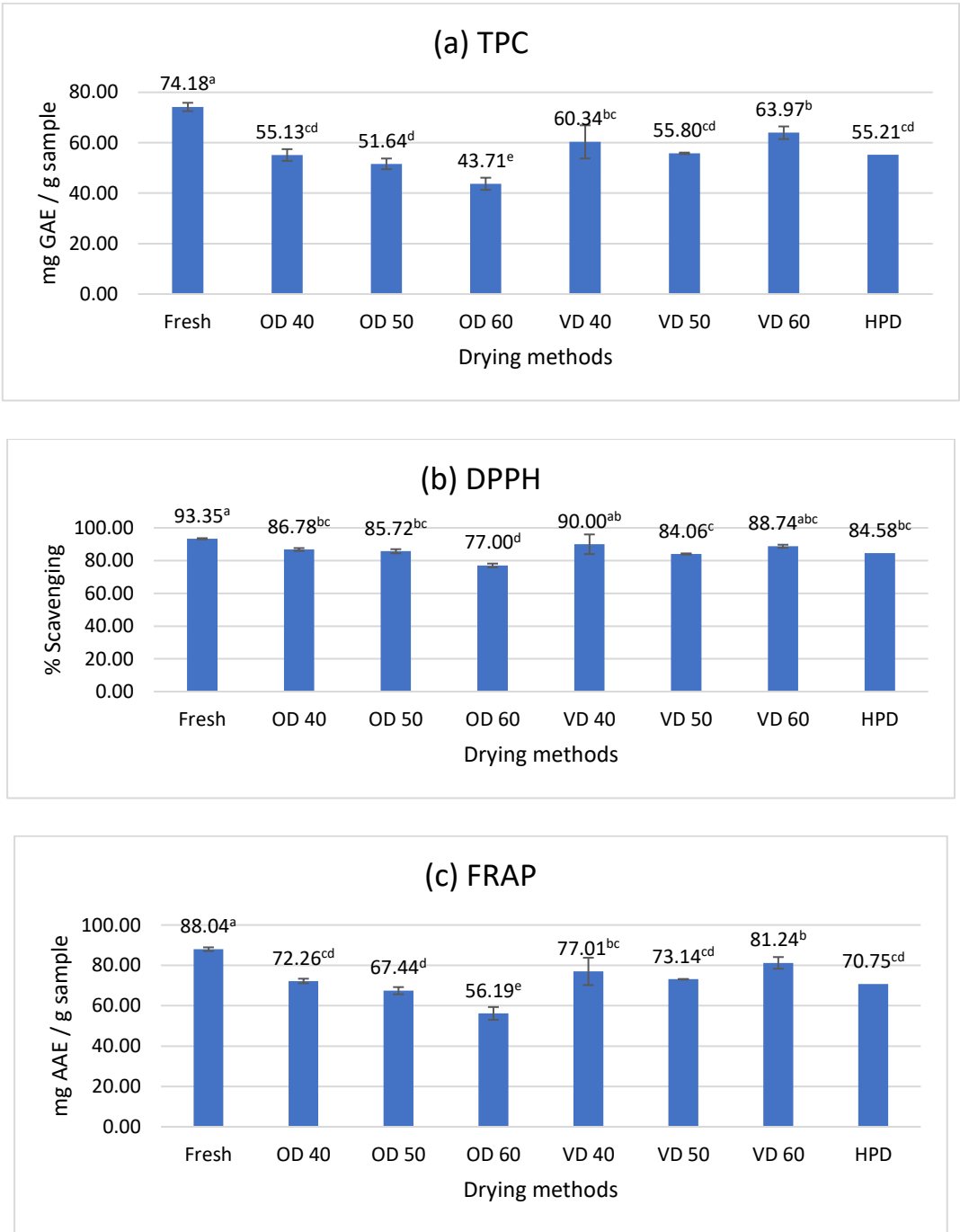


Figure 4.6 The influence of different drying methods and conditions on (a) Total phenolic content (TPC); (b) Percentage of scavenging (%) and (c) Ferric ion reducing potential (FRAP). Vertical bars indicate standard deviation

and values marked by the same letter are not significantly different (p>0.05).

The percent inhibition of DPPH radical reflects the antioxidant activity of the extracts (Alothman et al., 2009). This method is based on reduction of DPPH solution in the presence of hydrogen-donating antioxidant as result of non-radical DPPH-H formation in the reaction (Lavanya et al, 2012). The reduction is indicated by decolourisation of DPPH from purple to yellow. The more the decolourisation, the more is the reducing ability and is expressed as percentage of scavenging that indicates the percentage of free radical DPPH inhibition.

Figure 4.6(b) shows the DPPH as the percentage of radical scavenging activity of fresh and dehydrated LML subjected to HPD and different conditions of OD and VD. LML extracts from oven-, vacuum- and heat pump drying exhibited scavenging activity in the range of 77 to 90%. The highest percentage of scavenging was obtained from VD40, whereas the lowest percentage of scavenging was obtained from OD60. Higher percentage of scavenging was indicative of more free radicals were neutralised by more antioxidant present in the extract.

The DPPH values showed a relatively wide variation among the drying methods and conditions. For OD, DPPH scavenging activity

(%) reduced with an increase in drying temperature. The reduction of antioxidant activity caused by OD indicates the significant loss of natural antioxidants which are relatively unstable. Moreover, during drying, the leaves experience structural changes that might cause adherence of the plant cell components in the absence of water and probably inhibit the extraction of antioxidant from dehydrated samples (Garau et al., 2007). Higher drying temperature resulted in lower antioxidant activity as proven in this study. This might be due to drying at lower temperature constrains enzymatic degradation of antioxidant compound. The impact of heat treatment on the scavenging and degradation of bioactive compound was very much dependent on the duration of drying (Mediani et al., 2014). Long duration of drying may lead to the degradation of antioxidants, therefore affect their antioxidant activity.

Both DPPH and FRAP measure the antioxidative effect of the extract but they are based on different mechanisms. DPPH assay is based on the ability of antioxidant to act as radical scavengers, while FRAP assay measures the ability of antioxidant to perform as reducing agents (Orphanides et al., 2013). When the FRAP solution is mixed with sample's extract, a dark blue colour of the solution appears, which refers to the ferrous tripyridyltriazine complex and its absorbance is read at 595 nm (Benzie and Strain, 1996; Rabeta and Lin, 2015). The reaction is linearly related to the molar concentration

of the antioxidants. The FRAP value is expressed as mg Ascorbic Acid Equivalent (AAE) per g sample.

Figure 4.6(c) shows the FRAP values of fresh and dehydrated LML subjected to HPD and different conditions of OD and VD. According to Figure 4.6(c), the FRAP values were between 56.19 to 81.24 mg AAE/g sample for dehydrated leaves and 88.04 mg AAE/g sample for fresh LML. Higher FRAP value indicated more ferrous tripyridyltriazine that was produced by the reaction of LML extract and FRAP solution. The more ferrous tripyridyltriazine was produced by the reaction, higher antioxidant activity (FRAP) was obtained from the extract. The FRAP values for OD, VD and HPD samples show reduction after drying. The FRAP values of different dehydrated LML were in the order: VD60 > VD40 > VD50 > OD40 > HPD > OD50 > OD60. The highest FRAP value was obtained from VD60 but the lowest FRAP value was obtained from OD60 sample.

From Figure 4.6(c), the FRAP values show that different drying methods and conditions significantly affect the antioxidant capacity of LML (p<0.05). VD resulted in higher FRAP value of 73.14 to 81.24 mg AAE/g sample compared to the other two drying methods. This might be due to the operation of vacuum drying that is absence of oxygen and thus help to delay the potential oxidation, therefore more antioxidant compounds are preserved. The variation can also

be explained by the presence of oxygen in the drying air during OD and HPD that lead to enzymatic oxidation, especially redox enzyme that causes the conversion of antioxidant compounds (Nguyen et al., 2009). Similar to TPC and DPPH, an increase in drying temperature results in higher reduction of FRAP value in OD. This is due to the fact that high temperature would destroy the antioxidant components that thus lower the FRAP value in the extract. In addition, high temperature also decomposes the heat-unstable compounds, volatile compounds that escape to the external environment (Yen and Vu, 2017). Previous study by Alibas (2009) also reported that vacuum drying is the preferable method for drying collard leaves in comparison to hot air drying. This is also in agreement with Yen and Vu (2017) who reported that the reaction of enzyme at higher temperature and longer drying time resulted in less antioxidant compounds in *Limnophila aromatica* that was subjected to convective and vacuum drying.

Correlation of total phenolic content and antioxidant activities.

Antioxidant capacity of LML may be related to the amount of total phenolics. These compounds act as scavengers of free radicals produced during oxidation reactions. In order to determine the influence of the phytochemical compounds on LML antioxidants capacity, linear correlation was determined between the antioxidant

capacity and the TPC. Figures 4.7 and 4.8 show the correlation of TPC on DPPH scavenging activity and FRAP, respectively.

The coefficient of determination (R^2) of DPPH scavenging activity and FRAP were determined at 0.8023 and 0.943, respectively. The DPPH value of R^2 shows lower value than 0.9. This indicated that the weak correlation of FRAP value with TPC of dried LML subjected to OD, VD and HPD. The value of 0.8023 also indicated that the FRAP value is not reliable in predicting the total phenolic content in the different dried LML as compared to DPPH value. On the other hand, the measured antioxidant activities by DPPH was found to be highly correlated with TPC. The higher the TPC indicates the higher the antioxidant activity. This finding is almost similar with Yap et al. (2020) on the coefficient of determination (R^2) which was determined at 0.935 and 0.9536 for correlation of TPC with ABTS and DPPH, respectively in papaya leaves. Moreover, Youssef and Mokhtar (2014) also reported the high correlation of DPPH and ABTS with total phenolics in range of 0.9043 to 0.9885 that can be represented as useful predictors of antioxidant capacity in purslane leaves.

These results show that the preservation of phenolics is important for the retention of the antioxidant activities of LML. High value of correlation coefficient reveals that the phenolic compounds are the

major contributor to LML antioxidant activities. Overall, phenolic compounds play an important role in the antioxidant capacity of LML and thus can be further isolated and identified for better understanding on their potential of the biological properties and health benefits.

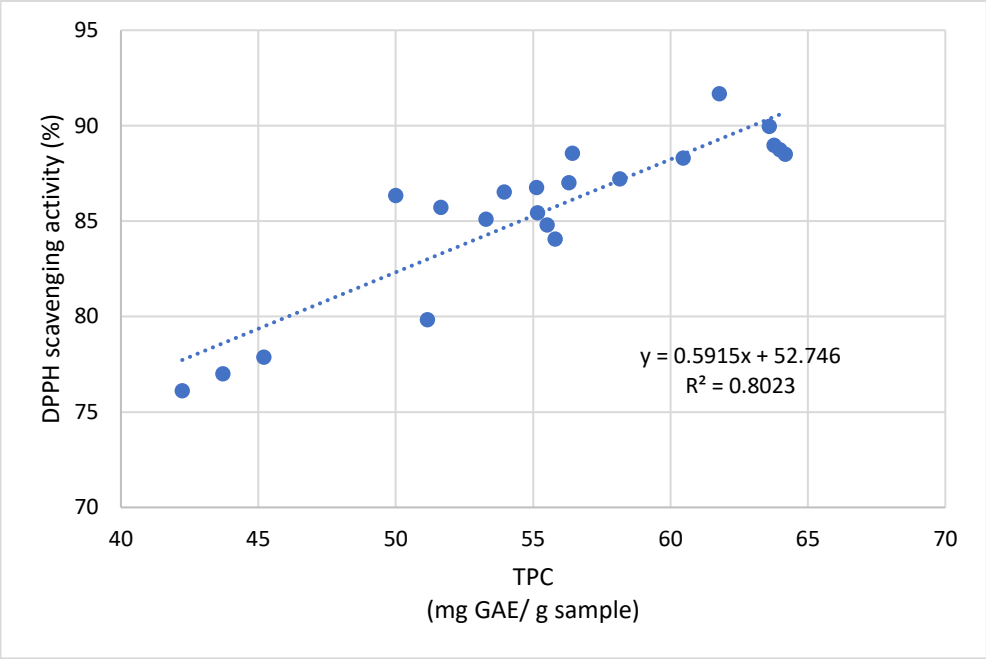


Figure 4.7: Correlation of total phenolic content (TPC) on DPPH scavenging activity in lemon myrtle leaves

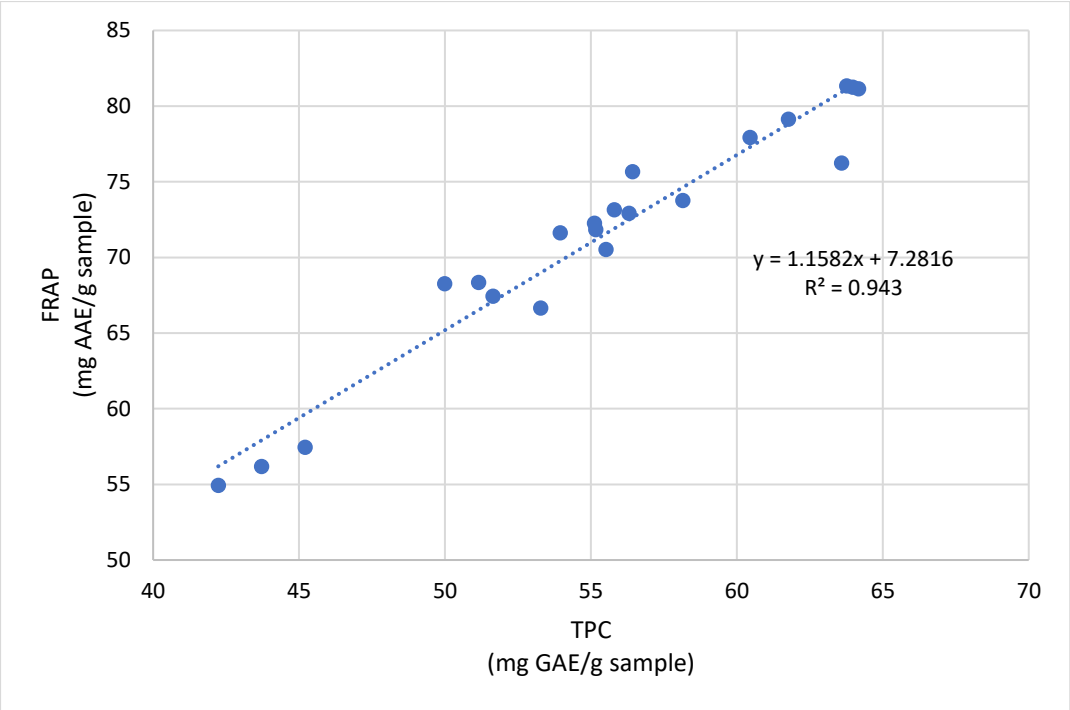


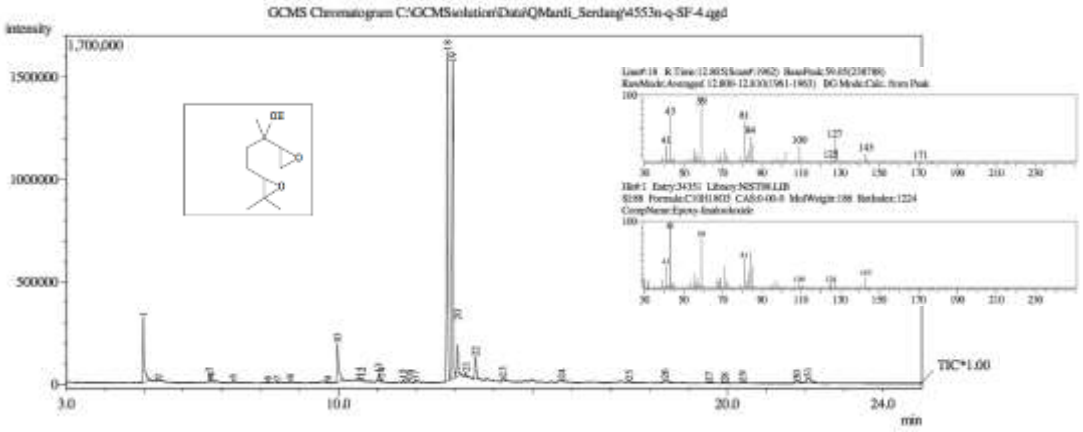
Figure 4.8: Correlation of total phenolic content (TPC) on FRAP in lemon myrtle leaves

As mentioned above, the findings on the effect of drying methods and conditions on the final moisture content, drying performances, biochemical content and colour changes, it was found that in favour of the maximum retention of each drying method, LML subjected to OD50, VD50 and HPD were chosen for the volatile retention analysis.

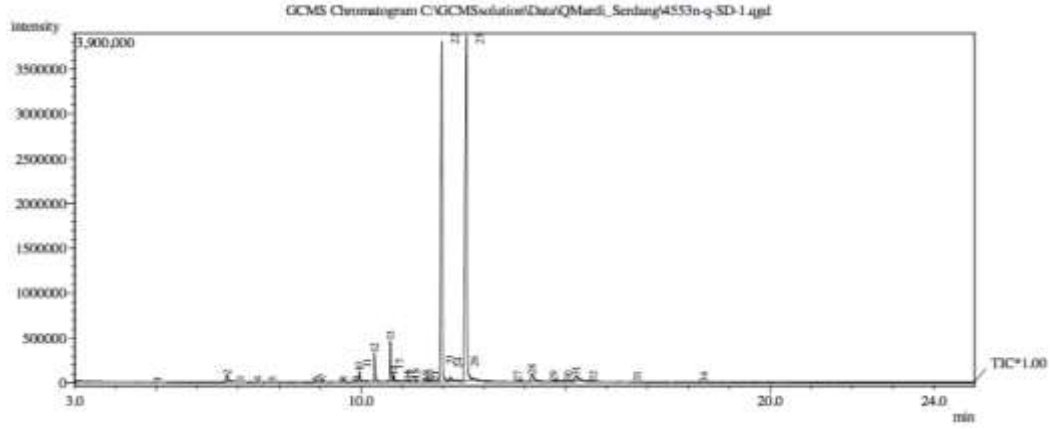
4.1.4. Volatiles retention

In this study, the LML essential oil (EO) was extracted by using simultaneous distillation and extraction (SDE) method and resulted in pale-yellow oil with strong lemony odour. After the extraction of the LML essential oil, GCMS has been used for the separation, identification and quantification the volatile compounds. Therefore, after SDE of OD50, VD50 and HPD samples, EO for each drying method was analysed for its volatile compound retention by using GC-MS. The chromatogram for the EO of each drying method is shown in Figure 4.9.

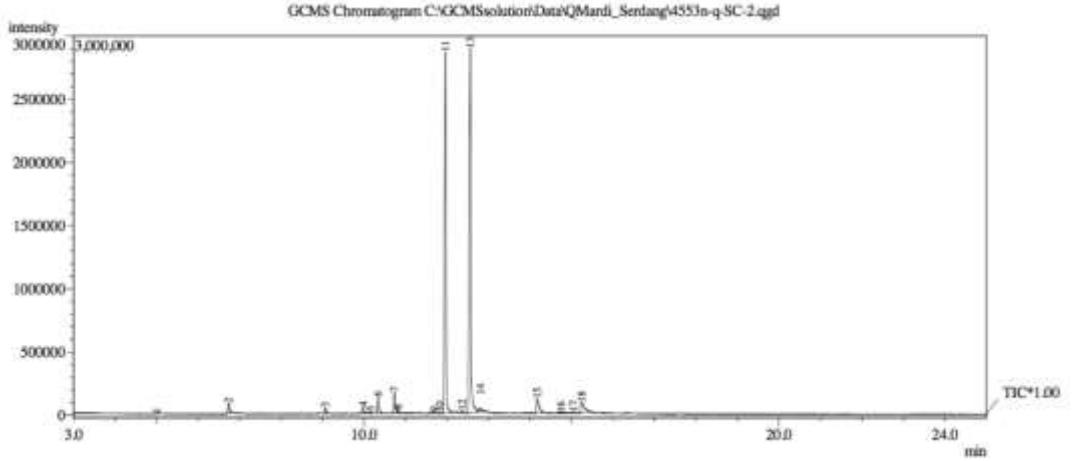
In Figure 4.9, two distinguished peaks are detected from GC-MS analysis chromatogram for OD50, VD50 and HPD. For each drying method, the peaks are detected at different retention time. OD50 samples showed peaks' retention time at 12.804 and 12.939 min. However, for VD50 and HPD, the peaks' retention time were at 11.975 and 12.578 min, and 11.963 and 12.562 min, respectively. OD50 results in later retention time might be due to the difference in volatile compounds that contained in the EO. The compound might have more molecular weight that results higher retention time compared to the peaks in VD50 and HPD (Buchaillot et al., 2009. Further details on the identified volatile compound are presented in Table 4.5.



(a)



(b)



(c)

Figure 4.9: Chromatogram of (a) OD50; (b) VD50 and (c) HPD

Table 4.5 shows the summary of identified and concentration of the volatile compounds with the retention time for LML subjected to OD50, VD50 and HPD. The percentage of area (% area) in the chromatogram indicates the concentration of the identified volatile compound in the EO. According to Table 4.5, two distinguished peaks from VD50 and HPD are identified as neral (cis-citral) and geranial (trans-citral). Whereas, the distinguished peaks in GC-MS analysis of OD50 (Figure 4.9(a) are both identified as epoxy-linalooloxide. This explains the difference of the distinguished peaks' retention time in OD50 compared to VD50 and HPD. Epoxy-linalooloxide was also identified in HPD sample but only at low concentration (0.67%). On the other hand, it was not detected in VD50.

Table 4.5: 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)
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
Epoxy-linalooloxide	12.804	33.59	nd	nd	12.808	0.67
	12.939	34.4				
	15.743	0.36				
	18.397	0.97				
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
citranellal hydrate (3,7-dimethyl-7-hydroxy-octanal)	14.23	0.78	nd	nd	nd	nd
	19.929	0.3				
2,7-dimethyl-2,7-octanediol	nd	nd	14.18	1.41	14.17	3.02
3,7-dimethyl-2,6-octadienoic acid	nd	nd	13.83	0.16	14.745	0.75
			14.71	0.27		
Citranellol	nd	nd	11.68	0.25	11.69	0.13
3,7-dimethyl-2,6-octadien-1-ol	nd	nd	12.175	0.61	nd	nd
Citranellol epoxide	nd	nd	nd	nd	nd	nd
TOTAL		**70.58**		**88.53**		**89.5**

Note: OD50: Oven drying at 50°C, VD50: Vacuum drying at 50°C and 50 mbar, HPD: heat pump drying, nd: not determined, % area: concentration of volatile compound

From Table 4.5, the maximum retention of volatile compounds was obtained from HPD, slightly higher than VD50 and the least was from OD50. This result might be due to shorter drying time of HPD compared to VD50 and OD50. This result is in agreement with the report by Phoungchandang et al. (2008) as HPD provided higher citronellal retention because of shorter drying time in white mulberry leaf drying. Moreover, as mentioned by Kerekes et al. (2019), VD was a better method in maintaining volatile compositions compared to convective and natural air drying in basil leaves essential oil. VD50 resulted in slightly lower retention of volatiles as indication of oil losses was slightly higher than HPD. This might be due to the split opening of the outer (exogenous) essential oil containers of the leaf as the vacuum (50 mbar) was applied during vacuum drying (Kereskes et al., 2019). According to Table 4.5, epoxy-linalooloxide was determined at four corresponding retention time and areas. The four-retention time which was at 12.804, 12.939, 15.743 and 18.397 min corresponded to areas of 33.59, 34.4, 0.36 and 0.97%, respectively. This might be due to the isomerisms of the epoxy-linalooloxide. Further differentiation of the isomers can be done by using GC-MSMS.

There were other citral derivatives identified in samples which were subjected to OD50, VD50 or HPD (Table 4.5). For instance, 3,7-dimethyl-6-octenal (citronellal) and linalool were identified only from

VD50 and HPD sample. Linalool has a floral, citrus-like odor and this volatile terpenoid is important for the quality of food because of their contribution to flavour and fragrance (Yang et al., 2013). Citronellal that was detected at RT of 10.150 min from VD50 has olfactive description of sweet, floral rosy, waxy and citrus green (Sultanbawa, 2016). The recovery of these identified volatile compounds including epoxy-linalool, neric acid and citral is important indication that the functionality of the LML EO is preserved because these compounds are responsible for the insecticidal activity of LML (Garba, 2016).

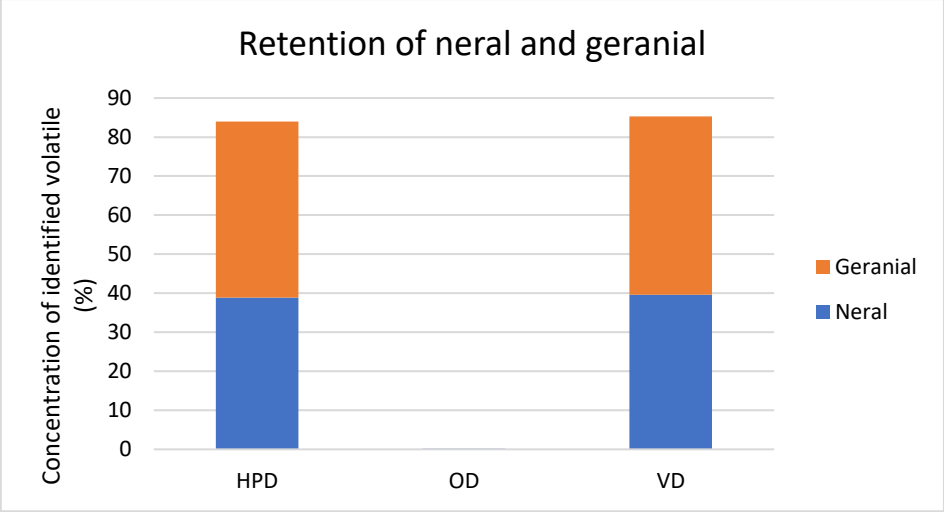


Figure 4.10 Retention of neral and geranial of essential oil of lemon myrtle leaves subjected to heat pump-, oven- and vacuum drying.

Figure 4.10 shows the concentration of two citral isomers, cis-citral (neral) and trans-citral (geranial) of LML EO subjected to HPD, OD and VD. The good quality of dehydrated LML is defined by its

retention of the citral which is related to the flavor of LML EO. As shown in Figure 4.10, only low concentration of neral was retained from dried LML by OD50. Whereas, higher retention of neral and geranial was obtained from VD50 and HPD samples. This might be due to the duration of OD50. Prolonged exposure to thermal environment might change the molecular structure of neral and geranial to other citral derivatives. Furthermore, convective drying that uses high temperature and intensive ventilation tends to cause undesirable alteration of the volatile compounds in the finished products (Abdollah et al., 2014; Antal et al., 2011).

Among the three drying methods, HPD is a more suitable method for LML drying as the samples remarkably maintained their volatile compounds especially neral and geranial content as comparable to VD50. In terms of colour, HPD sample also showed lower *a** value (greener), TCD, BI and higher chroma value. The biochemical content of the HPD samples also showed high retention of 74%, 91% and 80% for TPC, DPPH and FRAP, respectively. Since air velocity can affect the drying kinetic of HPD, the influence of drying air velocity on colour, biochemical content and volatile retention of LML was further investigated and this was discussed in the following topic.

4.2 Effect of drying air velocity on heat pump assisted drying performances, quality parameters and volatiles retention

Four different drying air velocities namely 1.5 m/s (FR1.5), 2.0 m/s (FR2.0), 2.5 m/s (FR2.5) and 3.0 m/s (FR3.0) were tested to investigate the effect on the heat pump drying performances, colour changes, biochemical content and the retention of the volatile compounds of lemon myrtle leaves essential oil.

4.2.1 Drying kinetics and final moisture content

Figure 4.11 shows the variation of moisture content of LML as a function of drying time at different drying air velocities in heat pump drying. After drying, the moisture content of LML was reduced from an initial value of 63.27 ± 4.66 gH_2O/g DS to a final moisture content of around 8.55 to 10.36 g H_2O/g DS. The final moisture content of the leaves was influenced by the drying air velocity. Final moisture content of 8.55 % w.b, 10.36 % w.b, 9.02 % w.b and 9.23 % w.b were achieved at the drying air velocities of 1.5 m/s, 2.0 m/s, 2.5 m/s and 3.0 m/s, respectively. The total drying time to achieve the equilibrium moisture contents were 34 h, 30 h, 28 h and 27 h for drying air velocities of 1.5, 2.0, 2.5 and 3.0 m/s, respectively. FR1.5 as the lowest drying air velocity gave the longest drying time, while the highest drying air velocity (FR3.0) showed reduction in total

drying time from 34 h to 27 h. Among all drying air velocities, FR1.5 gave the lowest final moisture content, while FR2.0 gave highest final moisture content.

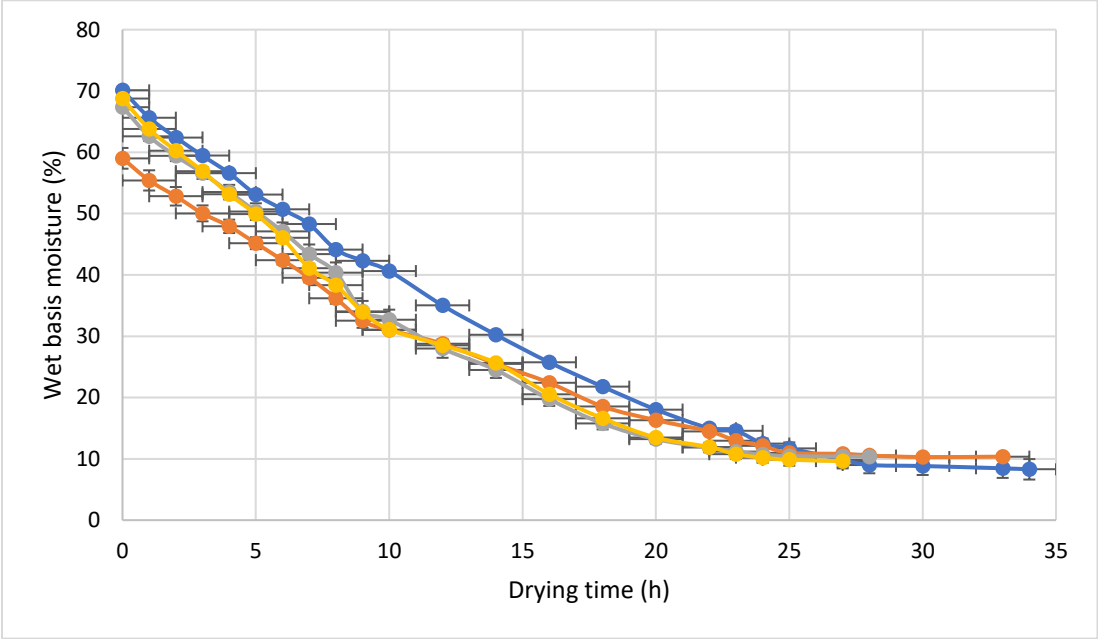


Figure 4.11 Impact of drying air flow rate on the moisture content (%wb) and time of drying (blue dots: FR1.5; orange dots: FR2.0; grey dots:FR2.5; yellow dots: FR3.0)

It is expected that higher drying air velocity would result in shorter drying time as compared to lower air velocity. From Figure 4.11, the drying time needed to reach the equilibrium moisture content (EMC) was shortened with an increase in drying air velocity from 1.5 to 3.0 m/s. This might be due to larger driving force for mass transfer at higher velocity of air. Thus, higher the drying air velocity, higher the removal of moisture from the product's surface. Therefore, increment in the drying air velocity resulted in the reduction of the

drying time from 34 to 27 h as the drying air velocity increased from 1.5 to 3.0 m/s.

Figure 4.12 shows moisture ratio against drying time for air velocity 1.5, 2.0, 2.5 and 3.0 m/s. The curves in Figure 4.12 show that the decrease in moisture content versus time is a non-linear curve which has the similar shape with the drying curves performed by Song et al. (2016). The shape of the curves indicates the moisture movement is governed by diffusion and the diffusion is dependent on the moisture content of the samples. In addition, it can be seen that at the lower moisture content, the removal of moisture from the product become increasingly difficult. Figure 4.12 also shows that drying air velocity affects the variation of the drying rate with the drying time. This might be caused by the higher drying air velocity that intensifies the drying rate which changes with time. Therefore, higher drying air velocity allows more moisture removal from the product surface. As the velocity of drying air is increased, higher drying rate is obtained that might due to the rapid circulation of air across the surface of the product. This is attributed to the better removal of the stagnant boundary layer of air around the moist product because of faster air flow (Babu et al., 2018). Therefore, more moisture removal is resulted from faster drying air velocity and thus, decreases the drying time of the LML. This is in agreement with the report by Taheri-Garavand and Meda (2018) that increasing of

air velocity resulted in increasing drying rate and reduction in drying time of savory leaves drying.

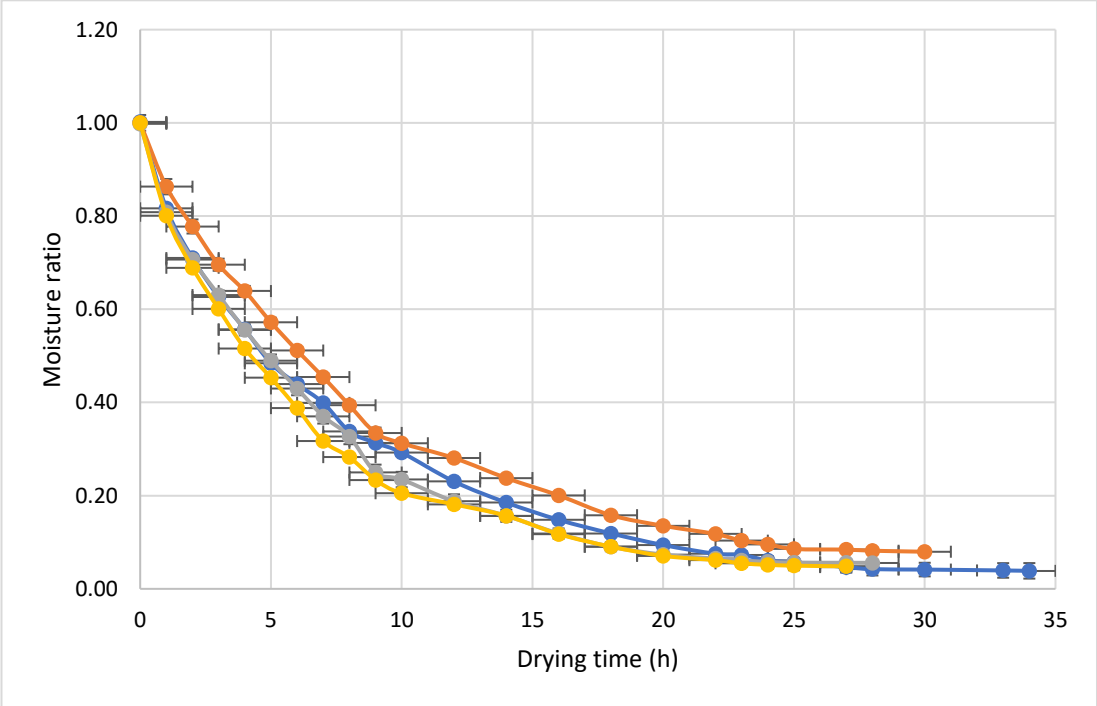


Figure 4.12 Drying ratio versus drying time for different drying air velocities in heat pump drying of lemon myrtle leaves (blue dots: FR1.5; orange dots: FR2.0; grey dots:FR2.5; yellow dots: FR3.0)

Variation in drying rate, according to moisture content for different air velocity of HPD is shown in Figure 4.13. According to Figure 4.13, higher rate was obtained from FR3.0, the highest air-drying flow rate. From Figure 4.13, it can be observed that all of the drying conditions exhibited falling-rate period which indicated the internal diffusion is the physical mechanism controlling the moisture

movement in heat pump drying. All drying conditions exhibited the falling-rate period until approximately 25 h indicating that higher rate was obtained at the beginning of the drying process and continued to decrease until the moisture reaches equilibrium. This result is in agreement with Aktas et al., (2017) who reported that the air velocity variation had significant role in conveying evaporated water from product surface hence reducing the drying time. Similar results were also reported by Shi et al. (2013) and Kaya and Aydin (2009) that the higher drying rate was obtained with an increase drying air velocity in drying of yacon slice and herbal leaves (nettle and mint leaves), respectively.

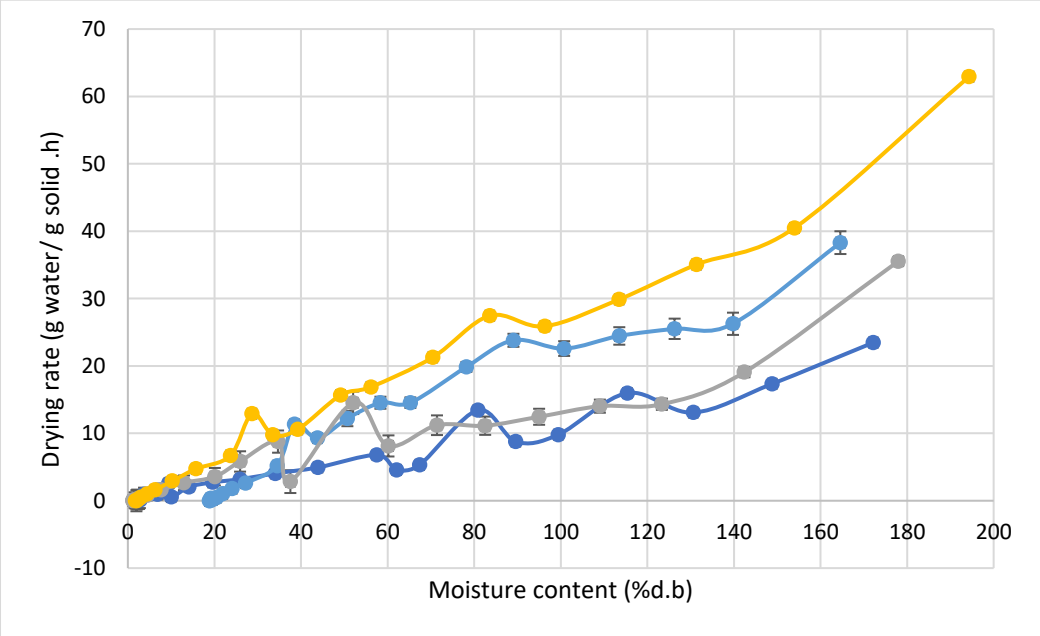


Figure 4.13 Variation in drying rate, according to moisture content for different conditions (dark blue dots: FR1.5; light blue dots: FR2.0; grey dots: FR2.5; yellow dots: FR3.0)

4.2.2 Effective Moisture Diffusivity

The simplified Fick's second law equation for diffusion was applied to estimate the effective moisture diffusivity of LML during HPD at different drying air velocity.

Figure 4.14 shows the plot of ln (MR) against the drying time of lemon myrtle leaves for all drying air velocities of HPD. With regard to the small thickness of LML (0.3574 mm), one falling period occurred in the drying of LML. According to the curves in Figure 4.14, the increasing velocity of drying air increased the slope of the straight line. This indicated the increment of the D_{eff} value as the drying air velocity was increased from 1.5 to 3.0 m/s.

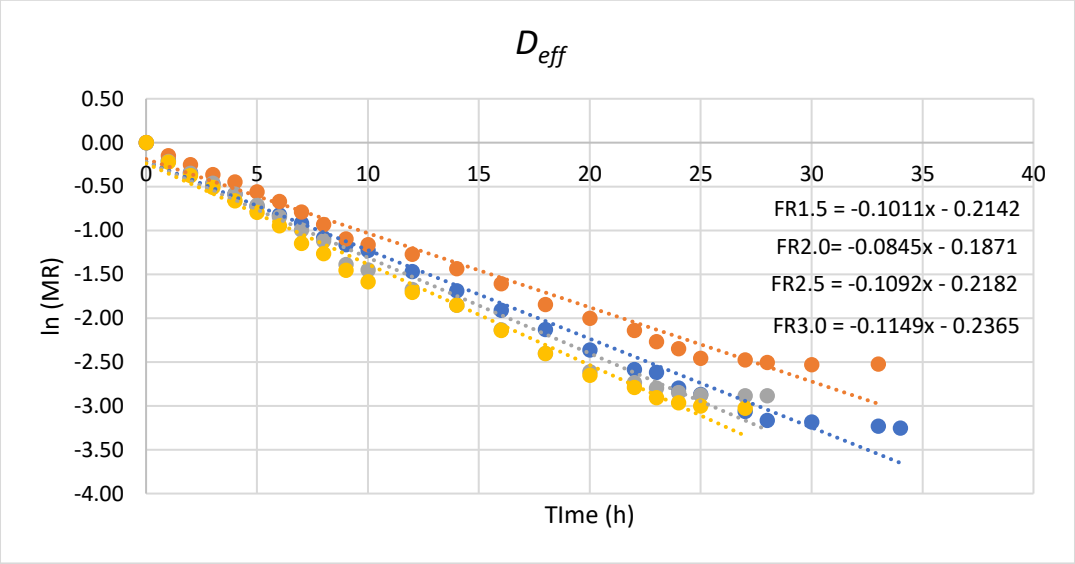


Figure 4.14 Variation of ln (MR) with drying time at different air velocity (FR) for heat pump drying of lemon myrtle leaves (blue dots: FR1.5; orange dots: FR2.0; grey dots: FR2.5; yellow dots: FR3.0)

The values of effective moisture diffusivity (D_{eff}) for different drying air velocities of the heat pump drying of the lemon myrtle leaves are given in Table 4.6(a). The effective moisture diffusivity, D_{eff} values for HPD at drying air velocities of 1.5 to 3.0 m/s are in the range of 1.0933×10^{-9} to 1.4867×10^{-9} m^2/s. The highest D_{eff} was estimated from FR3.0 followed by FR2.5, FR2.0 and finally FR1.5. The coefficient of determination, R^2 for each of the drying air velocities are also given in Table 4.6(a) and are in the range of 0.9729 to 0.9839. These values of R^2 indicated the good correlation of ln (MR) with time as the values are closely plotted to the regression line for all drying air velocities. The values of D_{eff} obtained from this study meet the standard range for food and agricultural products which are in the range of 10-^{12} to 10^{-8} (Zogzas et al., 1996). Table 4.6(b) shows the value of D_{eff} of other leafy materials subjected to HPD. The value for D_{eff} in this study is lower than the D_{eff} reported by these authors. The variation might be due to the difference of biomaterials, composition and geometry (i.e thickness and radius), drying air temperature, initial moisture content and physical or chemical pretreatment before drying process (Shi et al., 2013).

Table 4.6(a) Effective moisture diffusivity (D_{eff}) and coefficient of determination (R^2) for different drying air velocity for heat pump drying of lemon myrtle leaves

Drying air velocity (m/s)	D_{eff} (m^2/s)	R^2
1.5	1.093×10^{-9}	0.9836
2.0	1.3081×10^{-9}	0.9726
2.5	1.4129×10^{-9}	0.9747
3.0	1.4867×10^{-9}	0.9839

Table 4.6(b) Values of effective moisture diffusivity for other leafy materials from HPD

Leaves	D_{eff} (m^2/s)	References
Mint	4.27×10^{-11} to 2.96×10^{-10}	Ardestani et al., 2016
Centella asiatica L.	3.91×10^{-11} to 1.82×10^{-10}	Trirattanapikul and Phoungchandang, 2012
Sweet basil (*Ocimum baslicum* Linn.)	9.44×10^{-13} to 1.75×10^{-11}	Phoungchandang and Kongpim, 2011
Orthosiphon aristatus leaves	3.91×10^{-11} to 1.82×10^{-10}	Klungboonkrong et al., 2018

The D_{eff} values varied according to the drying air velocity. This result shows the influence of drying air velocity on the estimated D_{eff}. The values of D_{eff} increase as the drying air velocity is increased. Higher drying air velocity facilitates the removal of the first layer of water vapour from the product surface (Dorneles et al., 2019). Thus, in order to replace the great loss of moisture, the diffusion or speed of moisture transfer is increased and hence the effective moisture diffusivity is also increased. The variation of D_{eff} value on different drying air velocities also might be due to higher drying air velocity, higher mass transfer rate and easier diffusion which results in higher and more uniform D_{eff} value (Taheri-Garavand and Meda, 2018). The same drying behavior of moisture diffusivity was observed from the drying of Avishan leaves, as the mean values of the moisture diffusivity parameter were increased about 2.3 times by increasing of the air velocity from 0.5 to 1.2 m/s (Khazaei et al., 2008).

4.2.3 Colour changes

The impact of different drying air velocity in heat pump drying of lemon myrtle leaves on values of colour parameters of L^*, a^*, b^*, chroma, hue angle, total colour difference (TPC) and browning index were compared to the fresh leaves' colour parameters and discussed below.

Table 4.7 shows the values of L^*, a^* and b^* for heat pump drying of lemon myrtle leaves at different drying air velocity. The L^* represents the lightness level of the sample. From Table 4.7, all of the L^* values of the dried LML showed reduction in comparison to the L^* value of fresh leaves. Drying LML at air velocity of 2.0 m/s gave the highest L^* value and followed by velocity of 1.5 m/s and 3.0 m/s while 2.5 m/s gave the lowest L^* value. The higher L^* value indicate the lighter colour of the sample, while lower L^* value represents the darker colour of the sample. Drying at velocity of 2.0 m/s resulted in higher a^* value with no significant difference ($p<0.05$) from fresh sample. Whereas, drying air velocity at 2.5 m/s resulted in lower L^* value as indication of darker sample. However, higher velocity at 3.0 m/s, the L^* value was higher than the velocity of drying air at 1.5 m/s and 2.5 m/s. This could be due to shorter drying time as LML was dried at 3.0 m/s and thus reducing the exposure to the thermal environment. Thus, resulted in brighter colour of the dried sample.

Table 4.7 Values of *L, *a** and *b** of different drying air velocity for heat pump drying of lemon myrtle leaves**

Drying air velocity (m/s)	*L**	*a**	*b**
Fresh	46.38 ± 0.08^{a}	-5.87 ± 0.11^{e}	28.98 ± 0.15^{a}
1.5	43.79 ± 1.22^{b}	-3.11 ± 0.11^{a}	26.38 ± 0.61^{a}
2.0	45.51 ± 0.41^{a}	-4 ± 0.15^{c}	27.94 ± 0.37^{b}
2.5	42.88 ± 0.91^{b}	-3.67 ± 0.17^{b}	27.79 ± 0.41^{b}
3.0	43.71 ± 0.67^{b}	-4.8 ± 0.05^{d}	27.35 ± 0.89^{a}

Mean values ± standard deviation (n=3 replications) within the same column with the same letter are not significantly different (p>0.05)

The greenness of the sample is represented by its *a** value from greenness (negative value) to redness (positive value). Higher *a** value indicates the closer the samples' colour to redness, whereas lower *a** value indicates the greener the samples' colour. From Table 4.7, the *a** values for different drying air velocity of HPD are in the range of -4.8 to -3.11 with -5.87 for the fresh sample. Regardless of the air velocity, the dehydrated leaves are less green than the fresh samples. The *a** values are significantly (p<0.05) influenced by the different drying air velocities. According to Table 4.7, the lowest *a** value was obtained from the highest drying air velocity of 3.0 m/s. The low *a** value (more negative) represents the high intensity of green colour in the sample. It can be seen that the highest drying air velocity resulted in the lowest *a** value, while the lowest drying air velocity resulted in higher *a** value. This is because drying at 1.5 m/s took longer time for drying to be completed. Therefore, a

remarkable loss of green colour was observed at lower drying air velocity of 1.5 m/s with 47% reduction compared to the fresh sample.

The yellowness or blueness of a sample is indicated by the *b** value. The lower *b** value represents the blueness, while higher *b** value indicates the yellowness of the samples' colour. Table 4.7 shows the variation of *b** value of LML dried at different drying air velocities. The *b** value for dehydrated samples are in the range of 26.38 to 27.94, whereas 28.98 for the fresh sample. This indicated that the dehydrated leaves showed darker colour than the fresh sample. The highest *b** value was obtained by drying LML at 2.0 m/s, while drying at 1.5 m/s gave the lowest *b** value. The lower *b** value indicates the less yellowish colour that leads to less bright colour of the sample. The lower *b** value from LML dried at air velocity of 1.5 m/s might be related to longer drying time. The prolonged drying time could promote LML discolouration that was associated to the degradation of chlorophyll when exposed to heat and oxygen. Therefore, *b** value was reduced and resulted in darker colour sample. This result is in agreement with Rocha et al. (1993) who reported that lower b* values were obtained at longer total time of drying and lower drying air velocity in drying of sweet basil (*Ocimum basilicum).*

The variation of the *L**, *a** and *b** values was influenced by the drying air velocity. At higher velocity, the *L** value was lower and the *a** value was higher than the *L** and *a** values for lower drying air velocity. This might be due to higher velocity tends to give shorter drying time and therefore results in less colour change. On the other hand, low velocity gives longer duration of drying which in turn causes sample to be darker. Faster air flow facilitates the rapid removal of the water on the surface of the leaves. As more loss of the superficial layer of moisture on the surface of the leaves, more chlorophyll is exposed to the thermal degradation and oxidation. Therefore, chlorophyll oxidation, hydrolysis and isomerisation can occur and cause the colour change from brighter to darker colour that is represented by the lower *L** value. Decomposing chlorophyll during thermal processing causing the central magnesium atom of the porphyrin ring of chlorophyll is replaced by two hydrogen atoms and form pheophytin that causes the undesirable colour change from bright green to olive brown (Potisate et al., 2014; Meyer, 1973). As the protective layer of water film is removed, the rate of Maillard reaction is increased, allow the colour degradation and results in significant colour changes (Song et al., 2016). Therefore, as consideration to shorter drying time, drying at air velocity of 2.0 m/s is effective in minimising colour changes especially the value of greenness (*a**) during HPD.

Chroma is the colour parameter that represents the colour saturation. Figure 4.15(a) shows the chroma values of LML subjected to HPD at different drying air velocity. The values of chroma for fresh leaves were obtained as 29.56 and the dehydrated leaves were in the range of 26.56 to 28.22. According to Figure 4.15(a), the highest chroma value which indicates the more intense (pure) colour was observed from FR2.0 sample. Whereas, FR1.5 resulted in significantly lower chroma values compared to other drying air velocities. Lower chroma value for 1.5 m/s was confirmed by higher *a** value (more positive) compared to other drying air velocities which gave lower *a** value (more negative). The higher value of chroma for lower drying air velocity might be due to long duration of exposure that leads to loss of chlorophyll from the leaves during drying. Positive increase in *a** value and lower *b** values from drying air velocity of 1.5 m/s represents remarkable change as the values of *a** and *b** are associated for lower chroma value of dried LML.

Hue angle describes the relative amount of redness and yellowness where 0°/360° is defined for red/magenta, 90° for yellow, 180° for green and 270° for blue or purple colour, or intermediate colours between adjacent pairs of these basic colours (Kortei and Akonor, 2015). A higher hue angle indicates a greener colour product for green samples. According to Figure 4.15(b), the hue angle values are in the range of 178.55 to 178.60 for dried LML subjected to HPD

at different drying air velocity and 178.63 for fresh leaves. The highest hue angle value was obtained from the highest drying air velocity, 3.0 m/s. This might be due to the reduced total drying time for higher air velocity that could retain more chlorophyll pigments. Higher preservation of chlorophyll which is represented by high *a** value contributes to higher hue angle resulted from drying at 3.0 m/s. Similar to this result, Ardestani (2015) mentioned that the low negative *a** value confirms the more greenness the colour and consequently, resulted in high hue angle thereby indicating more greenness of the dried mint leaves.

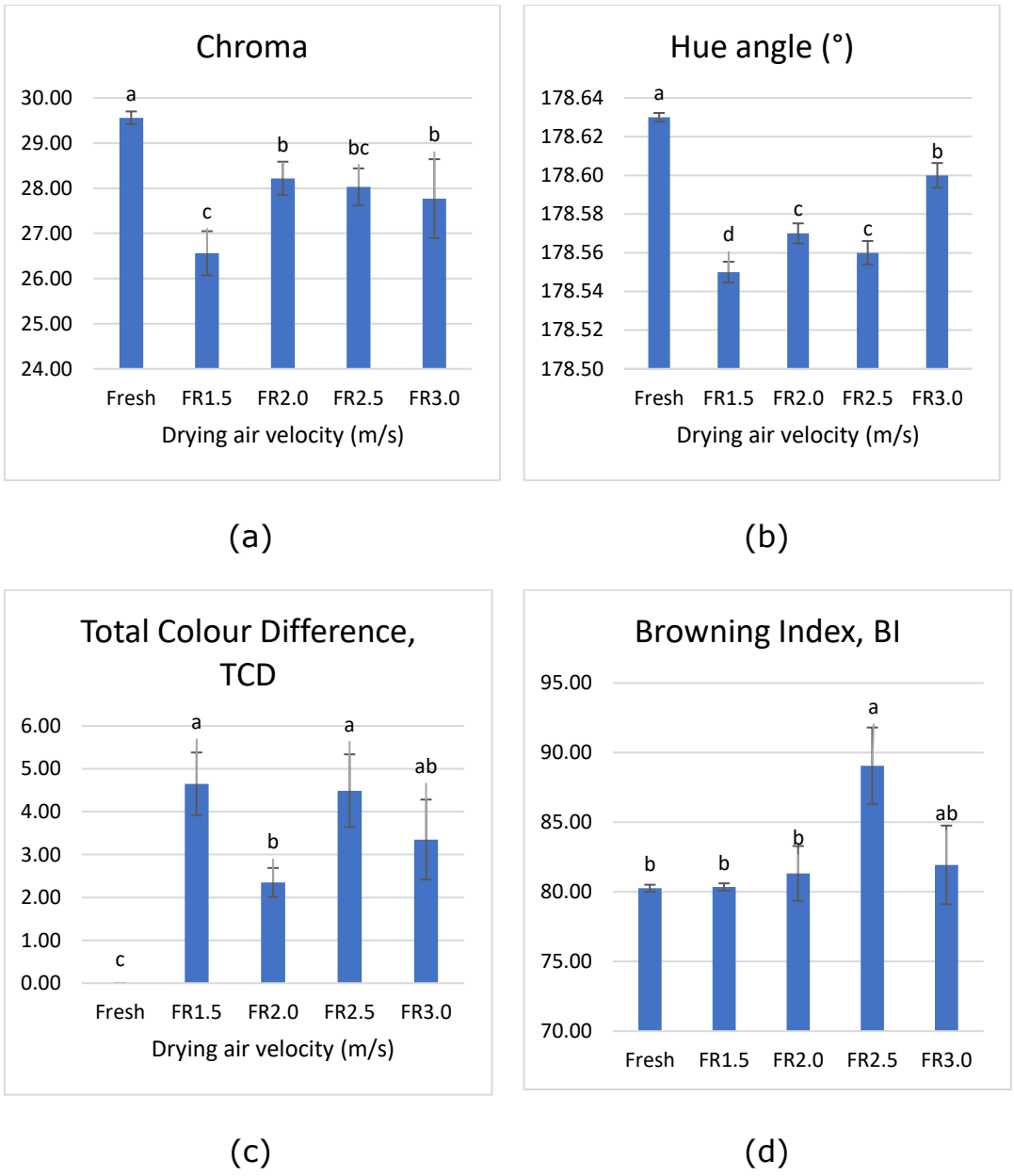


Figure 4.15 Values of (a) Chroma, (b) hue angle, (c) total colour difference and (d) browning index of different drying air velocity for heat pump drying of lemon myrtle leaves. Vertical bars indicate standard deviation and values marked by the same letter are not significantly different (p>0.05)

Total colour difference (TCD) shows the degree of overall change in dried samples with respect to the colour of fresh LML. Minimum value of total colour change indicates a good colour preservation of the dehydrated products, whereas higher TCD value indicates more changes the leaves experience during dehydration process. The TCD of different drying air velocities of heat pump drying of lemon myrtle leaves are presented in Figure 4.15(c). The TCD for dehydrated LML was in the range of 2.35 to 4.35. The values of TCD were varied between the drying air velocities. Drying air velocity of 2.0 m/s gave the lowest TCD value whereas, drying air velocities of 1.5 m/s gave the highest TCD. The results indicate that more colour difference could be found when lower air velocity was applied. This might be related to the longer total time of drying that causes the significant changes in colour parameters, especially the greenness that contribute to great colour difference compared to the fresh leaves. This finding was also supported by the highest a^* value (more positive) that was obtained from FR1.5 samples, furthest from the fresh leaves that represents significant changes LML experienced during HPD. This result is in agreement with Salarikia et al. (2016) that the maximum TCD was resulted from lower drying air velocity (0.5 m/s) whereas, higher drying air velocity of 1.5 m/s gave minimum TCD value of 18.48 from peppermint drying.

Browning index (BI) shows the purity of brown colour in the sample. Higher BI value indicates higher purity of brown colour of the samples. Figure 14.5(d) shows the browning index for LML dried at each air velocity in heat pump drying. BI values for different drying air velocities were not significantly different from the fresh LML except for drying air velocity of 2.5 m/s that resulted in significantly (p<0.05) higher BI compared to other drying air velocities. FR2.5 gave the 11% increment in BI value, while only 0.1%, 1.3% and 2.1% for drying air velocity of 1.5 m/s, 2.0 m/s and 3.0 m/s, respectively. The colour of dried LML changes due to browning reaction, which is always associated with the Maillard reaction (Kumar et al., 2014). The presence of oxygen and heat during drying promotes simultaneous and frequent Maillard reaction to occur and therefore, increases the formation of brown pigment in dried LML. Hence, the BI is higher.

Drying at lower drying air velocity of 2.0 m/s shows lower a^* and b^* value, chroma as well as low total colour difference and BI value. These results show the suitability of selecting FR2.0 as the drying air velocity for dehydration of LML by using heat pump drying. The minimal changes of colour indicates small degradation of the pigments of LML, which is shown in the dehydrated colours of LML in HPD. The effect of drying air velocity variation on biochemical content of LML will be discussed in the following topic.

4.2.4 Biochemical analysis

Qualitative evaluation of total phenolics in water extract of the different dried lemon myrtle leaves was estimated by the method of Folin-Ciocalteu and total antioxidant activities in term of DPPH and FRAP assays. Figure 4.16(a), (b) and (c) show the influence of different air velocity on total phenolic content (TPC), percentage of radical scavenging activity (DPPH) and ferric ion reducing potential (FRAP). TPC was evaluated from both fresh and dehydrated leaves subjected to different drying air velocity in heat pump drying of lemon myrtle leaves. According to Figure 4.16(a), the TPC content was in the range of 49.60 to 55.21 mg GAE/g sample, whereas TPC for fresh LML was 74.18 mg GAE/g sample. This indicates that the degree of reduction of TPC depends on the drying condition. The highest TPC value was obtained from drying air velocity of 2.0 m/s but the lowest TPC value was obtained from drying at 1.5 m/s.

From Figure 4.16(a), the TPC value for dehydrated leaves are not significantly different except for drying air velocity of 1.5 m/s. Longer drying time (34 hours) at lower drying air velocity resulted in higher TPC reduction. This result might be due to the longer exposure to thermal environment causes degradation of phenolic content. The loss of phenolic compounds during drying might be attributed to the oxidation of phenolic compounds. Exposure to

thermal environment may cause cell disruption that leads to releasing of oxidative and hydrolytic enzymes that are responsible for the oxidation of phenolic compounds. Longer the thermal exposure, longer the time polyphenol oxidase gets to oxidise the polyphenolic contents (Garau et al., 2007; Goula et al., 2016) and causes the lower retention of TPC in lower drying air velocity. This result is in agreement with Taseri et al. (2018) who reported that higher decrease-loss ratios (%) of TPC was obtained from drying air velocity of 1.5 m/s compared to 2 m/s in heat pump drying of grape pomace.

To evaluate the free radical scavenging activity of different antioxidant substances, DPPH assay has been widely used (Youssef and Mokhtar, 2014). Figure 4.16(b) shows the DPPH as the percentage of radical scavenging activity of fresh and dehydrated LML subjected to HPD at different drying air velocities. LML extracts from drying air velocities of 1.5 m/s to 3.0 m/s exhibited scavenging activity in the range of 79.44 to 88.65%. The highest percentage of scavenging was obtained from drying air velocity of 2.0 m/s. The higher value of DPPH indicates more free radicals were neutralized as result of DPPH reaction that lead to higher antioxidant activity present in the extract.

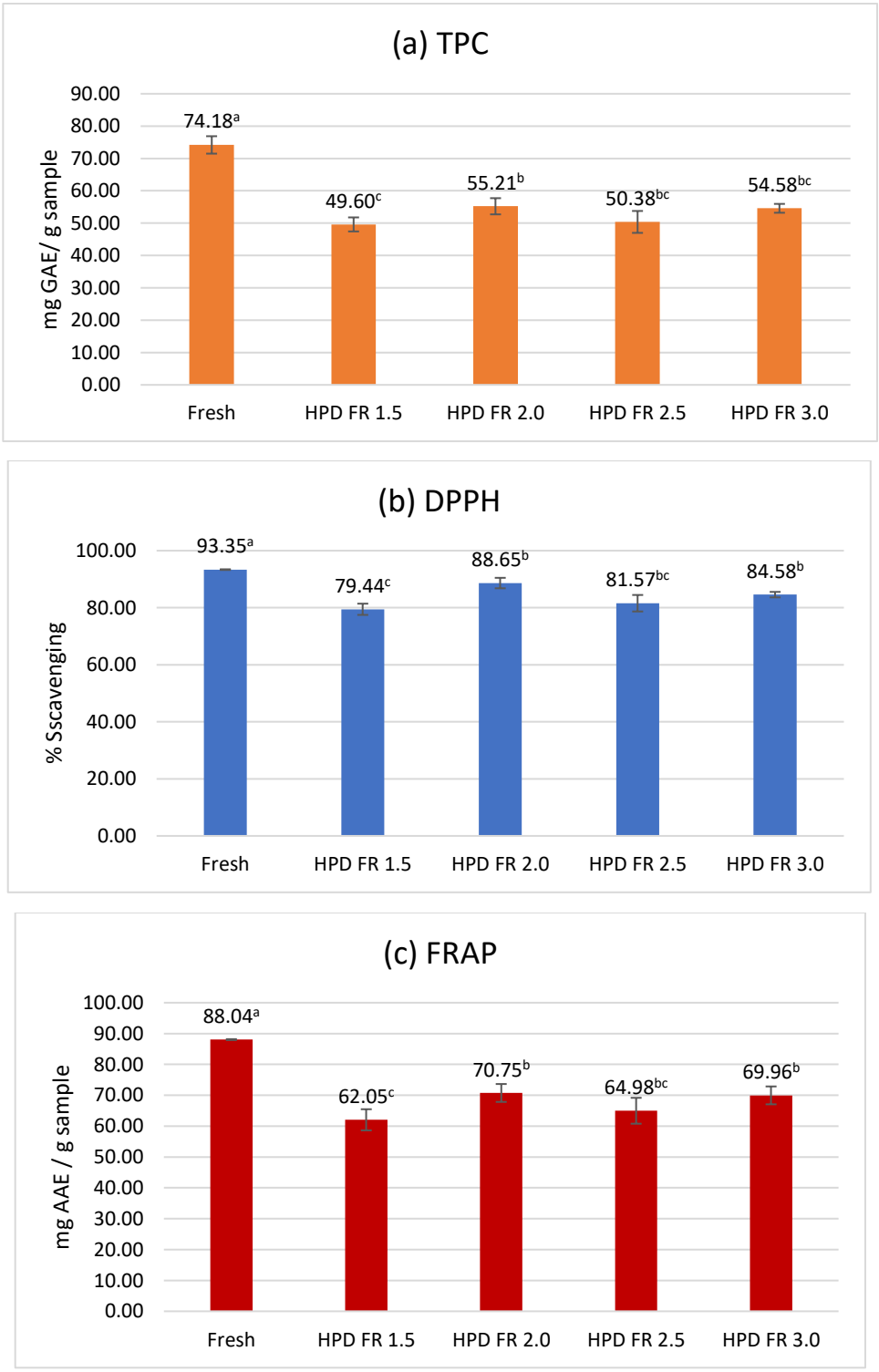


Figure 4.16 The influence of different air flowrate for heat pump drying on (a) Total phenolic content (TPC); (b) percentage of radical scavenging activity (DPPH) and (c) Ferric ion reducing power (FRAP). Vertical bars indicate standard deviation and values marked by the same letter are not significantly different (p>0.05)

From Figure 4.16(b), all of the drying air velocity exhibit reduction of DPPH value compared to fresh leaves. Drying velocity of 1.5 m/s showed significant (p<0.05) lower retention of DPPH compared to other drying conditions. This result indicates the significant loss of unstable natural antioxidants during drying process. This might be due to long drying times that promote a decrease in antioxidant activity (Garau et al., 2007). This is in agreement with the report by Rodriguez et al. (2013) that the lower antioxidant activity was obtained from lower drying air velocity in thyme drying as result of prolonged drying time. Therefore, the proper selection of drying conditions that includes drying rate (drying time) is important to maximise the retention of the bioactive compounds as prolonged duration of the drying may lead to degradation of antioxidants and reducing their antioxidant activity.

Similar to DPPH, ferric reducing antioxidant potential (FRAP) assay also has been used to measure the ability of the antioxidant as reducing agents in the extract and indicates the total antioxidant potential of food and plant extracts. FRAP value is indicated by variation of absorbance due to the production of ferrous tripyridyltriazine as reaction of the sample extract and FRAP solution. Figure 4.16(c) shows the FRAP of fresh and dehydrated LML subjected to heat pump drying at different drying conditions. According to Figure 4.16(c), the FRAP values are between 62.05 to

72.75 mg AAE/g sample whereas 88.04 mg AAE/g for fresh sample. Similar to TPC and DPPH, regardless drying air velocity, the dehydrated leaves gave lower retention of FRAP value compared to fresh leaves. The highest FRAP value was obtained from drying air velocity of 2.0 m/s whereas, lowest FRAP value was obtained from drying air velocity of 1.5 m/s. Higher FRAP value indicates more ferrous tripyridyltriazine was produced by the reaction of FRAP solution and LML extract. The more ferrous tripyridyltriazine produced by the reaction, the higher the FRAP value is obtained from the extract.

From Figure 4.16(c), the FRAP values of the dehydrated leaves are not significantly (p>0.05) different among the drying air velocity except for drying air velocity of 1.5 m/s. FR1.5 resulted in the lowest FRAP value (p<0.05) compared to other three velocities. This result might be due to the long duration of drying for LML until the equilibrium moisture content was achieved. Similar to DPPH and TPC, lower drying air velocity resulted in higher reduction of FRAP value. This is due to the fact that prolonged exposure to thermal degradation that leads to the activation of redox enzymes that attributes to the conversion of antioxidant compounds in the extract. This is also in agreement with the report by Paslawska et al. (2020) that reported on effect of dehydration time on the antioxidizing potential of lemon thyme extract. The authors found out that higher

bioactive compound degradation effect was obtained as the longer dehydration process proceeded in drying of lemon thyme. This indicates that the major parameter which impacts the retention of valuable properties of LML subjected to HPD is the process time. Therefore, in order to maximise the retention of the biochemical content of LML extract, the proper drying condition that can lead to shorter drying time can be applied to reduce the degradation of phytochemical and thus increases the antioxidising potential of dehydrated LML.

Correlation of total phenolic content and antioxidant activities

To determine the contribution of the polyphenolics present in LML extracts to scavenging activity and ferric reducing antioxidant potential, a correlation analysis was performed (Figure 4.17 and Figure 4.18). Total phenolic content of extracts obtained from different drying air velocity was found to exhibit linear relationship to the scavenging activity and FRAP of the LML extracts (R^2=0.9248 and 0.966, respectively). These results are slightly higher than R^2 reported by Vuong et al. (2013) which was 0.87 and 0.84 for relationship between total polyphenol content with scavenging and total antioxidant activity of papaya leaf extracts. The coefficient of determination in this study are almost similar to with Yap et al.

(2020) that R^2 for TPC with DPPH and FRAP were determined at 0.9536 and 0.935, respectively. The highly linear correlation represents a useful antioxidant capacity predictor and suggests that LML can be a good source of antioxidants.

These results show that to maintain high antioxidizing potential from LML extract, it is important to apply good preservation technique on its total phenolic content. This justifies the importance on applying proper preservation technique on the biochemical content in way to avoid active components deterioration upon drying process.

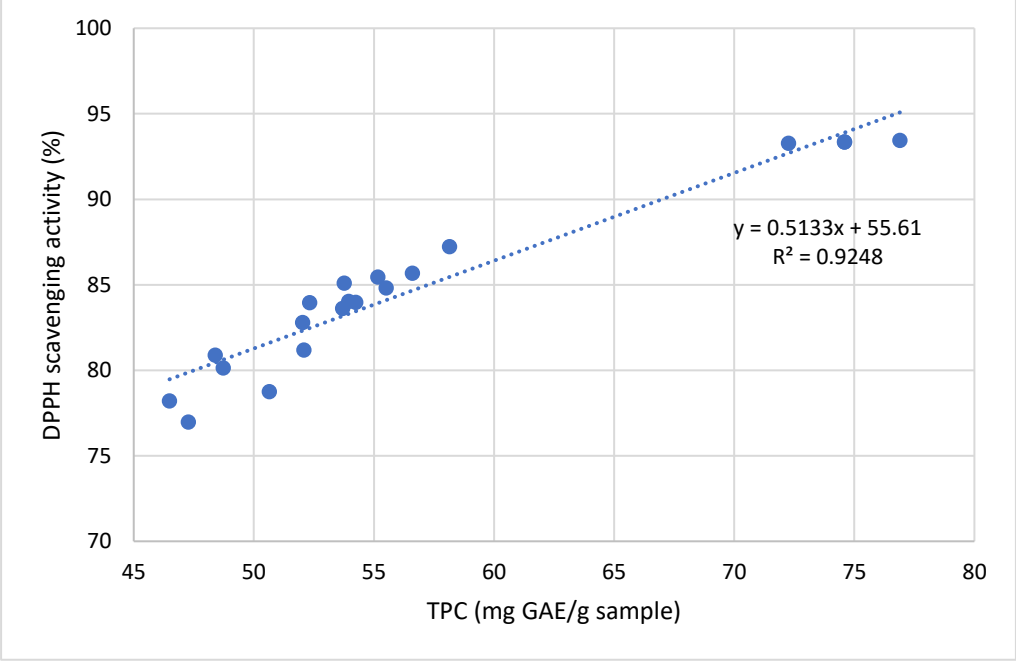


Figure 4.17 Correlation of total phenolic content (TPC) on DPPH scavenging activity in lemon myrtle leaves subjected to heat pump drying

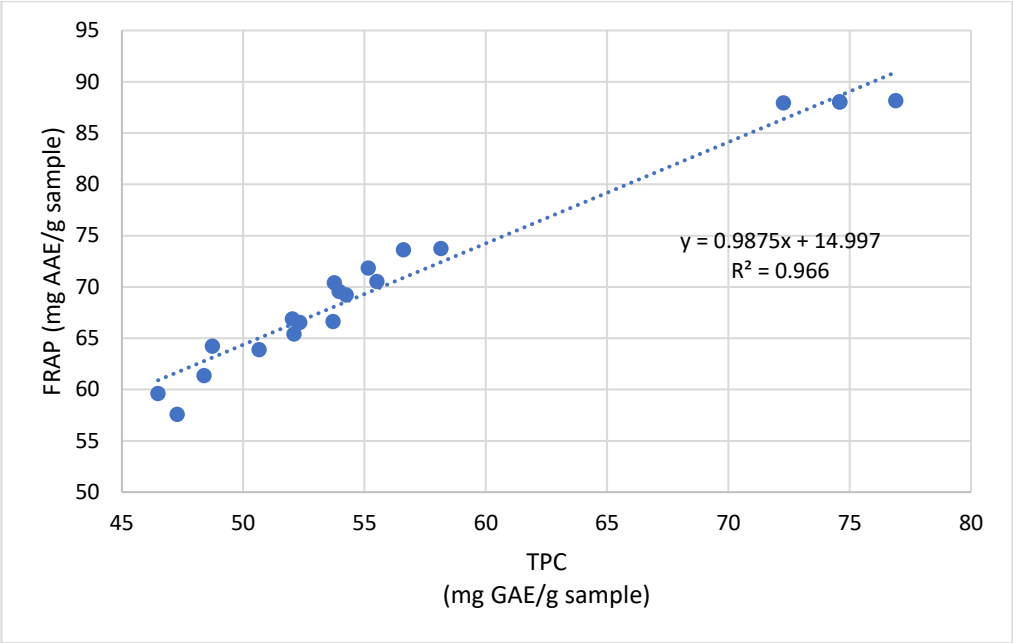


Figure 4.18 Correlation of total phenolic content (TPC) on FRAP in lemon myrtle leaves subjected to heat pump drying

Drying at air velocity at 2.0 m/s shows higher retention of TPC, DPPH and FRAP. These results show the suitability of choosing FR2.0 as drying air velocity on heat pump drying of LML. High retention of biochemical content indicates that the deterioration of active components in the extract is minimal, which is shown in high values of TPC, DPPH and FRAP in LML extract. The effect of drying air velocity on retention of volatiles will be discussed in the following topic.

4.2.5 Volatiles retention

Figure 4.19 shows the Gas Chromatography – Mass Spectrometer (GC-MS) chromatogram for each essential oil of LML subjected to heat pump drying at different drying air velocity. As shown in Figure 4.19, two distinguished peaks are detected from GC-MS analysis of chromatogram for drying air velocity of 1.5, 2.0, 2.5 and 3.0 m/s. The first and second distinguished peaks for each of the chromatogram is detected at the range of 11.963 to 11.985 min and 12.562 to 12.599 min. However, from Figure 4.19(a), there are another two peaks can be detected which are at retention time of 12.829 and 12.958 min. Further details on the identified volatile compound are presented in Table 4.8.

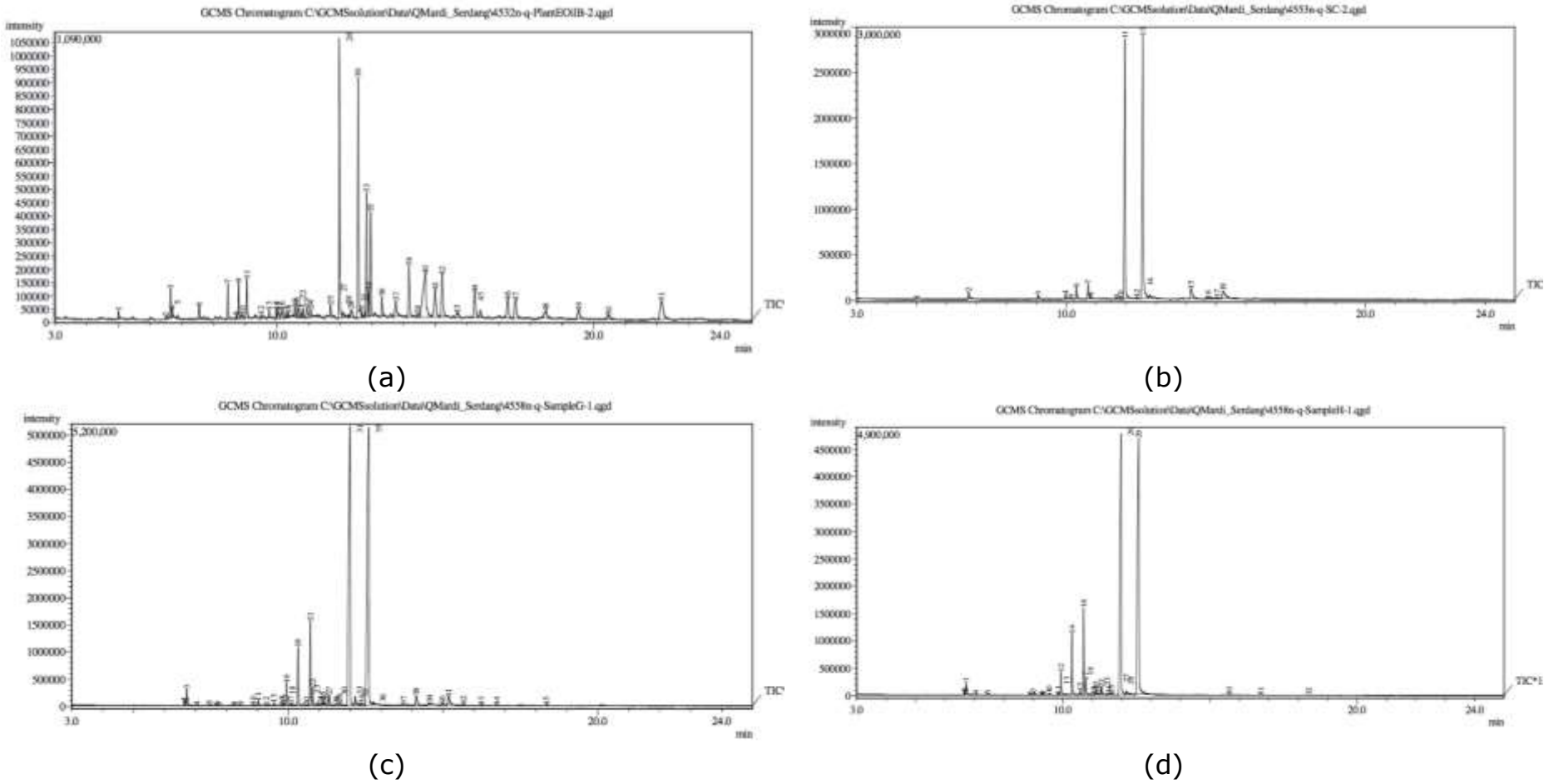


Figure 4.19 Chromatogram for lemon myrtle leaves subjected to heat pump drying at different drying air velocities of (a) FR1.5 (b) FR2.0 (c) FR2.5 (d) FR3.0

Table 4.8 shows the summary of identified volatile compounds in lemon myrtle leaves subjected to heat pump drying at different drying air velocity with respect to its peak's number and retention time (min). Two main components of citral which are neral (cis-citral) and geranial (trans-citral) were detected from all of the drying conditions. The cis-citral and trans-citral were detected in the range of 14.59 to 38.88% and 14.64 to 45.08% among all drying air velocity. Drying at air velocity of 2.0 m/s gave the highest retention of cis- and trans-citral which were 38.88 and 45.08%, respectively. Whereas, drying at lower air velocity, 1.5 m/s gave the lowest retention of cis- and trans-citral which were only 14.59 and 14.64%, respectively. The variation of the citral content in essential oil of dehydrated LML is due to the drying time. Change of composition might be due to the loss of more volatile substances (such as monoterpenes) by volatilisation due to longer drying time. This result is in agreement with Dorneles et al. (2019) who reported that the longer drying time, higher loss of volatile substances due to volatilization in *Piper umbellatum* L.

From Table 4.8, the variation of volatile compounds identified varied among the different drying air velocity. Other than neral and geranial, there are other volatile compounds identified from the LML essential oil of different drying air velocity that contributes to the lemon and sweet aromas of LML such as beta-myrcene, citronellal

and linalool (Forbes-Smith and Paton, 2002). Myrcene is an acylic monoterpenes that is used as an intermediate in the production of aroma and flavour chemicals (Buchaillot et al., 2009). According to Table 4.8, beta-myrcene or 7-methyl-3-methylene-1,6-Octadiene can only be identified from EO of FR2.5 while citranellol and citronella hydrate can only be identified from FR2.0 and FR1.5 samples, respectively. Whereas, retention of other citral derivatives which were 3,7-dimethyl-2,6-octadienoic acid and 2,7-dimethyl-2,7-octanediol showed influence to drying air velocity. It can be seen that the percentage of concentration decreases as the drying air velocity was increased and was not determined in the highest drying air velocity, 3.0 m/s. This might be due to the intense or higher airflow that influence the evacuation of these substances. Therefore, the higher airflow allows stronger evaporation of the surface moisture that then increase the evacuation of the molecules to surrounding air (Pirbalouti et al., 2013). This variation of identified volatile compounds also can be due to the loss of the volatile substances. The losses of the volatile compounds may be due to the rate of drying air velocity during the dehydration process. Higher rate of drying air velocity, higher evaporation rate as more air flow is supplied on the surface of the leaves that causes more loss of the volatile compounds. This indicates that the drying condition of a drying process influence the variation of volatile compounds' retention in LML subjected to HPD.

Table 4.8 Identified volatile compounds of lemon myrtle leaves subjected to HPD at different drying air velocity

Identified volatile compound	FR1.5		FR2.0		FR2.5		FR3.0	
	RT (min)	(% area)	RT (min)	(% area)	RT (min)	(% area)	RT (min)	(% area)
Cis-citral (3,7-dimethyl-2,6-octadienal)	11.970	14.59	11.963	38.88	11.996	37.42	11.985	37.97
Trans-citral (3,7-dimethyl-2,6-octadienal)	9.98 12.565	0.48 14.16	12.562	45.08	12.599	40.08	12.585	40.34
Beta-myrcene (7-methyl-3-methylene-1,6-Octadiene)	nd	nd	nd	nd	6.680	0.23	nd	nd
Epoxy-linalooloxide	6.685 8.715 9.500 12.829 12.958	0.37 0.19 0.21 6.92 6.03	12.808	0.67	nd	nd	nd	nd
6-methyl-5-hepten-2-one	6.64	1.63	nd	nd	6.637	0.38	6.65	0.21
Trans-geraniol (3,7-dimethyl-2,6-octadien-1-ol)	nd	nd	nd	nd	11.660 12.17	0.27 0.84	11.660 12.170	0.23 0.62
Linalool (3,7-dimethyl-1,6-octadien-3-ol)	9.050	2.12	9.06	0.72	9.03	0.36	9.040	0.35
Citronellal (3,7-dimethyl-6-octenal)	10.16	0.63	10.16	0.25	10.135	0.11	10.135	0.16
Citranellol	nd	nd	11.69	0.13	nd	nd	nd	nd
Citranellal hydrate (3,7-dimethyl-7-hydroxy-octanal)	11.695	0.80	nd	nd	nd	nd	nd	nd
3,7-dimethyl-2,6-octadienoic acid	13.76 14.686	1.72 8.58	14.745	0.75	13.725 14.570	0.14 0.27	nd	nd
2,7-dimethyl-2,7-octanediol	14.17	4.12	14.17	3.02	14.135	1.16	nd	nd
Total		**62.55**		**89.50**		**81.26**		**79.88**

Figure 4.20 shows the concentration of two isomers, cis-citral (neral) and trans-citral (geranial) of LML EO subjected to heat pump drying at different drying air velocity. The retention of the citral that is associated to the flavour of LML defines the good quality of dried LML. As shown in Figure 4.20, only low concentration of neral and geranial were retained after drying at 1.5 m/s which were 14.59 and 14.64%, respectively. Whereas, higher retention of neral and geranial were obtained from higher drying air velocity (more than 2.0 m/s). This might be due to the longer time of exposure of sample to heated air that allowed the oxidation of sample and thus caused the compositional change in dried samples (Buchaillot et al., 2009). Furthermore, more thermal degradation occurs as result of prolonged exposure of drying air and causes undesirable alteration in molecular structure of neral and geranial to other citral derivatives. As mentioned by Buchaillot et al. (2009) that a reduction in drying time is helpful to minimise the time for the volatiles to be lost. Therefore, in order to maximise the retention of the citral, drying time is important to be shortened to reduce the volatilization of these compounds during LML dehydration process.

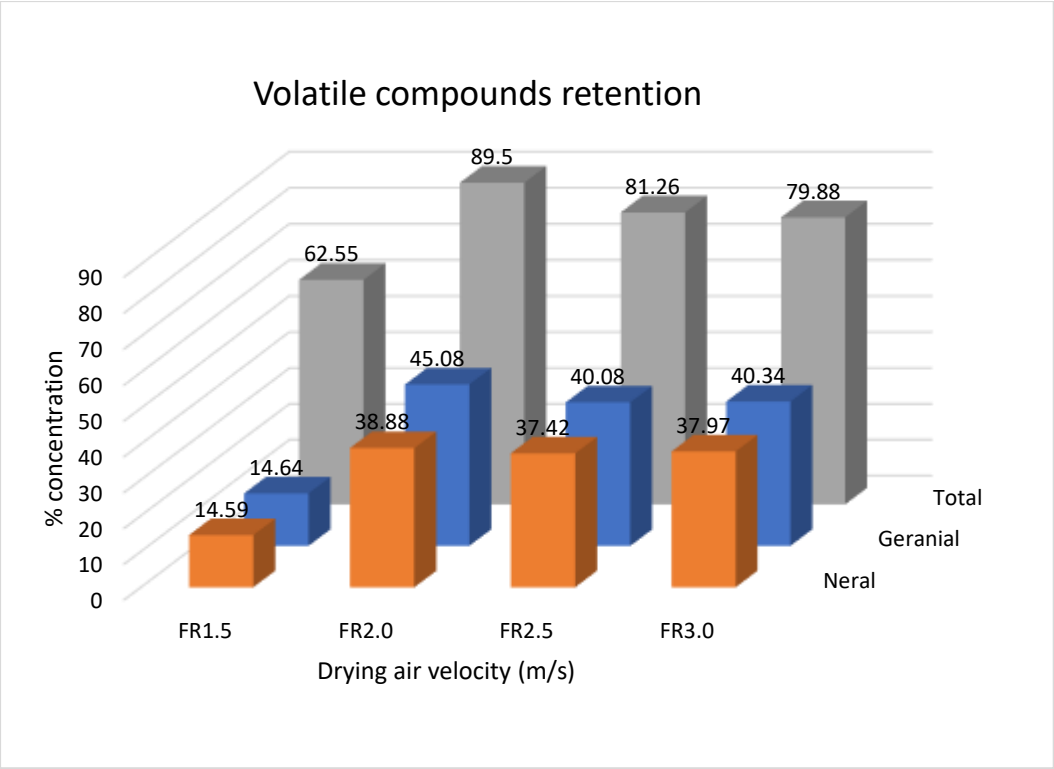


Figure 4.20 Retention of neral and geranial of essential oil of lemon myrtle leaves subjected to heat pump drying at different drying air velocity

Among the four drying air velocities, FR2.0 was the most suitable air velocity for LML subjected to HPD as it resulted in high retention of volatiles content especially neral and geranial content. In terms of colour, FR2.0 sample also showed low *a** and *b** value, TCD, BI and high chroma. The total phenolic content and antioxidant activities of samples dried at 2.0 m/s also showed high retention of 74%, 95% and 80% for TPC, DPPH and FRAP, respectively. Therefore, the conditions of drying played an important role in improving the quality of herbs especially in LML drying to ensure the good preservation of the bioactive components and thus, maintain its functionality to human's health. As HPD FR2.0, OD50 and VD50 showed good

preservation of biochemical and volatile content for each of the drying method, these drying methods were tested for their stability during 6 month storage and this is discussed in the following topic.

4.3 Effect of packaging conditions and temperature during storage on dried lemon myrtle leaves quality

In the process of drying, water is removed appreciably from the product, resulting in the reduction of moisture content. Moisture content of a product that is required to be stored for an extended period of time, needs to be reduced to a level that is low enough to ensure that the moisture contained in the material is not available to microbial activity. Hence, it can minimise microbial spoilage as well as physical, chemical changes and also undesirable chemical reactions that may occur during storage (Zhu and Shen, 2014).

4.3.1 Final moisture content of dehydrated LML and moisture profile during 6 month storage

The final moisture content of the dried LML subjected to the three drying methods namely OD50, VD50 and HPD FR2.0 are shown in Table 4.9. In addition, the moisture variation profile from storage period of the first month to the sixth month is also shown in Table 4.9. The moisture content of the dried sample before storage ranged from 9.88 to 11.10% whilst the fresh sample had a moisture content of 63.27 ± 4.66%. During storage, the stored samples showed

varying degrees of moisture loss that depended on the temperature of storage environment and packaging condition. After 6 months of storage, the highest loss ($p<0.05$) in moisture content was found in samples (subjected to the three drying methods) that was stored in vacuum packaging at chilled condition (VCH). The changes of sample moisture content during storage were significantly affected ($p<0.05$) by the drying methods. The variation of the sample's moisture content may be associated with the storage humidity in the different packing of storage bags applied in this study. The same result was reported by Norawanis et al. (2018) where decrement of moisture content was exhibited by sample packed in the plastic bags and glass container at 8 weeks of storage period of *Orthosiphon stamineus* dried leaf.

Table 4.9 Moisture content of different dried lemon myrtle leaves at Month 0 (after drying) until Month 6 of storage

Sample	Storage condition	Month of storage						
		0	1	2	3	4	5	6
Fresh	Before drying	63.27 ± 4.66						
HPD	After drying	10.31±0.11[a]						
OD50		11.10±0.09[b]						
VD50		9.88 ± 0.19[b]						
HPD	NRT		12.13±0.52[a]	12.05±0.16[a]	12.19±0.23[a]	9.42 ± 0.07[a]	10.16±0.78[a]	10.17 ± 0.13[a]
	NCH		11.76±0.46[a]	11.25±0.24[a]	11.82±0.62[a]	10.65±0.15[b]	10.78±0.57[a]	9.31 ± 0.85[a]
	VRT		11.17±0.31[a]	11.09±0.16[a]	11.02±0.27[a]	9.38 ± 0.6[a]	9.63 ± 0.2[ab]	9.65 ± 1.09[a]
	VCH		10.83±0.26[a b]	11.14±0.48[a]	11.36±0.18[a]	10.42±0.45[a b]	9.82 ± 0.86[a]	8.63 ± 0.38[ab]
OD50	NRT		11.31±0.4[a]	11.53±0.19[a]	11.3 ± 0.32[a]	9.97 ± 0.41[a]	10.37±0.18[a]	9.96 ± 0.28[a]
	NCH		10.73±0.46[a]	11.62±0.32[a]	11.65±0.14[a]	10.81±1.21[a]	9.89 ± 0.89[a]	9.24 ± 0.65[a]
	VRT		10.97±0.21[a]	11.17±0.11[a]	10.81±0.22[a b]	9.87 ± 0.11[a]	9.66 ± 0.38[a]	9.28 ± 0.17[a]
	VCH		10.22±0.11[a b]	11.13±0.61[a]	11.05±0.12[a]	10.16±0.53[a]	10.64±0.53[a]	9.08 ± 0.59[a]
VD50	NRT		9.62±0.02[a]	10.38±0.07[a]	10.19±0.21[a]	8.95 ± 0.09[a]	8.75 ± 0.47[a]	8.99 ± 0.31[a]
	NCH		9.42±0.16[a]	10.58±0.14[a]	10.82±0.26[a]	9.43 ± 0.71[a]	10.67±0.97[a]	9.00 ± 0.04[a]
	VRT		9.48±0.08[a]	10.16±0.52[a]	9.63 ± 0.5[a]	9.1 ± 0.35[a]	8.69 ± 0.05[a]	8.53 ± 0.23[a]
	VCH		9.23±0.35[a]	10.06±0.31[a]	9.97 ± 0.3[a]	9.99 ± 0.03[a]	10.06±1.24[a]	7.93 ± 0.02[a]

Values are means of three (3) replications ± standard deviation. HPD, heat pump drying, OD50, oven drying at 50°C, VD, vacuum drying at 50°C and 50 mbar, NRT, Normal packaging stored at room temperature, NCH, normal packaging stored at chilled condition, VRT, vacuum packaging stored at room temperature, VCH, vacuum packaging stored at chilled condition. Values marked by the same letter are not significantly different (p>0.05).

Figure 4.21 shows moisture reduction of oven-, vacuum- and heat pump dried LML after 6-months storage. The moisture reduction variation for different packaging and storage temperature is also shown in Figure 4.21. As shown in Figure 4.21, among the three drying techniques, VD samples gave the highest moisture reduction which was recorded at 19.9% as compared to HPD that was 16.29% after 6 months of storage. This result shows storage temperature and packaging condition has a significant effect on the moisture content of dried lemon myrtle leaves for 6 months storage. This indicates that the degree of moisture reduction is dependent on the drying methods where HPD shows lower moisture of reduction compared to the other two drying methods, regardless of the storage conditions. This is in agreement with Ji and Pi (2012) who reported on the effect of storage period of 2 months on the moisture content of oven- and sun-dried okra stored at dark dry place and over a hearth. The sample stored at 30^{o}C and 60% relative humidity showed varying degrees of moisture reduction.

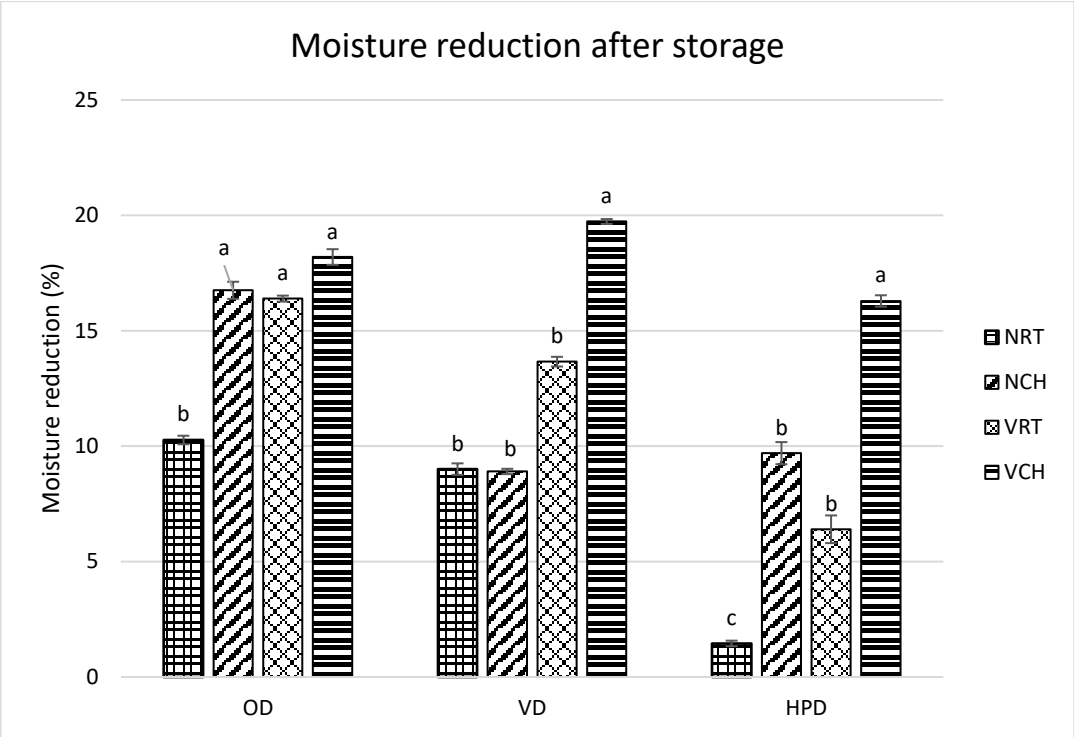


Figure 4.21 Moisture reduction after 6 months of storage for HP, OD and VD in different storage conditions. Vertical bars indicate the standard deviation and values marked by the same letter are not significantly different (p>0.05).

By comparing the storage at room temperature and chilled condition in Figure 4.21, it can be found that chilled condition recorded higher moisture reduction. Higher value of moisture difference between the after drying and after 6 months storage might be due to the change in relative humidity that facilitate the moisture transfer from sample to the chilled environment. Therefore, storing dried LML at chilled condition resulted in more moisture reduction compared to room temperature storage. As shown in Figure 4.21, HPD samples gave lower moisture reduction (p<0.05). This might be due to less alteration of the cell structure of the HPD sample. Less alteration to

the dried product's structure leads to better preservation of the original full and regular shape of parenchyma cell structures of the HPD sample.

On the other hand, LML subjected to OD and VD, the cell structure experienced more damage and alteration that made the structure unable to retain the moisture and hence the moisture easily evaporated from the dried LML (Thamkaew et al., 2020). Thus, more moisture reduction was observed in OD and VD samples. This finding is in line with Tummanichanont et al. (2017) and Klungboonkrong et al. (2018) that HPD was reported to be a better drying method at maintaining the fuller and regular shape of parenchyma cells structure in *Andrographis paniculata* and *Orthosiphon aristatus* as compared to convective drying. However, for NCH and VCH samples, higher amount of moisture loss was found as indication of lower values of absorbed moisture from the storage environment. This finding is in line with the findings reported by Razak et al. (2018) that higher moisture content was shown in *O. stamineus* powder stored at 25°C compared to 10°C during 4 weeks of storage. This is because 10^{o}C has a lower relative humidity hence it tends to give higher humidity gradient that facilitate moisture removal. Therefore, it can be concluded that storing dried sample in chilled condition would avoid the sample to absorb moisture from the storage ambient, instead it will reduce the moisture content of the samples.

Hence keeping the sample at a relatively low moisture level. This is key in ensuring longer shelf life of the sample.

4.3.2 Effect of packaging and storage temperature on dried lemon myrtle leaves quality

4.3.2.1 Impact on bioactive compound

Total phenolic content for oven-, vacuum- and heat pump dried lemon myrtle leaves during storage at a one-month interval is shown in Figure 4.22. Figure 4.22 also shows the variation of TPC content at different storage conditions. From Figure 4.22, all storage conditions showed decreasing amount of TPC during the 6-month storage period. From Figure 4.22(a), VD samples showed higher reduction of TPC compared to OD and HPD samples. The same trend is observed in Figure 4.22(b), 4.22(c) and 4.22(d). VD samples shows the most TPC loss after 6 months storage in all storage conditions. The TPC loss in VD samples were recorded as 55.98 to 20.19, 31.32, 16.54 and 27.9 mg GAE/g sample for samples stored in NRT, NCH, VRT and VCH. Meanwhile, the TPC retention from HPD samples was the least compared to other methods. The TPC was only reduced to 30.19, 35.8, 33.13 and 36.49 mg GAE/g samples for NRT, NCH, VRT and VCH storage condition from initial value of 53.2 mg GAE/g sample.

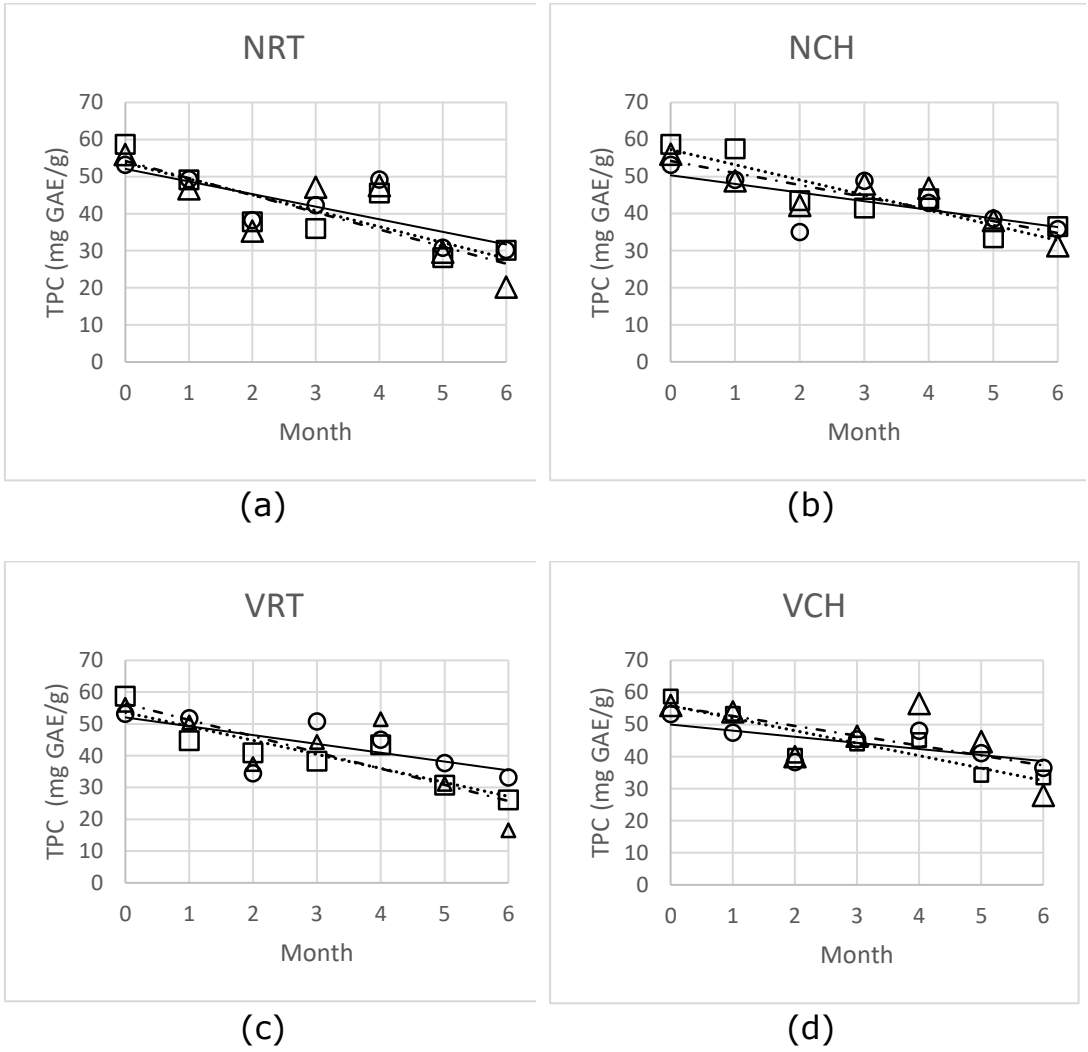


Figure 4.22 Total phenolic content (TPC) for dried lemon myrtle leaves after drying and during storage at a one-month interval (○: HPD; □: OD; △: VD)

Among all storage conditions and drying types, the highest TPC retention after 6 months storage was vacuum-packed of HPD sample stored at chilled condition (VCH), while the lowest TPC retention was VD samples that were vacuum-packed and stored in room

temperature (VRT). Regardless of drying methods, samples stored at lower temperature tend to retain higher TPC values compared to room temperature storage. The results indicate that temperature of the storage affects the TPC retention during storage. This is due to chilled condition that is not conducive for enzymatic reactions that can cause the degradation of biochemical content of herbs. Mediani et al. (2014) compared air-dried and freeze-dried *Cosmos caudatus*' stored at low (-20°) and high temperature (room temperature) and reported that low temperature storage could retain higher TPC than storage at room temperature. This is because higher temperature (ie. room temperature) can promote enzymatic reactions and consequently results in the degradation of bioactive compounds. Phahom et al. (2017) also reported the same findings that the TPC values greatly decreased at higher temperature storage of dried *Thunbergia laurifolia* after 180 days of storage.

Figure 4.23 shows the impact of drying method on TPC retention after 6 months in different storage conditions of LML subjected to oven-, vacuum- and heat pump drying. The final TPC content of all samples stored at different conditions are in small range for HPD samples from 30.19 to 36.49 mg GAE/g sample, compared to VD that had very large range from 16.45 to 31.32 mg GAE/g sample. From Figure 4.23, it can also be seen that leaves stored in all types of conditions have no significant difference of TPC among the HPD

samples. However, there is a significant difference of final TPC for OD and VD samples in all storage conditions. Both OD and VD samples stored in VRT had significantly lower TPC as compared to other storage conditions. The significant difference (p<0.05) in OD and VD samples indicated that storage conditions impacted the final TPC values of the dried samples.

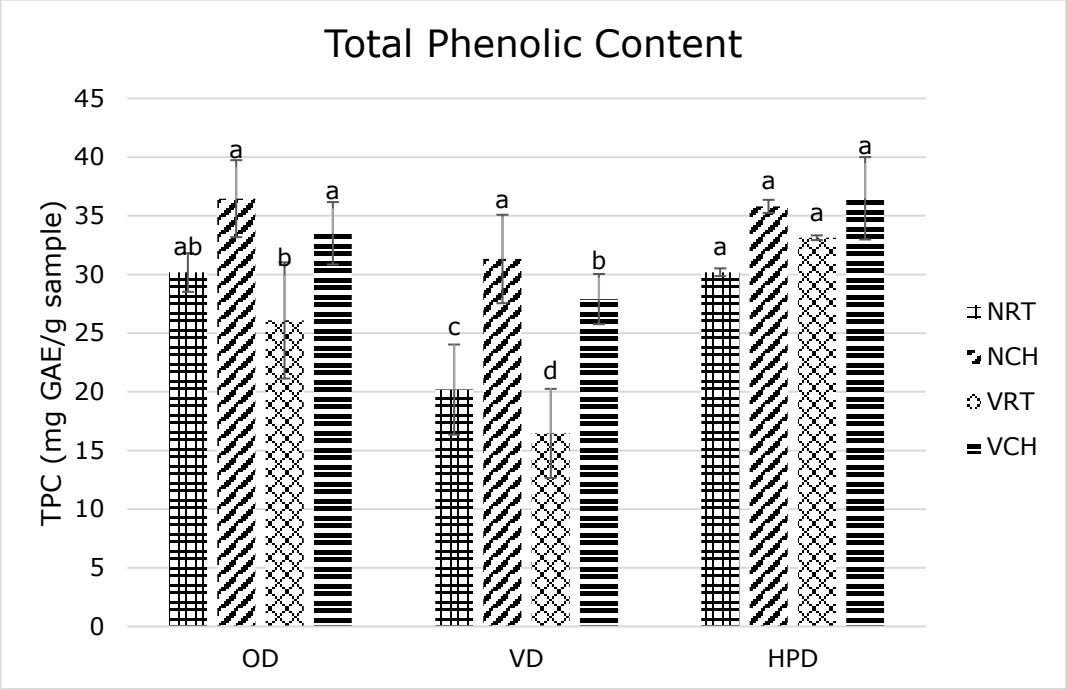


Figure 4.23 Impact of drying methods on TPC retention after 6 months in different storage conditions. Vertical bars indicate standard deviation and values marked by the same letter are not significantly different (p>0.05).

Lower TPC retention for OD and VD samples stored in VRT condition are probably due to injuries of the samples as vacuum is applied for sample packaging. Vacuum packaging might cause the structure of

LML cells to explode that injured the leaves (Janowicz and Lenart, 2018). The injuries might cause the modification in the chemical structure of the polyphenols in vacuum packed samples and thus lower the TPC values. However, higher TPC retention was observed in dried leaves stored at lower temperature and vacuum-packed condition (VCH) for HPD samples. The variation of TPC reduction from different drying methods was influenced by the preservation of the cell structure that are better for HPD samples. Moreover, compared to convective dried product, the microstructure of HPD product showed fuller and more regular shape of cell structure (Thamkaew et al., 2020). In general, HPD samples can give more uniform final TPC after 6 months storage in all storage conditions. The vacuum packed with lower storage temperature (VCH) is the most effective storage method in maintaining good amount of TPC in the HPD leaves, as it gives relatively higher TPC values after 180 days. This result is in agreement with a report by Araújo et al. (2017) that lower reduction of TPC was obtained when vacuum packaging was employed in dried galega kale after 20 weeks of storage.

The radical scavenging assay is the most widely used analysis in evaluating the activity of antioxidant in functional product extract. Figure 4.24 shows the DPPH Radical scavenging activity (%) of dried lemon myrtle leaves after drying and during storage in a one-month

interval for HPD, OD and VD samples. All DPPH values reduced throughout 6-months storage for all samples under the 4 types of storage conditions. Considering the DPPH values after drying (Month 0) as a standard, the highest reduction was shown in VD sample which was in range of 27-50% followed by OD at the range of 16-26% and the least reduction was HPD that was between 15-17%, regardless the storage conditions. The variation of the DPPH reduction might be influenced by the drying operations that cause the fragility of the cell wall and provide great extraction efficiency due to the easy and liberation of metabolites responsible for this bioactivity (Mediani et al., 2014). Since HPD is better at maintaining the structure of cells in leaves (Klungboonkrong et al., 2018), this explains the least reduction of DPPH value from HPD samples.

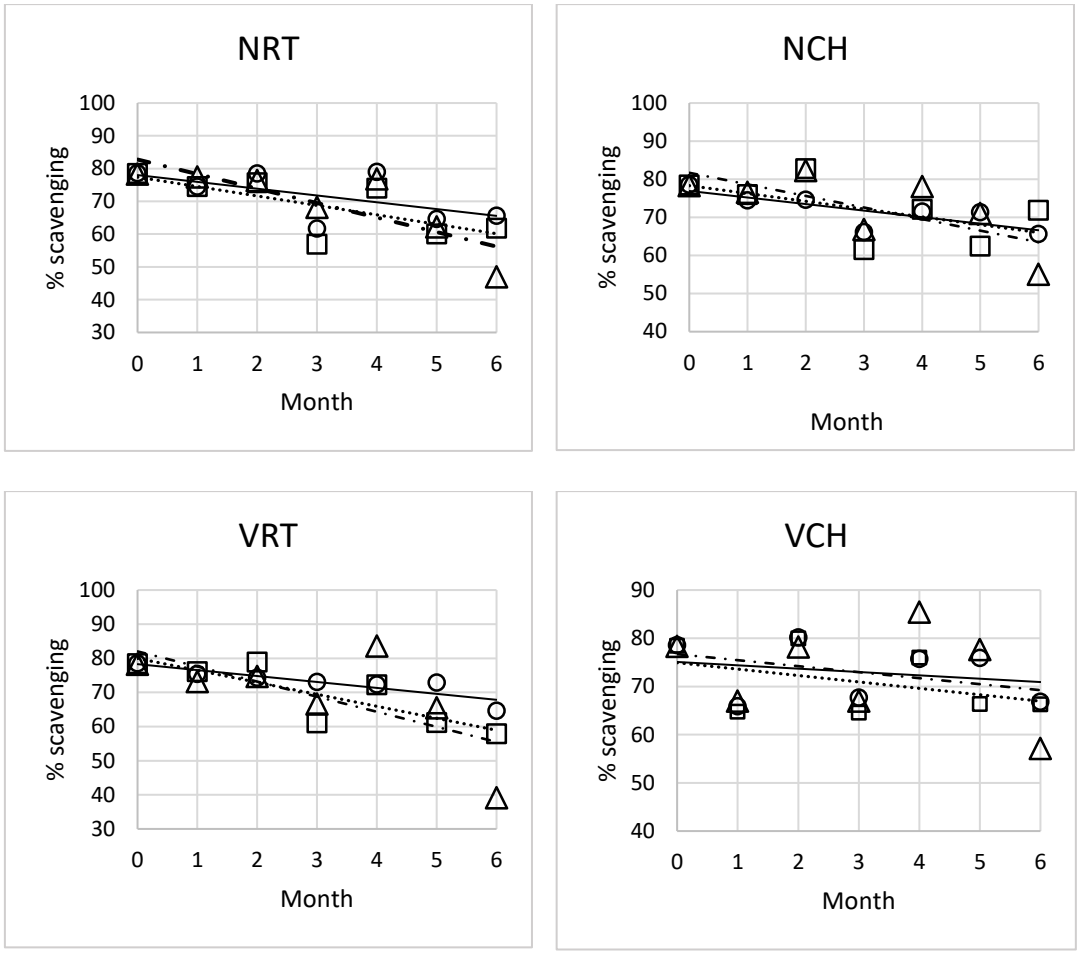


Figure 4.24: DPPH Radical scavenging activity (%) for dried lemon myrtle leaves after drying and during storage at a one-month interval (○: HPD; □: OD; △: VD)

Figure 4.25 shows the impact of drying method on DPPH retention after 6 months storage of different lemon myrtle leaves. The final DPPH value of all samples stored under different conditions was in the small range for HPD and OD samples which were from 64.65 to 66.88% and 57.96 to 66.31%, respectively. Whereas, VD samples gave larger range of final DPPH from 39.19 to 57.17%. From Figure 4.25, it can also be seen that HPD and OD leaves have no significant difference of DPPH among all drying conditions. However, there is a

significant difference in the final DPPH values for VD samples. From VD sample, VRT resulted in significantly (p<0.05) lower DPPH value compared to other storage conditions. The significant difference in VD sample indicated the lower availability of antioxidant to neutralise the DPPH radicals especially from vacuum-packed stored at room temperature storage (VRT) sample. This is explained by the lower DPPH value determined after 6 months of storage that correlated to the lower antioxidant present in the extracted VD samples.

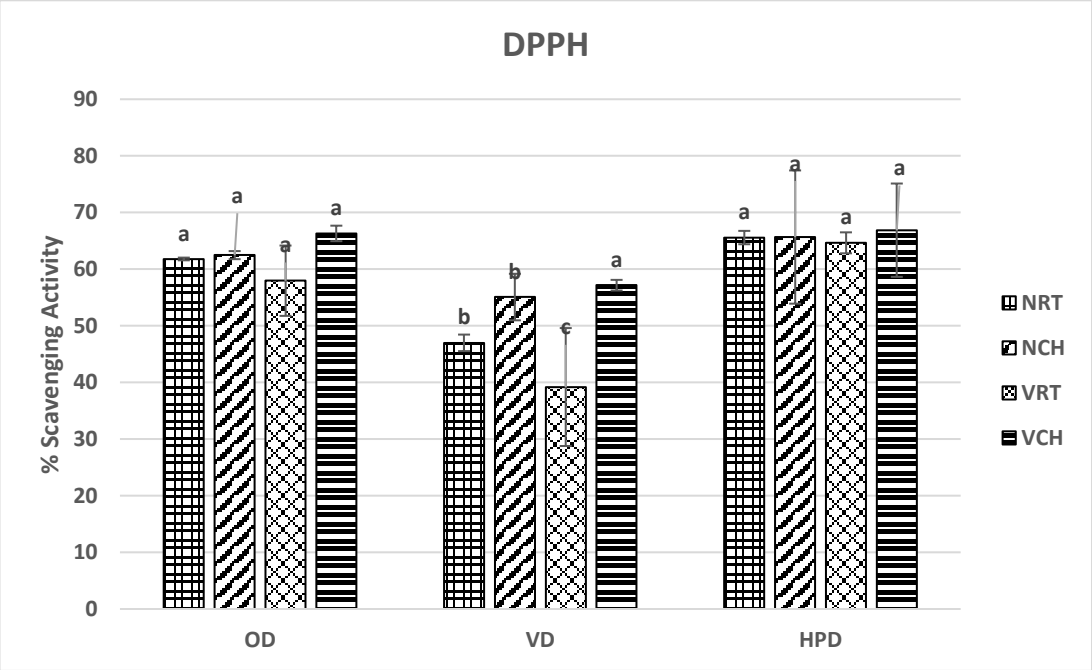


Figure 4.25 Impact of storage conditions on DPPH for HPD, OD and VD samples after 6 months storage. Vertical bars indicate standard deviation and values marked by the same letter are not significantly different (p>0.05).

The FRAP is expressed as mg Ascorbic acid equivalent (AAE) per g sample. Figure 4.26 shows the FRAP variation during the storage

period at a one-month interval for a duration of 6 months for HPD, OD and VD samples under different storage conditions. Different from DPPH reaction mechanism, FRAP is based on electron transfer reaction (Jemli et al., 2016). The FRAP values for all drying methods showed reduction throughout the storage period. In comparison to the fresh sample, which FRAP value was 88.04 ± 0.15 mg AAE/g sample, the dried LML showed retention values in a range of 25 to 56%, regardless of the drying methods.

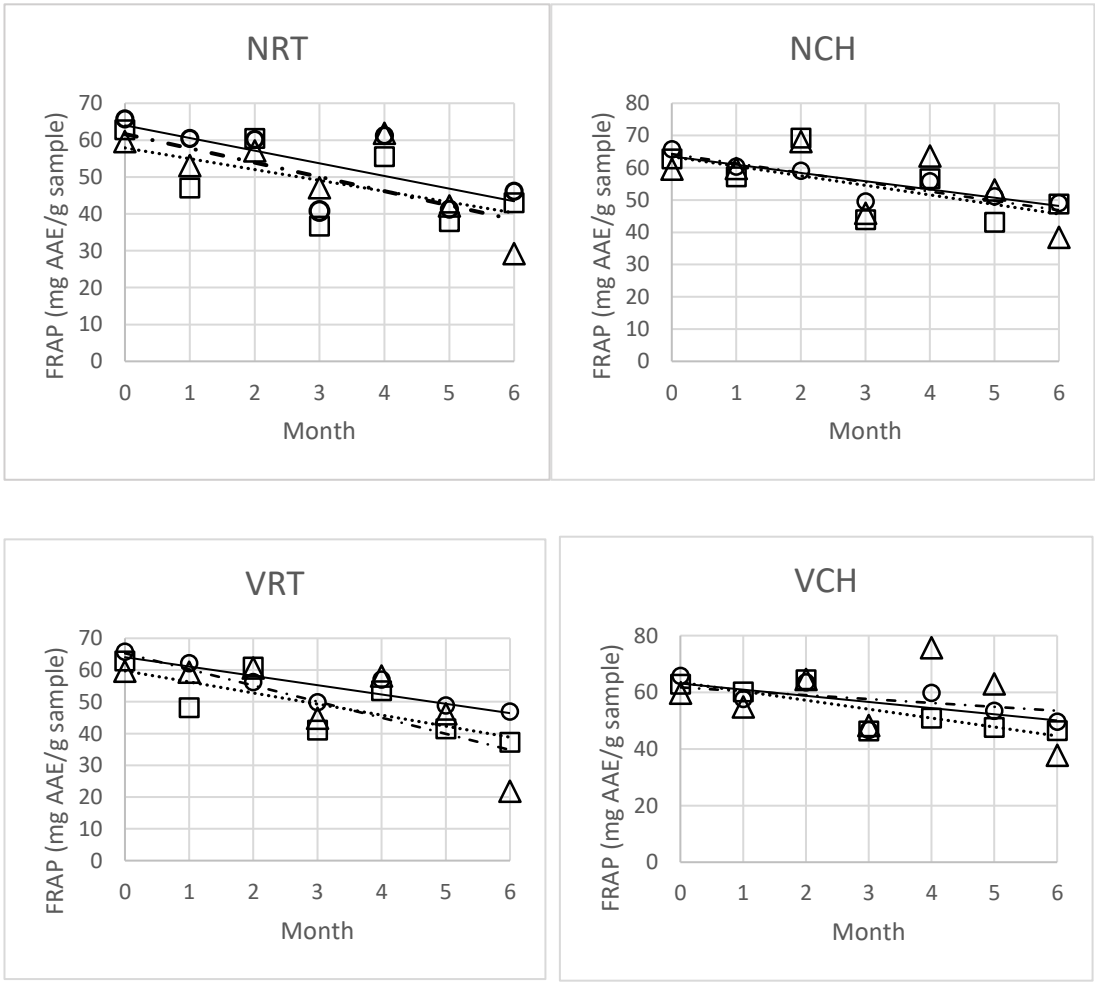


Figure 4.26 Ferric reducing antioxidant power (FRAP) of dried lemon myrtle leaves after drying and during storage at a one-month interval (○: HPD; □: OD; △: VD)

In general, VD resulted in greater FRAP loss for all 4 different storage methods with VRT showing the lowest retention. This might be due to the longer drying time for VD samples that increased the exposure to thermal degradation. Whereas, for HPD samples, the difference between storage conditions is not significant (p>0.05).. This is similar to the findings shown in TPC. Thus, the results indicated that HPD was a drying technique that can give a more stable antioxidant content during storage and by applying vacuum packed and chilled condition (VCH) to the HPD samples, it is beneficial in terms of maintaining the bioactive ingredients during storage.

Figure 4.27 shows the impact of drying method on FRAP retention after 6 months of different storage conditions of lemon myrtle leaves subjected to oven-, vacuum- and heat pump drying. The final FRAP value of all samples stored at different conditions are in the small range for HPD and OD samples which are from 37.23 to 46.35 mg AAE/g sample and 46.16 to 49.52 mg AAE/g sample, respectively. However, VD samples gave larger range of FRAP value between 21.86 to 38.49 mg AAE/g sample. From Figure 4.27, it can also be seen that HPD and OD leaves have no significant difference (p>0.05) in FRAP value among all drying conditions. However, there is a significant difference of final FRAP value for VD samples. Similar to DPPH, VD samples stored in VRT condition resulted in significantly

(p<0.05) lower FRAP value compared to other storage conditions. The significant difference in VD sample indicated the lower availability of antioxidant to act as reducing agent especially from vacuum-packed stored at room temperature storage (VRT) sample.

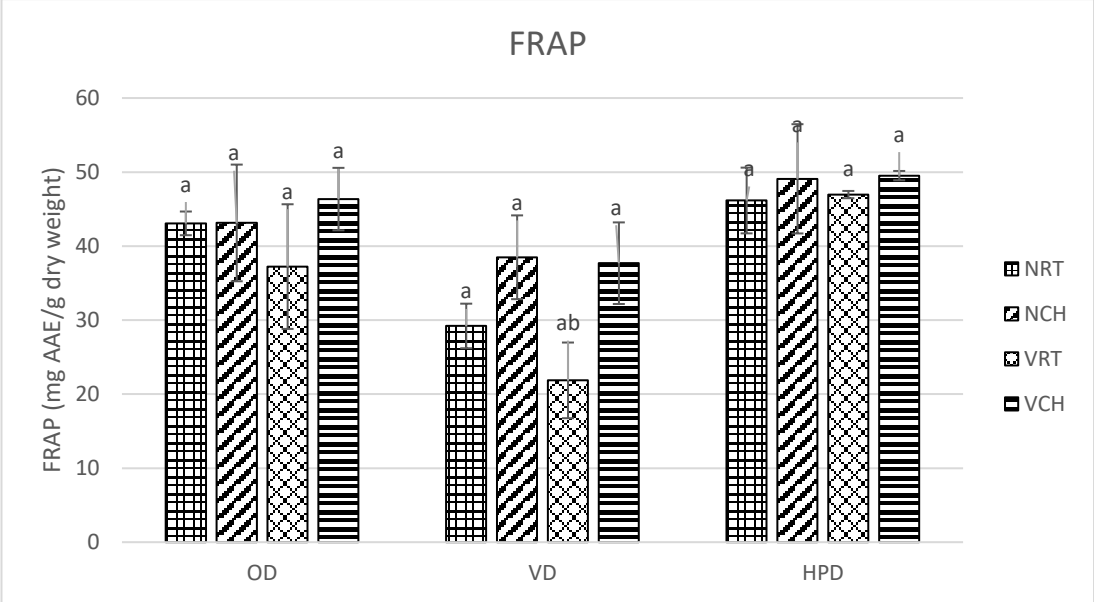


Figure 4.27 Impact of storage conditions on FRAP for HPD, OD and VD samples after 6 months storage. Vertical bars indicate standard deviation and values marked by the same letter are not significantly different (p>0.05).

Table 4.10 shows the mean values of TPC and the significant interaction between drying method and storage conditions on TPC value after 6-months of storage. In Table 4.10, the interaction between the type of dryer and storage conditions is presented to show the influence of these main factors on TPC retention after storage. As shown in Table 4.10, regardless the storage conditions, the highest TPC value was obtained from HPD samples while the

lowest was obtained from VD samples. These results might be due to the better preservation of cell structure of LML during HPD which in turn increase the stability of polyphenols after 6 months storage. Among the storage conditions, storage at chilled conditions (CH) samples showed better retention of TPC values regardless the drying methods. Chilled condition inactivates the Maillard reaction that might lead to the oxidation of polyphenols in LML throughout storage. Table 4.10 also shows the significant interaction ($p<0.05$) between the drying method and storage condition on TPC retention after 6-months storage. Significant interactions between the main factors which are drying methods and storage conditions indicate that content of TPC in leaves stored in different conditions is highly influenced by the types of dryer used.

Referring to Table 4.10, the column on DPPH, it shows no significant interaction ($p>0.05$) between the types of drying and storage conditions. Regardless of any storage conditions, LML dried using HPD and OD had significantly higher content of DPPH as compared to VD. LML stored in NCH and VCH had significantly higher DPPH as compared to NRT and VRT, irrespective of the drying methods. This could be due to the impact of heat treatment which tends to occur at higher temperature, consequently causing enzymatic denaturation by the Maillard reaction which in turn lowers the bioactive ingredients (Mediani et al., 2014). However, by comparing vacuum (V) and without vacuum (N) storage condition, it is found

that vacuum packaging is beneficial when the samples are stored in chilled conditions (VCH) and not in room temperature (VRT). Among all drying methods and storage conditions, HPD stored under VCH gave the highest retention of DPPH while the greatest loss of DPPH value is found in VD VRT samples.

Table 4.10 Effects of types of dryer, storage conditions and its interaction on total phenolic content (TPC) and antioxidant activities (DPPH and FRAP) after 6 months storage.

Factor		TPC (mg GAE/g dry weight)	DPPH (% scavenging)	FRAP (mg AAE/g dry weight)
Types of dryer	**OD**	31.54^{a}	63.98^{a}	43.85^{a}
	VD	24.17^{b}	49.61^{b}	31.81^{b}
	HPD	33.90^{a}	66.29^{a}	46.87^{a}
Storage conditions	**NRT**	27.09^{b}	56.38^{b}	38.06^{b}
	NCH	34.53^{a}	64.94^{a}	45.45^{a}
	VRT	25.23^{b}	53.62^{b}	35.35^{b}
	VCH	32.63^{a}	64.90^{a}	44.52^{a}
Types of dryers, D		**	**	**
Storage condition, S		**	**	**
Dryer*Storage		*	ns	ns

Note: **Significant at 1% probability level, *Significant at 5% probability level, ns: Not significant. Means in each column with the different letters within each factor indicate significant difference at $p<0.05$ level according to Tukey's HSD (Mean $\pm$ S.E, n=3)

Similar to DPPH, FRAP value also shows no significant (p>0.05) interaction between the different drying methods and storage conditions (Table 4.10). LML dried using HPD and stored at any storage condition has significantly higher content of FRAP as compared to LML dried using OD and VD. Leaves stored in chilled condition (CH) have significantly higher FRAP as compared to leaves stored in room temperature (RT), irrespective of the drying method. This is in line with Korus (2011) who reported that better retention of antioxidants was observed in dried kale leaves stored at cold-store temperature after 12 months of storage. In this study, the highest retention of FRAP was found in samples subjected to chilled storage condition for all drying methods. However, for HPD and OD samples, stability of the sample can be improved by applying vacuum packaging in combination with storage at chilled condition to reduce the FRAP value loss throughout storage period.

Therefore, it can be concluded that storing dried sample in chilled condition would reduce the degradation of phenolic and antioxidant compounds, thus retain high TPC, FRAP and DPPH content even after 180 days of storage. Furthermore, HPD could serves as a better drying method as the TPC, FRAP and DPPH are highly retained after 6 months storage. High retention of bioactive compounds is an important indication of the product stability as a guarantee of its functionality throughout storage.

4.3.2.2 Colour assessment

After drying, long term storage can lead to loss of colour, nutrients and bioactive compounds (Phahom et al., 2017). The retention of colour is usually associated with the quality parameters (such as biochemical content) for green-leaf products. The colour parameters (L^*, a^*, b^*) after 6 months of storage for HPD, VD and OD subjected to various storage conditions are shown in Table 4.11. In addition, colour changes (chroma, hue angle and browning index) after 6 months storage are also presented in Table 4.11. In general, the values of colour parameters varied for different drying methods and storage conditions. Table 4.11 shows that different drying methods and storage temperature resulted in significant difference ($p<0.05$) between colour parameters of LML.

Table 4.11 Effects of types of dryers, storage conditions and its interaction on colour assessment, greenness (*a), lightness (*L**), yellowness (*b**), Chroma, hue angle and browning index (BI) after 6 months storage.**

Factor		*a**	*L**	*b**	Chroma	Hue Angle (°)	Browning Index (BI)
Types of dryer	**OD**	-1.64 ± 0.17^{a}	48.21 ± 0.75^{a}	26.42 ± 0.29^{c}	26.66 ± 0.18^{b}	178.53 ± 0.01^{a}	78.99 ± 1.22^{c}
	VD	-1.69 ± 0.18^{a}	46.39 ± 0.56^{b}	27.41 ± 0.31^{a}	26.86 ± 0.19^{ab}	178.49 ± 0.004^{b}	88.06 ± 1.72^{a}
	HPD	-1.77 ± 0.24^{a}	47.67 ± 0.68^{a}	26.85 ± 0.32^{b}	27.12 ± 0.20^{a}	178.53 ± 0.01^{a}	76.91 ± 0.98^{b}
Storage conditions	**NRT**	-1.07 ± 0.1^{a}	45.11 ± 0.21^{b}	26.00 ± 0.26^{b}	26.42 ± 0.23^{b}	178.50 ± 0.23^{c}	82.71 ± 1.68^{a}
	NCH	-1.69 ± 0.20^{b}	49.50 ± 0.38^{a}	27.72 ± 0.14^{a}	27.3 ± 0.13^{a}	178.51 ± 0.13^{b}	80.48 ± 2.07^{c}
	VRT	-1.55 ± 0.11^{b}	45.59 ± 0.41^{b}	25.91 ± 0.15^{b}	26.38 ± 0.22^{b}	178.52 ± 0.22^{b}	81.53 ± 1.84^{b}
	VCH	-2.49 ± 0.16^{c}	49.49 ± 0.48^{a}	27.96 ± 0.22^{a}	27.44 ± 0.16^{a}	178.53 ± 0.16^{a}	80.58 ± 2.13^{c}
Types of dryers, D		ns	**	**	*	**	**
Storage condition, S		**	**	**	**	**	*
Dryer*Storage condition		*	ns	ns	ns	ns	ns

Note: **Significant at 1% probability level, *Significant at 5% probability level, ns: Not significant. Means in each column with the different letters within each factor indicate significant difference at $p<0.05$ level according to Tukey's HSD (Mean ± S.E, n=3)

Greenness of a sample is represented by the CIE *a** value in the negative (greenness) to positive (redness) axis. From Table 4.11, in terms of drying methods, the lowest *a** value (more negative) was obtained from HPD sample followed by VD and OD. Whereas, VCH condition gave the lowest *a** values after 6 months of storage irrespective of the drying method. The lower *a** value indicates higher green colour intensity of the sample. From Table 4.11, the interaction between drying methods and storage conditions is significant ($p<0.05$) on the *a** value after 6-months of storage. Significant interactions between the storage conditions and drying methods indicates that the *a** values in dried LML stored in different conditions is highly influenced by storage conditions.

As shown in Figure 4.28, storage at room temperature (NRT) resulted in the higher *a** values for all three types of drying methods. The higher *a** values indicates that the samples' colour is less green and closer to redness. The variation of *a** value subjected to different storage conditions might be caused by the oxidation of chlorophyll in packaging in the presence of oxygen at higher storage temperature. Hence, as oxidation occurs during storage in non-vacuum packed at room temperature, the magnesium atom of the chlorophyll is replaced by two hydrogen atoms, thus, change the sample's colour from bright green (more negative *a** value) to

darker green-brownish (more positive *a** value). This result is also in line with Phahom et al. (2017) who reported that significant change of green colour from bright green to olive green of dried *Thunbergia laurifolia* at higher storage temperature and non-laminated packaging.

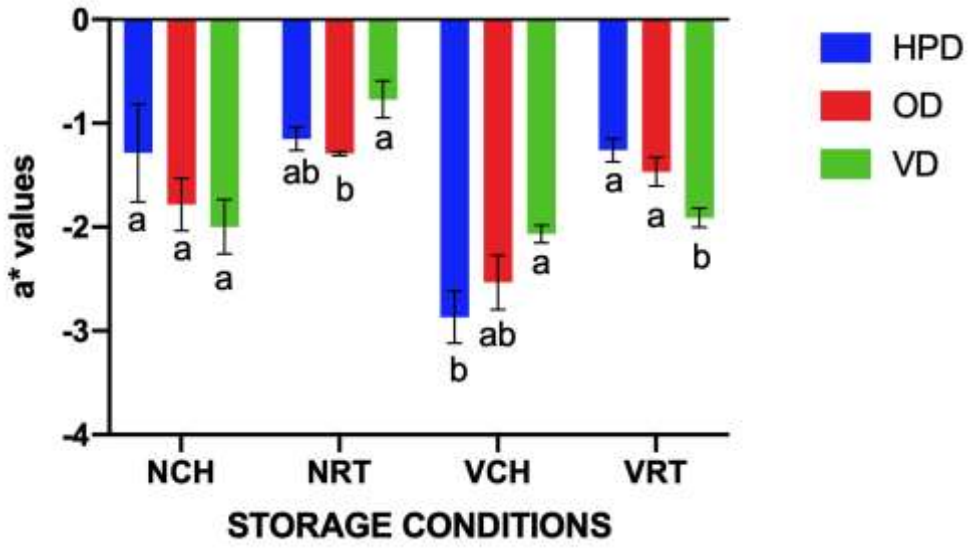


Figure 4.28 Effect of storage condition on *a values of dried LML that were subjected to HPD, OD and VD. Means in each graph with the different letters indicate significant differences at $p<0.05$ level according to Tukey's HSD. (Mean ± S.E; n=3)**

Lightness of a sample is represented by *L** value in the scale from 0 to 100. Higher *L** value indicates the lighter the colour of the sample, while lower *L** value represents the darker colour of the sample. The value of the lightness after the 6 month storage is shown in Table 4.11. The *L** value for each dryer of each sample was not influenced by the storage conditions as there was no significant interaction

(p>0.05) between these two factors, namely drying methods and storage conditions. LML dried by HPD and OD gave higher value of L^* compared to VD samples, regardless of the storage conditions. This is because VD tends to take longer time for the drying process to be completed. Therefore, it results in a significant colour change and causes the VD sample's colour to be darker.

Furthermore, storage in chilled condition gave higher L^* value than room temperature storage on both vacuum and non-vacuum packaging irrespective of the drying method. After 6 months storage at room temperature, colour of the sample was darker (indicated by lower L^* value) due to the oxidation of chlorophyll that led to loss of magnesium atom contributing to colour change from brighter to darker colour.

The b^* value indicates the yellowness or blueness of a sample. The higher b^* value represents the yellowness while lower b^* value represents blueness of the sample's colour. Table 4.11 shows that there are no interactions between the storage conditions and drying method on b^* value. VD samples showed higher value of b^* independent to the storage conditions. Storage at chilled conditions also resulted in higher b^* value compared to room temperature storage regardless the drying method. Higher b^* value indicates the yellowish colour that leads to brighter colour of the sample.

Storage conditions show positive influence on *a**, *L** and *b**. The lower the temperature, the lower the changes. This might be due to the chilled condition that can inhibit the enzymatic reaction and thus preserve chlorophyll that correlates to the colour of the sample. Chlorophyll in leaves can undergo oxidation, hydrolysis and isomerisation that lead to colour changes. This result is similar to the findings reported by Mudau et al. (2018) where the nature of spinach leaves stored under normal air packaging showed significantly lower *a** values than samples stored in modified air. Therefore, storage at chilled condition is effective in minimising colour changes especially value of greenness (*a**) during storage.

Table 4.11 also depicts the chroma, hue angle and browning index of dried samples after the 6-month storage. Chroma is a polar coordinate that indicate the dull or vivid the product's colour that ranging from 0 to 60 (Phahom et al., 2017). Table 4.11 shows the colour intensity chroma of the sample after storage for each storage conditions and drying methods. Generally, values of chroma were highly influenced ($p < 0.01$) by the storage conditions. Drying by using HPD and storage in vacuum-packed at chilled condition (VCH) gave higher chroma value compared to other drying methods and storage conditions. Higher chroma values which indicates more

intense (pure) colour were observed from samples subjected to chilled condition storage especially on VCH condition.

Hue angle is the degree to which the appearance colour of fruits or vegetables can be said to be similar to or different from the primary colours. An angle that is smaller than 180° signifies green hues and lesser value indicates a decrease in green colour of the sample. The hue angle value was lower in higher storage temperature (RT) in comparison to chilled storage, regardless the drying methods. This result is influenced by the value of *a** as hue angle is the result of arctan value of *b*/a**. Higher value in *a** gives a lower hue angle which indicate less degree of green colour is obtained at the end of storage (after 180 days). This result is also in agreement with the report published by Phahom et al. (2017) who stated the hue angle decreased at higher storage temperature after 180 days of storage of microwave heat pump dehumidified drying as indication of less green colour product of *Thunbergia laurifolia* leaves.

Browning index is an important parameter to define browning in the sample. BI represents the purity of brown colour and the calculated value are shown in Table 4.11. Storage at chilled condition gave lower BI compared to room temperature storage with higher value in non-vacuum packed than vacuum-packed storage, irrespective of the dryer used. This is in agreement with the findings of Yao et al.

(2020) and Alagoz et al. (2015). Both reported that the negative correlations between BI values and formation of brown colour. Samples stored at lower temperature also resulted in lower values of BI compared to room temperature storage. On the other hand, packaging condition did influence BI of the samples. Non-vacuum packaging contributes to higher brown pigment formation that cause by the oxidation of the pigments in presence of oxygen. This result is in agreement with Araújo et al. (2017) who reported that higher values of BI resulted from non-vacuum packaging is probably because of simultaneous and frequent Maillard reaction that occurs which is promoted by the presence of oxygen in non-vacuum packaging of dried kale.

The lower temperature storage also shows lower BI, higher greenness (lower *a**), hue angle and chroma values as well as the TPC and antioxidant activities. These results show the interaction between storage temperature on biochemical activities of dried samples during storage. These results indicate that colour change has direct correlation with the retention of bio-active ingredients in the dried product, where higher colour change indicates lower retention of bio-active ingredients which also means degradation of the ingredients. Phahom et al. (2017) also reported that storage at lower temperature gave greater value of TPC and FRAP in microwave heat pump drying of *Thurnbergia laurifolia.* In conclusion, VCH is

more suitable for storage of LML especially for HPD samples as it can produce in lower *a** value (greener) and BI with higher *L** (brighter) and chroma value. Thus, this storage condition was proven to preserve the colour better while extending the shelf life of dehydrated LML.

4.4 Non-destructive analysis of ground lemon myrtle leaves

4.4.1 Near-infrared spectroscopy

Final moisture content by oven method determination as mentioned in 3.4.2 for each drying method were 11.10 ± 0.093%, 9.88 ± 0.19% and 10.30 ± 0.11% for oven-, vacuum- and heat pump dried samples, respectively. Figure 4.29 shows the NIR relative reflectance (RR) spectra for different dried lemon myrtle leaves subjected to oven drying (OD), vacuum drying (VD) and heat pump drying (HPD). The average spectra of each drying method are shown in Figure 4.29. It could be seen that the average spectra of different drying methods have similar shapes but, the amplitudes are different as for different final moisture content of each drying method. From Figure 4.29, it is shown that the RR is increasing with the increase in different dried lemon myrtle leaves moisture content. The lowest final moisture content was determined in VD sample and the relative reflectance plot line for this sample was the lowest in Figure 4.29, followed by HPD and OD samples. This trend was verified by the same sequence of actual moisture content with VD as the lowest and followed by the moisture value of HPD and OD at 10.3 and 11.1%, respectively.

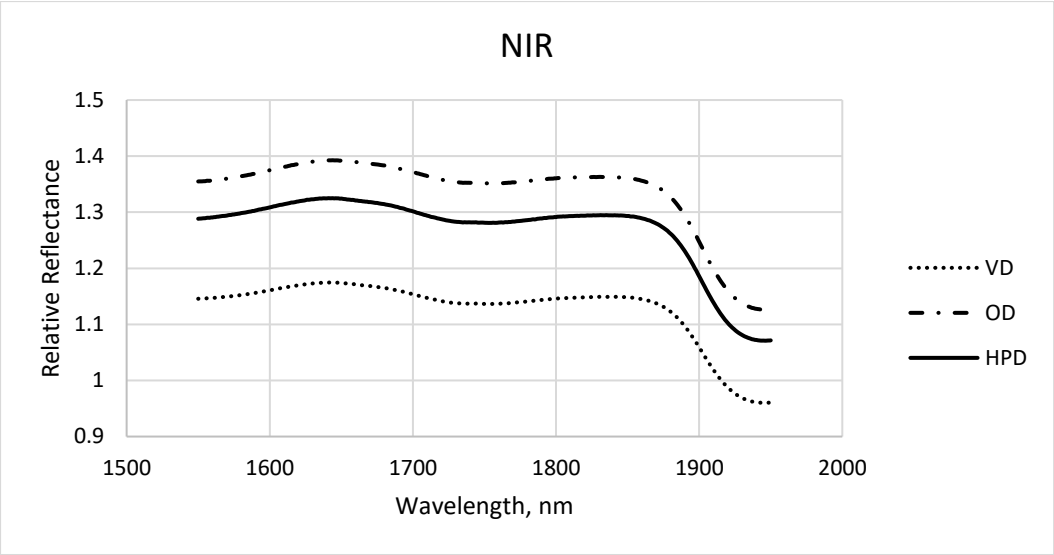
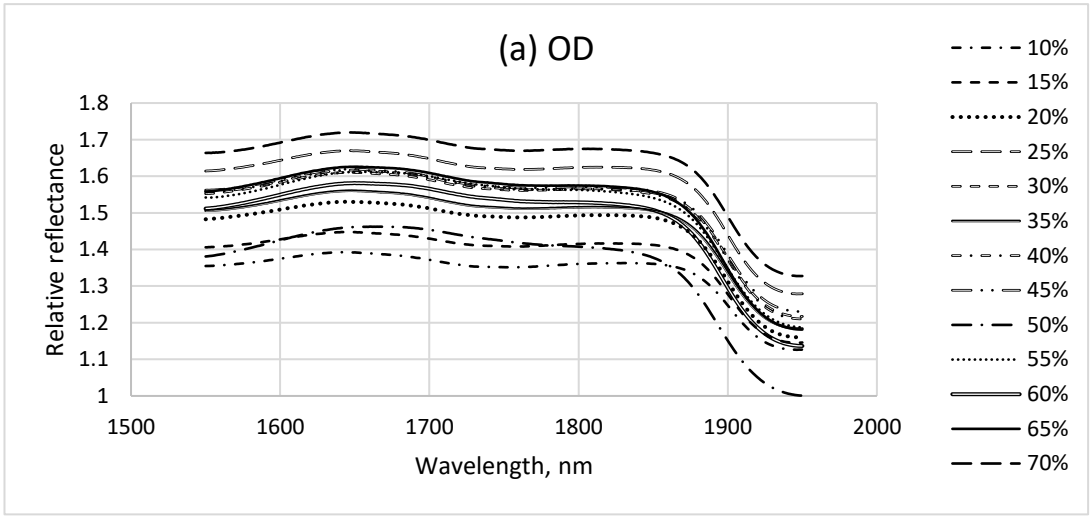
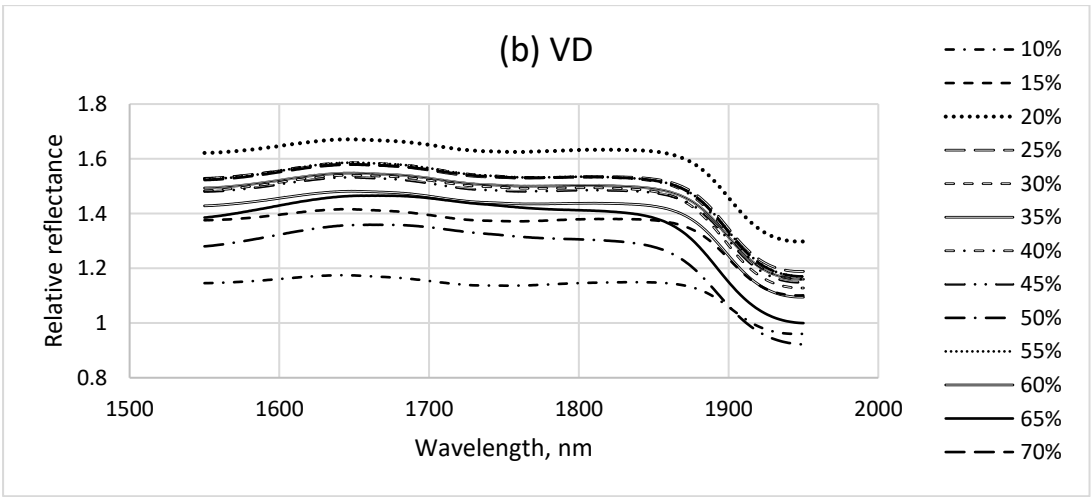


Figure 4.29 The NIRS spectra of the dried lemon myrtle leaves sample for different drying methods (Relative reflectance versus wavelength)

The variation of moisture content has an impact on the intensity of the NIR absorption spectra. This might be due to the modification of reflectance indices due to water presence in the sample. NIR light is sensitive to the ions, solutes and hydrogen bonds. Therefore, the more water content, the more light is absorbed by the sample. More water present in the sample gives more modification of the reflectance indices that results in higher intensity of NIR absorption from sample in higher moisture content. This trend is also in line as reported by Shimbori and Kurata (2017) on hardwood leaves; Sinija and Mishra (2011) on green tea; Cai and Corke (2001) on *Amaranthus* plant. All of these previous studies mentioned that the intensity of the spectra increased with increasing in water content of respective leaves samples.

The leaves were rehydrated to known moisture level to develop calibration model for the sensor. The LML were rehydrated from 15 to 70% of moisture content for every 5% increment for 13 moisture levels including original dried LML. Figure 4.30 shows the plot of average relative reflectance of NIR for different moisture level from 10% until 70%. The profile of the thirteen plots shown in Figure 4.30 has similar shape with the plot in Figure 4.29 but at a different intensity.





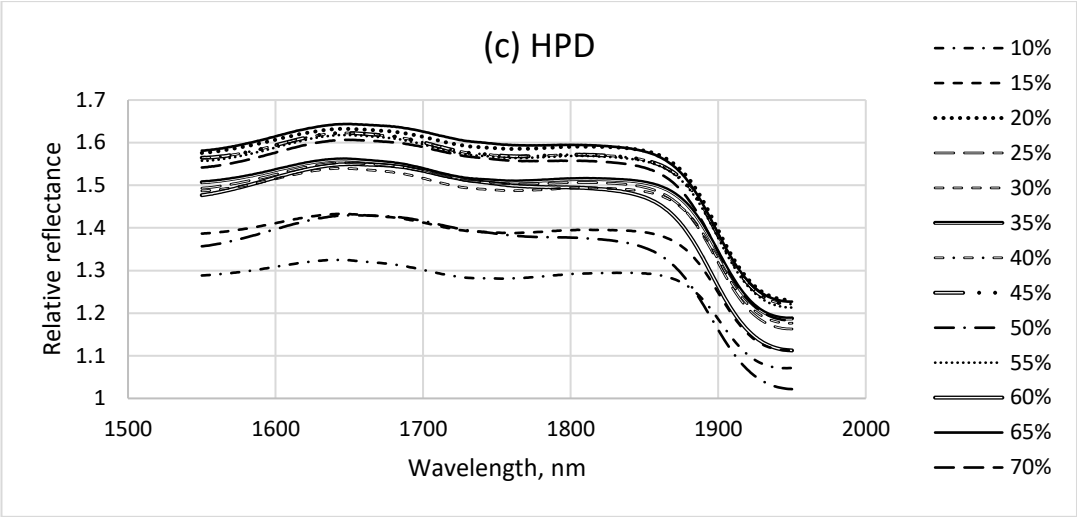


Figure 4.30 Plot of spectral data on different moisture level (NIR) (a) Vacuum drying; (b) Oven drying and (c) Heat pump drying

The intensity of the NIR relative reflectance increased with increasing of water content for samples obtained from the three drying methods. This might be due to the influence of water absorption bands to the hydrogen bonds of organic polymers. The light absorbance energy is sensitive to the ion strength of the hydrogen bonds. Therefore, the more water present in the sample, the more modification of the refraction indices that thus increase the intensity of the relative reflectance of the sample's absorbance. This is in agreement with Cai and Corke (2001) who reported that the different intensities resulted from plotting different moisture content of *Amaranthus* plant as the fresh leaves with higher moisture on top of the plot and dried leaves on the bottom of the plot. Despite the lack of distinctive lines for each moisture content, it has been shown

the PLSR can extract relevant information for quantitative determinations (McShane and Cote, 1998).

4.4.2 Dielectric spectroscopy

Dielectric properties of some agricultural products might be useful for rapid nondestructive quality determination (Nelson, 1992). Table 4.12 shows the bandwidth, center frequency, *Q* and loss tangent values of different lemon myrtle leaves drying. *Q* value is the dimensionless quality factor that describes the energy balance between the resonator. The Q value of a resonant cavity is defined as the ratio of the energy stored in the oscillating resonator to the energy dissipated per cycle by damping process. The results of dielectric properties were obtained from the dielectric properties measurement. From Table 4.12, there is not much difference in values of bandwidth, center frequency and loss tangent for dried samples obtained from the three drying methods. Only the value of the quality factor (*Q*) resulted in larger range which is in between 688 to 703.52 with OD as the lowest and HPD as the highest value.

Table 4.12 Dielectric properties of vacuum-, oven- and heat pump dried lemon myrtle leaves

Drying method	Bandwidth	Center frequency (MHz)	Quality factor, Q	Loss tangent
VD	1.35 ± 0.02	939.12 ± 0.49	694.84 ± 14.7	-42.74 ± 0.13
OD	1.37 ± 0.01	939.18 ± 0.55	688.03 ± 4.68	-43.09 ± 0.04
HPD	1.34 ± 0.01	938.98 ± 0.04	703.52 ± 2.23	-42.95 ± 0.06

Figure 4.31 shows the dielectric measurement versus frequency for the dielectric spectroscopy from VD, OD and HPD samples. The plots show that as the moisture content increased, the plot shifted to the left. As the moisture content of the rehydrated leaves increased from 8 to 11%, the plots shifted to left of the graph. This result might be due to the degree of changes detected in terms of the resonant patterns introduced by the moisture content in LML subjected to oven-, vacuum- and heat pump drying. These changes are correlated with the high (fresh LML) and low (dried LML) phases of total complex permittivity. Higher total complex permittivity is detected in higher water content, whereas lower moisture content is indicated by the low complex permittivity. The change in complex permittivity shows the variation of the water volume fraction in the sample and it can be indicated by the frequency shift inside the cavity.

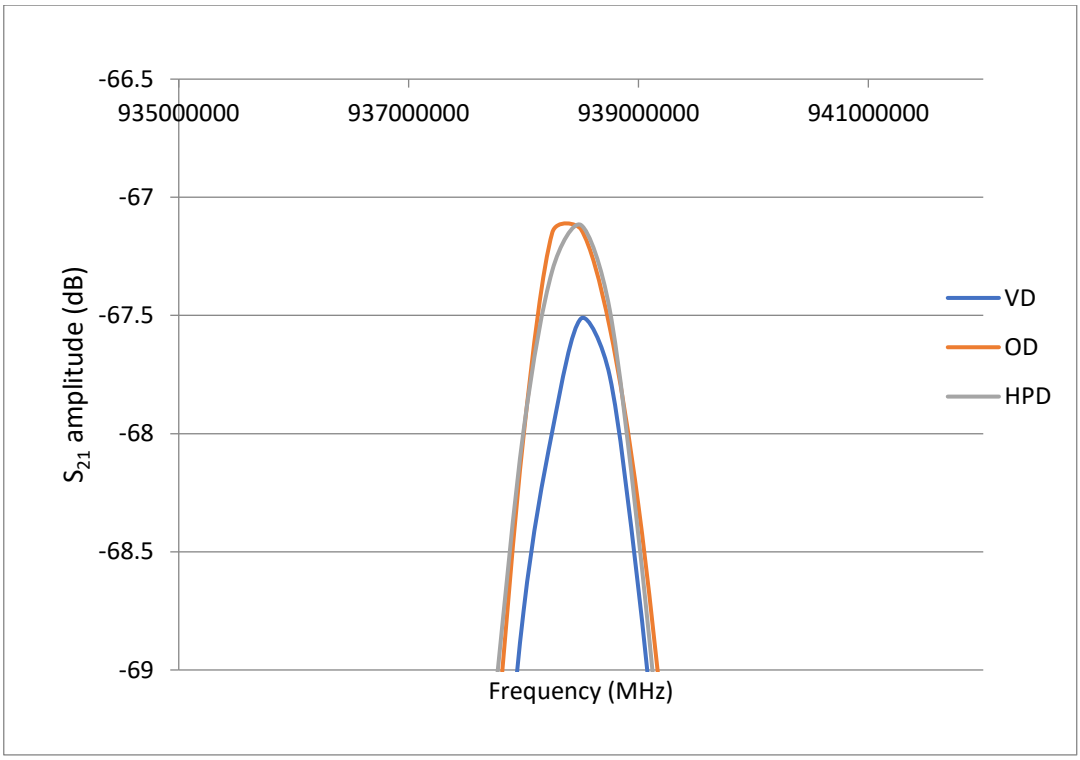


Figure 4.31 Resonant frequency of dried lemon myrtle leaves subjected to oven dried (OD), vacuum dried (VD) and heat pump dried (HPD)

Figure 4.32 displays the resonant peaks of TM010 mode with different moisture levels of dried LML subjected to oven-, vacuum- and heat pump drying. According to the figures, as the moisture level increases, the resonant frequencies continuously shifted from approximately 939.25 MHz to 932.5 and the resonant frequencies decreases by approximately 10-11 dB for OD, VD and HPD samples. This suggests that the frequency shift can be utilised to determine the moisture content in dried LML through the calibration process developed from different known moisture levels. This is also in agreement with Yuan et al. (2019) who reported that as the volumetric water fraction increased, the resonant frequencies shift

from 890 to 880 MHz and decreased the amplitude of resonant peaks for approximately 1 dB in oil-water mixtures. In order to describe the relative frequency shift due to various moisture levels, frequency shift, a ratio was calculated. This gives the ratio of resonant frequency of sample and empty sample holder and as shown in Figure 4.33.

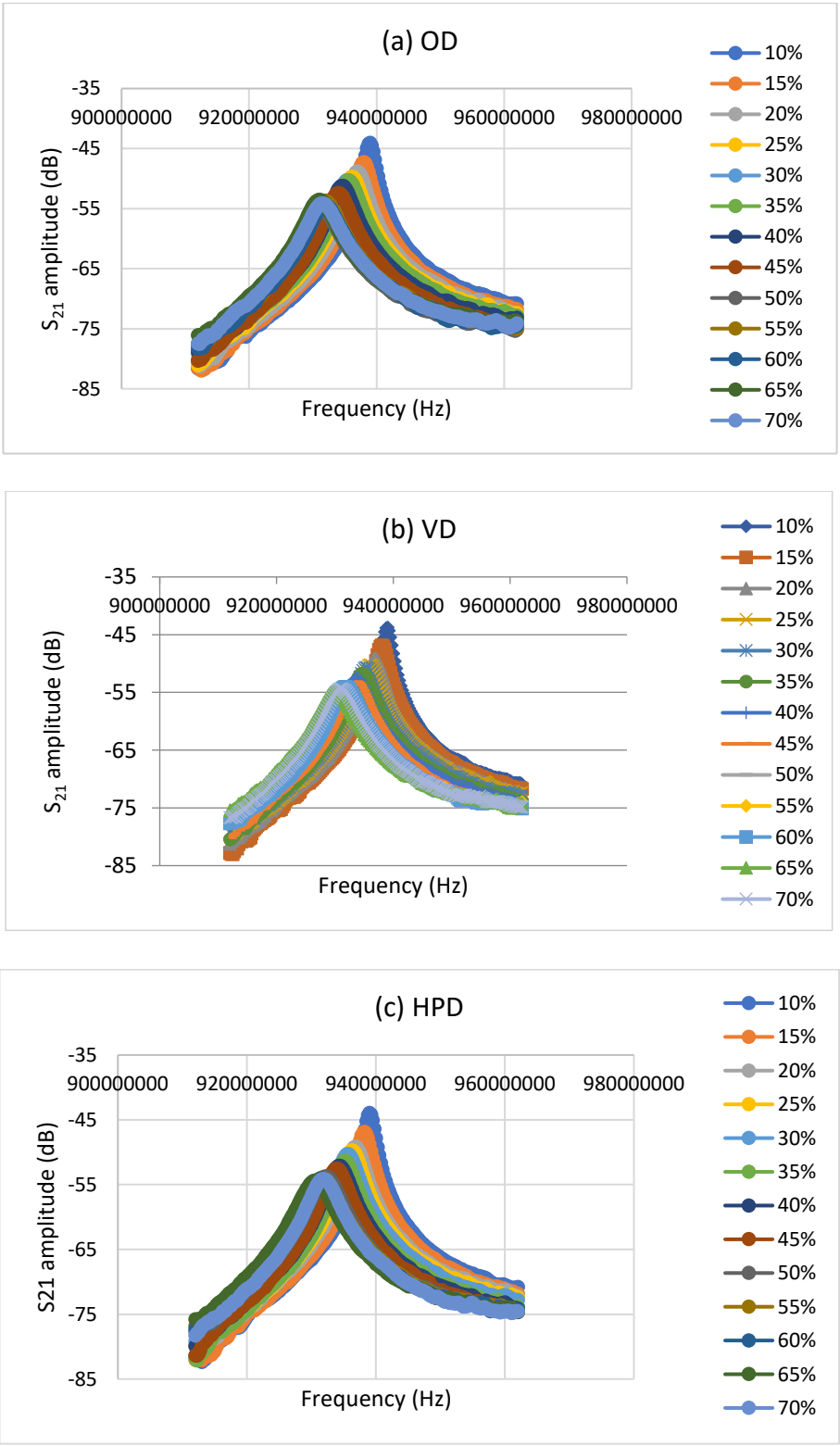


Figure 4.32 Resonant peaks of different moisture level of dried lemon myrtle leaves subjected to (a)oven dried (OD); (b)vacuum dried (VD); (c)heat pump drying (HPD)

Figure 4.33 shows the frequency shift value versus moisture content for the dielectric spectroscopy from VD, OD and HPD samples. When the LML samples were rehydrated, the frequency shifts were also measured and plot against the different moisture content for OD, VD and HPD samples. The samples were distributed closely to the regression line, which shows an excellent spectral analysis performance. The highest coefficient of determination was determined from the linear plot which was in the range of 0.924 to 0.9513. The highest R^2 was obtained from HPD followed by OD and VD samples. The high values of R^2 indicated that dielectric measurements applied was sensitive in detection of moisture content from range of 10 to 70% d.b. From Figure 4.33, the frequency shift increases with the increase of moisture content for all drying methods. The variation in moisture content is indicated by the frequency shift. Higher moisture content present in the sample results in more changes between the high (fresh LML) and low phase (dried LML) of total permittivity. Therefore, the more changes in high and low phase permittivity gives higher frequency shift value as plotted in Figure 4.33. This result is in agreement with Zhou et al. (2008) that reported on increasing moisture content resulted in increase of the resonance frequency shift. They concluded that the resonance frequency shift could provide a mean value in non-destructing testing of the water content of concrete.

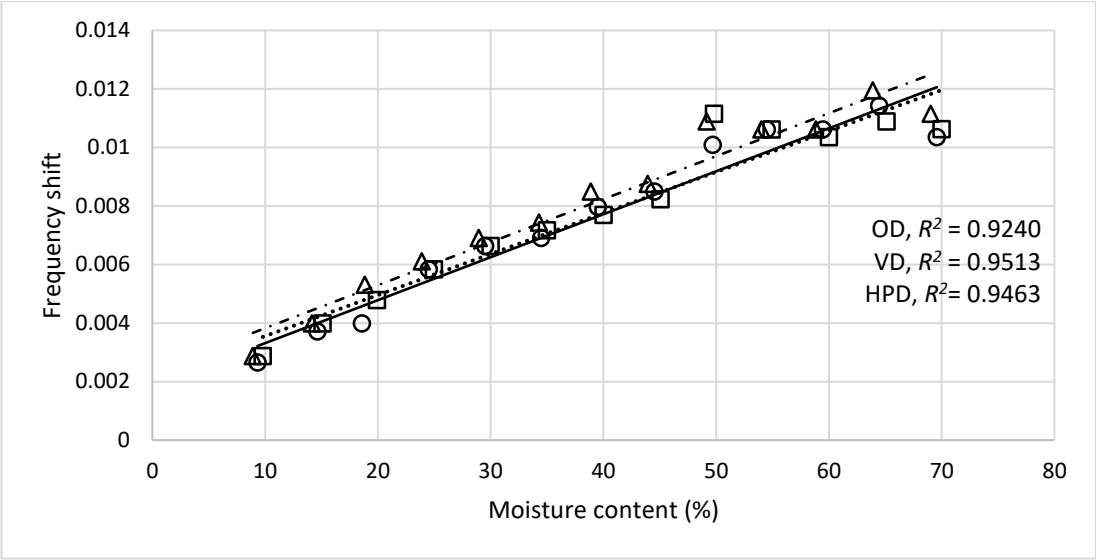


Figure 4.33 Frequency shift versus moisture content (○: HPD; □: OD; △: VD) for the TM010 resonant mode

4.4.3 Moisture content prediction models

4.4.3.1 Models of full features of NIR spectral data and dielectric measurement

Table 4.13 shows the performance of NIR models over the full NIR range (1550 – 1950 nm) and dielectric measurement (912 - 962 MHz) values for all 13 moisture levels for oven-, vacuum- and heat pump drying samples. NIR models were developed and validated using the same test and validation sample sets as the dielectric models; in general, this technique revealed excellent performance in the prediction of moisture content in powdered LML. The NIR predicted results for moisture content in powdered lemon myrtle leaves were analysed using the calibration model and cross-

validation method. For the validation models, PLSR method gave correlation coefficient (*r*) values of 0.9931 and 0.9951 for validation data set and RMSE_cv value of 0.0231% and 0.0427% for cross validation of NIR and DM, respectively. These results clearly demonstrated the capability of NIR and DM for moisture content prediction.

Table 4.13 Partial least square regression (PLSR) on full spectra of NIR and dielectric spectroscopy

Full spectra	Validation model			Test model		
	r	RMSE_ cv (%)	RPD_ cv	r_test	RMSEP (%)	RPD
NIR	0.9931	0.0231	8.59	0.9487	0.0589	3.16
DM	0.9951	0.0195	10.16	0.9183	0.0801	2.33

Generally, the test models in Table 4.13 show high value of coefficient of determination, *r*_test of 0.9487 and 0.9183, RPD of 3.16 and 2.33 and low value of RMSEP of 0.0589 and 0.0801% for NIR and DM, respectively. RPD value for the presented NIR model was 3.16 while 2.33 for DM test model. The RPD value for NIR is more than 3.0 which indicates that the model is good and excellent for prediction. It can be considered for prediction purposes and the calibration model could be used for the actual test (Nicolai et al., 2007). DM resulted in lower RPD value compared to NIR model

analysis. The RPD for DM indicated that the model has possibility for a coarse prediction of reference value. This result is also supported by the regression coefficient of predicted and actual plot for NIR and DM model as shown in Figure 4.34. From Figure 4.34, the regression coefficient (r^2) are 0.9001 and 0.8432, respectively for NIR and DM. The value of r^2 for NIR model of dried LML is lower than other NIR model reported in *Amaranthus* plant (Cai and Corke, 2001) with 0.99; green tea (Sinija and Mishra, 2011) with 0.9975 and commercial tea sample (Diniz et al.,2015) with r^2 value of 0.94 for moisture content determination. As for dielectric spectroscopy on leaves' moisture content successfully reported on maize leaves with value of r^2 of 0.77 (Afzal and Mousavi, 2008) which is lower than the r^2 for DM result in this study.

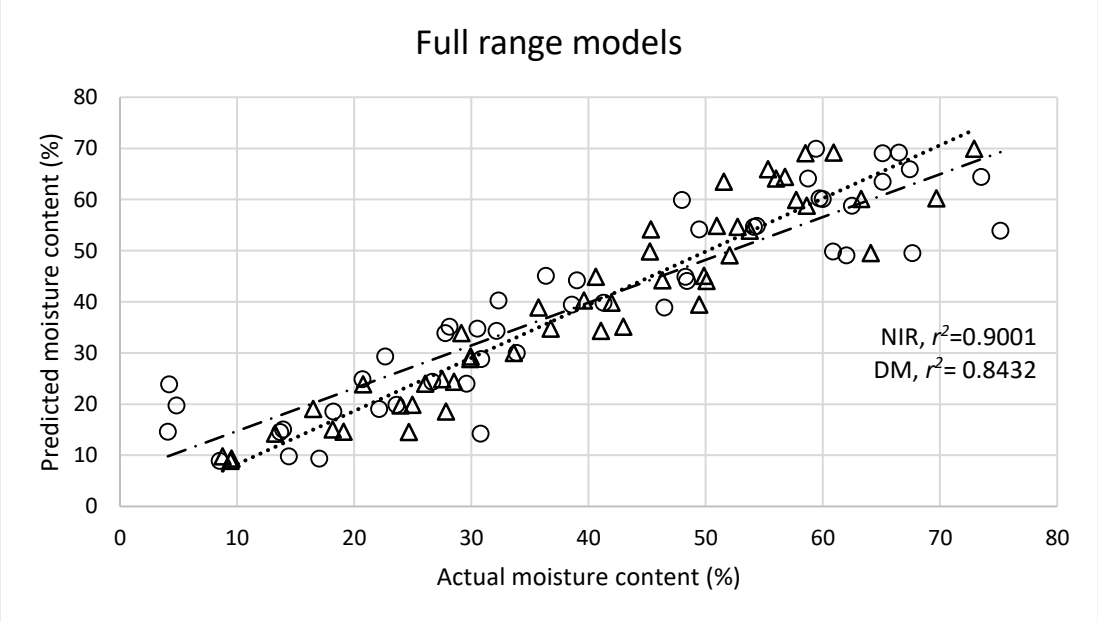


Figure 4.34 Moisture content (Full range of NIR and DM) prediction versus actual values content (∆: NIR; o: DM)

The difference of the PLSR results on the calibration and prediction models of NIR and DM are influenced by the method of measurements. NIR spectroscopy measures the spectral properties of organic molecules. NIR measurement involve in the surface measurement onto the surface of powdered lemon myrtle leaves sample by measuring the spectral properties of organic molecules, caused by the harmonic combinations of bands in the region of 1550 to 1950 nm by optical absorption and its reflectance. On the other hand, dielectric spectroscopy is a relative method that is directly proportional to any permittivity changes due to water and measures the total volume of water in the sample. Hence, both of the PLSR results showed different evaluation on the validation and test model as assessment for its performance in determining moisture content in the powdered LML.

In comparison between NIR and DM, NIR shows better results in predicting moisture content as higher RPD value and lower RMSEP value were obtained for NIR. These results are in agreement with Zhao et al. (2016) in comparing microwave dielectric and near-infrared spectroscopy for fat content determination in ground beef. They revealed that higher coefficient of determination (R^2P) of 0.99 and lower RMSEP of 0.71% were obtained for NIR spectroscopy while lower (R^2P) of 0.87 and higher RMSEP of 2.71% was obtained in

similar manner for microwave spectroscopy from PLS prediction models of the fat content. The difference might be influenced by variation of sample type and its application of both non-destructive techniques.

4.4.3.2 Models of selected ranges of NIR spectral data

Since the number of samples is limited, the selected range of spectra was tested to compare its efficiency in determining moisture content in powdered LML. From full spectral of 401 data points, it was reduced to 101 and 151 data points for two ranges of NIR spectral data. The selection of spectra was based on the most distinguished area of peak and lines obtained from the full range spectra of NIR as indication of clearer distinguished moisture levels of the sample as shown in Figure 4.27. There were 2 selected sets of spectra analysed for NIR method. The first range of NIR spectra was in the range of 1600 to 1700 nm (NIR1600-1700). Whereas, the second spectrum was in the range of 1650 to 1800 nm (NIR1650-1800). Table 4.14 shows the PLSR results on validation and test model evaluation for these two selected spectra.

Table 4.14 Partial least square regression (PLSR) on selected spectra of NIR

Selected spectra	No. of features	Validation model			Test model		
		r	RMSE_ cv (%)	RPD_ cv	r_test	RMSEP (%)	RPD
NIR1600 -1700	101	0.9611	0.0531	3.66	0.7958	0.1187	1.57
NIR1650 -1800	151	0.9846	0.0348	5.64	0.8496	0.103	1.80

Generally, NIR1650-1800 model shows better partial least square regression (PLSR) values compared to NIR1600-1700 for both validation and test models. For validation model, the NIR1650-1800 spectral range resulted in higher *r* value of 0.9846 compared to validation model of NIR1600-1700 spectra with only 0.9611. The root mean square error (RMSE_cv) value for validation set obtained from NIR1650-1800 was also lower than RMSE_cv value for NIR1600-1700 with 0.0348% and 0.0531%, respectively. These results are also in line with PLSR values of test model where NIR1650-1800 shows better model with higher value of *r*_test (0.8496) and RPD (1.80) and lower RMSEP value (0.103%) compared to the PLS values of NIR1600-1700 test model.

The regression of predicted values versus actual moisture content for both selected spectral ranges are shown in Figure 4.35. The plots for both selected range of spectra were not really close to the regression lines. This is seen as indication of the model are not really

sensitive in determining moisture content from powdered LML. Higher value of r^2 was obtained from NIR1650-1800 which was 0.7219, while lower r^2 of 0.633 was obtained from NIR1600-1700 spectral analysis.

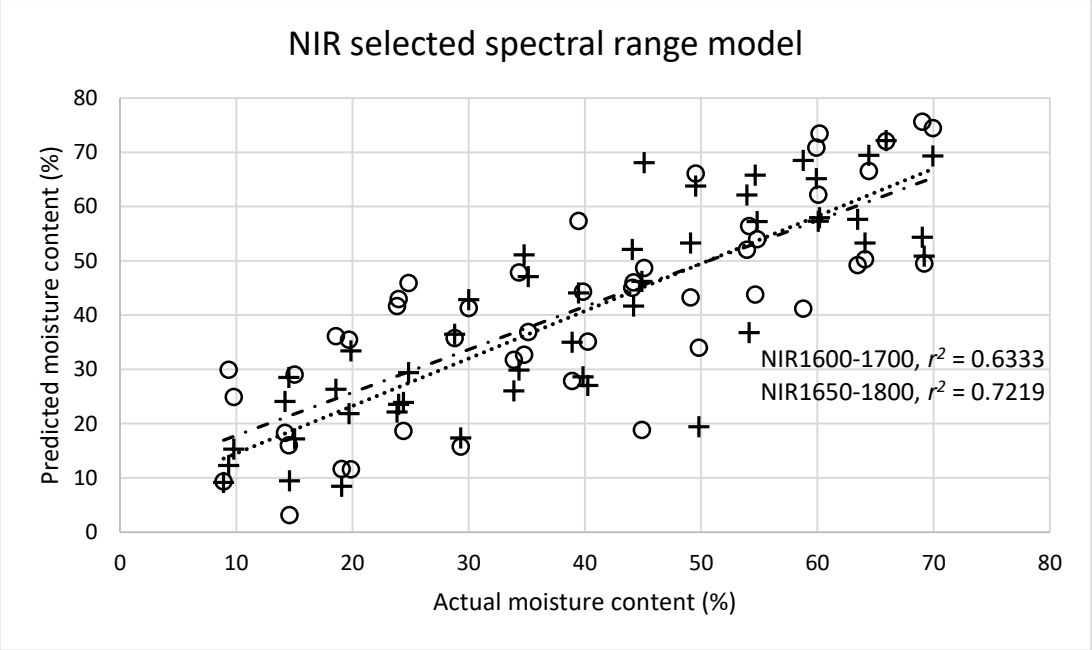


Figure 4.35 Moisture content (selected spectra of NIR) prediction versus actual values content (o: NIR1600-1700; +: NIR1650-1800)

For the comparison between full and selected spectral analysis, the performance of validation and test model of both selected ranges of NIR spectra shows no improvement in PLS values. The full spectra NIR model evaluation shows significant difference in values of *r*, RMSEP and RPD. The PLSR results for full NIR spectra resulted in higher value of *r* and RPD with much lower RMSEP in both validation and test models (Table 4.13). Although the number of data is larger

than the number of samples, full features of spectral data are more reliable for moisture content prediction. This result also indicates that for moisture content determination in dried LML, model of full spectra gives better prediction of moisture content instead of the selected spectral range. The same trend was also reported by Shimbori and Kurata (2017) where the PLSR modelling results for partial spectral ranges was not superior to the full spectral ranges for water content prediction in hardwood leaves using NIR spectroscopy.

4.4.3.3 Fusion models of full and selected ranges of NIR spectral data and full features of dielectric measurement

Fusion of models is the combination of full features for both methods tested in this study. The fusion was aimed to investigate the improvement of model's performance in determining moisture content of powdered LML as higher number of features used for the validation and test of the models. There are three fusion models evaluated in this study which were the fusion of full features of individual non-destructive techniques, NIR+DM; the fusion of selected spectra range and DM, NIR1600-1700+DM; and the fusion of second selected NIR range and DM, NIR1650-1800+DM. Table 4.15 shows the PLSR results on three fusion models on its validation and test performance.

Table 4.15 Partial least square regression (PLSR) on full and selected spectra of NIR infused with full range dielectric measurement

Selected spectra	Validation model			Test model		
	r	RMSE_cv (%)	RPD_cv	r_test	RMSEP (%)	RPD
NIR+DM	0.9935	0.0219	8.87	0.9323	0.0708	2.63
NIR1600-1700+DM	0.9932	0.0225	8.66	0.9219	0.0891	2.09
NIR1650-1800+DM	0.9960	0.0176	11.27	0.8811	0.0923	2.02

For the calibration model, all the *r* value resulted as more than 0.99, in the range of 0.9900-0.9960. The highest *r* was obtained from the fusion of full features of NIR1650-1800+DM and followed by NIR+DM and NIR1600-1700+DM. The same trend was also observed from the RMSE_cv where the lowest RMSE_cv values were from NIR1650+DM with 0.0176 followed by 0.0219 and 0.0225 % for NIR+DM and NIR1600-1700+DM, respectively. For test model evaluation, the *r*_test results were in range of 0.8811 to 0.9323 with NIR+DM as the highest value and the value of RMSEP are in the range of 0.0708-0.0923% with NIR+DM as the lowest value.

For RPD value, only NIR+DM model exceeded value of 2.5 as indication of good and excellence prediction. The highest RPD was recorded from NIR+DM which was 2.63, whereas the lowest was

obtained from NIR1650-1800+DM model and the value was 2.02. Generally, the fusion of full features of NIR+DM showed better evaluation as the models resulted in the highest *r*_test and RPD with the lowest RMSEP value compared to two other fusions models. This is in line with the finding above on the comparison between the model of full versus the selected range of spectra that gave better prediction for moisture content in dried LML.

Figure 4.36 shows the plot of prediction values against the actual moisture content for the three different models. The plots seem to distribute closely to the regression as indication of high correlation between the predicted and actual plots of moisture content. The highest r^2 from the plots was NIR+DM which was 0.8691, while NIR1600-1700+DM gave the lowest r^2 value of 0.7763.

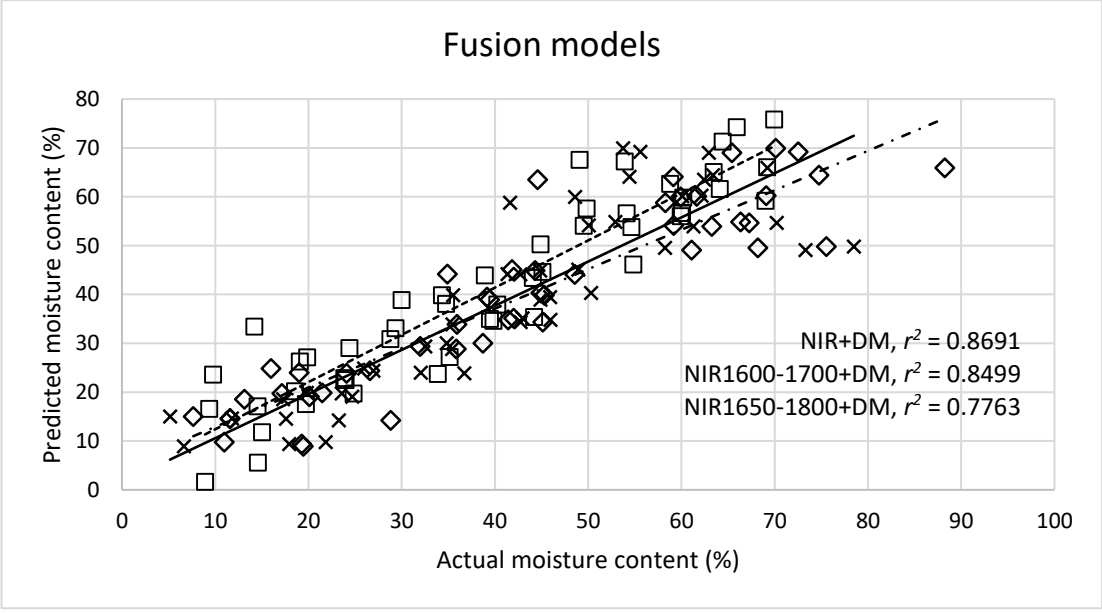


Figure 4.36 Moisture content (fusion of full and selected spectra of NIR+DM) prediction versus actual values content (□: NIR+DM; ◊: NIR1600-1700+DM; X: NIR1650-1800+DM)

In comparison to determine the efficiency between individual non-destructive techniques to the fusion of techniques, the PLSR for the fusion model showed only slight improvement for its *r*, RMSEP and RPD values. The value of *r* for NIR+DM was improved only by 0.04% for NIR and lower than single method of DM. As for the values of RMSEP in test model, the fusion model only reduced by 12% from DM value whilst NIR single method still resulted in the lowest RMSEP value. The same trend was also observed for RPD values where fusion models only improved the RPD value by 13% from DM model and NIR model still resulted in the highest value of RPD that exceeded 3.0. Therefore, it can be seen that the capability of dielectric measurement as single method application in moisture content prediction can be improved by combining the method with another non-destructive method (i.e NIR).

A good prediction of model is indicated by high value of r^2, RPD and low values of RMSE_cv and RMSEP values (Kujundžić et al., 2017). In this study, the model that fitted in the above criteria was obtained from NIR full spectra analysis. However, the fusion model, NIR+DM also show good and excellence prediction as the values of RPD in the range of 2.5 to 3.0 or higher.

Both non-destructive techniques were successfully evaluated for their performance in moisture content determination of dried LML. NIR have the advantage on light-weighted and compact sensor as easily adapted to the dryer for rapid, in and on-line monitoring for better understanding in the moisture changes through the drying process. On the other hand, dielectric measurement has disadvantage for in situ measurement as for the bulky instrumentation especially in featuring the sensor to dryers. Hence, another factor for consideration in adapting the sensor for the dryers is the adaptability to the dehydrators as more adaptable sensor can be easily featured to the drying instruments and provide in situ and on-line monitoring on the change's detection during drying process.

CHAPTER 5 CONCLUSION AND FUTURE WORKS

5.1 Conclusion

Drying of lemon myrtle leaves (LML) was performed using three different methods, namely oven drying (OD), vacuum drying (VD) and heat pump drying (HPD). For OD, the drying temperature was varied from 40°C (OD40), 50°C (OD50) and 60°C (OD60). The drying temperature for VD was also varied at 40°C (VD40), 50°C (VD50) and 60°C (VD60) and pressure of 50 mbar. It was found that the moisture content of LML was reduced from 58-70% w.b (fresh LML) to 8-10% wet basis for all drying methods. OD60 gave the highest drying rate, followed by OD50 and HPD while VD40 gave the lowest drying rate. The drying rate was influenced by the drying temperature significantly. Higher drying rate was obtained at higher drying temperature as a result of rapid removal of moisture from the samples. The effective moisture diffusivity, D_{eff} and activation energy, E_a values were found to vary with the different drying methods. The highest D_{eff} of 4.35 x 10^{-9} m^2/s was obtained from LML subjected to OD60 but the lowest D_{eff} was obtained from LML subjected to VD40 which was 4.17 x 10^{-10} m^2/s. The value of activation energy, E_a was found to be 72.85 kJ/mol and 45.41 kJ/mol for OD and VD, respectively. Whereas, the E_a for HPD was 13.42

kJ/mol as indication that HPD was an effective low operating temperature dryer for LML drying.

Colour changes of LML were also assessed to evaluate the difference of the colour parameter between the fresh leaves and the dried LML. The colour of the dried LML was found to be affected by the drying temperature and total drying time. Drying at higher temperature, OD60 samples exhibited the lowest lightness (L^*) value (darker colour), more positive in greenness, a^* value (less green) and the highest browning index (BI) value. On the other hand, VD40 sample showed the lowest colour intensity value (chroma) due to longer drying time in vacuum drying. Furthermore, OD60 gave the highest total colour difference (TCD) value which was 10.60 as indication of more chlorophyll oxidation due to higher temperature of drying. High quality of dried leaves was described by high retention of active ingredients and phytochemical content in leaves as indicated by the minimal change in colour, low BI and low a^* value (more green). All in all, HPD is a suitable drying method for LML drying as the HPD sample gave the lowest value of a^* (more green), whilst the TCD and BI which was very close to the fresh leaves.

The antioxidant and total phenolic content of the dried LML were retained after the drying process. Different methods and conditions showed variation on the retention of biochemical contents. VD

samples showed the highest retention of TPC and antioxidant activities followed by HPD and OD. OD60 sample showed the greatest loss of TPC, DPPH and FRAP values after drying. Whereas, VD60 sample showed the highest retention of TPC and FRAP, while VD40 sample showed the highest retention of DPPH value. The variation of the biochemical retention in dried LML was influenced by the drying temperature and drying time. Higher drying temperature caused higher loss of polyphenol and antioxidant content due to oxidation and enzymatic reaction. In addition, longer duration of drying prolonged exposure of the drying samples to thermal degradation of bioactive content and thus lowered the retention of biochemical content in LML after drying. In comparison between the different dryers, HPD samples showed comparable results of biochemical retention (p>0.05) to the VD samples. HPD samples also showed the highest retention of volatile compounds (cis- and trans-citral) at a total concentration of 89.5%, while 88.53 and 70.58% for VD and OD, respectively. The high retention of the volatile compounds of LML indicated that HPD was an effective drying method for preserving of dried LML quality especially in terms of the functionality of LML. It was also found that HPD required shorter time for drying to be completed compared to the other two drying methods (OD50 and VD50), hence HPD could retain higher amount of the volatile compounds.

The transport properties, biochemical and volatile retention and colour changes were varied in LML subjected to HPD at different drying air velocities. The drying time was reduced with increasing in drying air velocities. It was found that LML subjected to HPD at drying air velocity of 2.0 m/s resulted in the highest TPC, DPPH, FRAP and volatile retention as well as lowest *a** (greener) and TCD value (less colour changes). This showed that HPD carried out at moderate air velocity (in this case, 2.0 m/s) gave the best quality of dehydrated LML, in terms of retention of volatile compounds and colour change.

The effect of packaging and storage temperature on the quality parameters of dried lemon myrtle leaves, using three different drying methods, were analysed during six months of storage. VD resulted in lower concentration of TPC and antioxidant compounds, whereas HPD yielded the highest values of the biochemical content after 6 months storage. Packaging using vacuum and chilled condition (VCH) showed a better preservation on colour and biochemical content of the dried samples compared to non-vacuum packaging and storage at room temperature (NRT), especially for HPD samples. Small reduction of TPC, FRAP and DPPH values were exhibited by the HPD samples stored in vacuum-packed at chilled conditions (VCH) with 31, 15 and 25% of loss, respectively. This was because samples stored in vacuum packing experienced less degree

of Maillard reaction as there is no presence of oxygen hence oxidation was minimized significantly. This study shows that HPD can be an effective method as it could retain the volatile compounds, preserve colour after drying and the preservation of the volatile compounds as well as the colour parameters was more stable during storage.

Potentially, both non-destructive techniques, namely near-infrared (NIR) and dielectric (DM) spectroscopy showed the capabilities in determining moisture content in different dried LML subjected to oven-, vacuum- and heat pump drying. NIR spectroscopy resulted in the high value of coefficient of determination, r (>0.99) and ratio of performance to deviation, RPD (>3.0) indicating a good model in predicting moisture content of dried LML. Partial least square regression (PLSR) results for dielectric measurements were also comparable to NIR results. However, reduced number of data points in the selected range of NIR spectra showed no improvement in PLSR values with lower r and RPD values, indicating lower reliability on moisture content prediction in dried LML. On the other hand, fusion models of the full and selected ranges of NIR spectra and dielectric measurement showed an improvement on the prediction model performance for moisture content determination, better than single measurement performances. However, as for accessorising the non-destructive measurements to the drying process for rapid, in- and

on-line monitoring of moisture content to the dehydrators, NIR spectroscopy is more suitable to be implemented as it is a light and compact sensor, simple and economical compared to the bulky instrumentation of dielectric spectroscopy.

5.2 Future works

The present study revealed the potential of HPD as an alternative for drying of LML and preservation of its volatile, biochemical and colour retention. The study of HPD application on other herbs can be carried out as it shows good potential in preserving heat sensitive compounds in food and bioproducts. The potential non-destructive measurement facilitation can be further explored for a rapid, in and on-line monitoring of products quality during drying process.

The following future works are recommended:

- Characterisation of the cell structure of different dried LML subjected to OD, VD and HPD by using Scanning Electron Microscope (SEM)
- Performance and cost analysis of HPD based on optimum drying parameters from this research
- Incorporation of non-destructive technique(s) for rapid and real-time monitoring of drying process including the estimation of moisture and phytochemical contents

- Application of other packaging materials to improve the functionality stability of dried LML
- Modelling of water transport during drying based on physical and morphological structure of LML with detailed information on the stomatal opening and closing effect on transport properties

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APPENDIX A

Figure A1 Commercially dried lemon myrtle leaves

Figure A2 Lemon myrtle plantation in Sekinchan, Selangor, Malaysia

APPENDIX B

Figure B1 Control panel for Heat pump dryer

Figure B2 Actual diagram of inner compartment of heat pump dryer

APPENDIX C

Calibration curve of Antioxidant

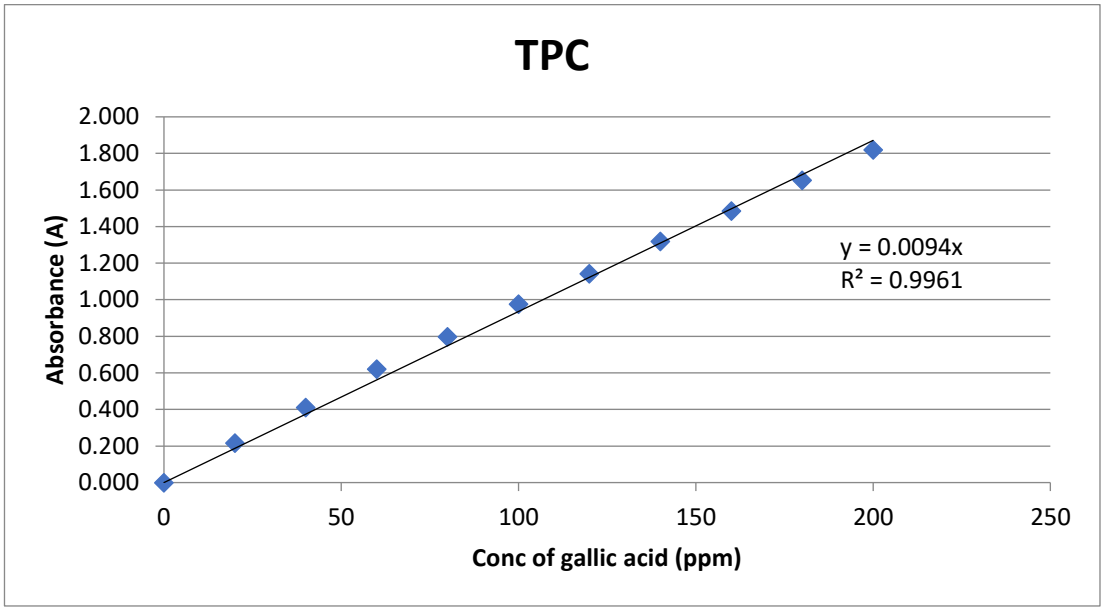


Figure C1 Calibration curve of standard for TPC assay

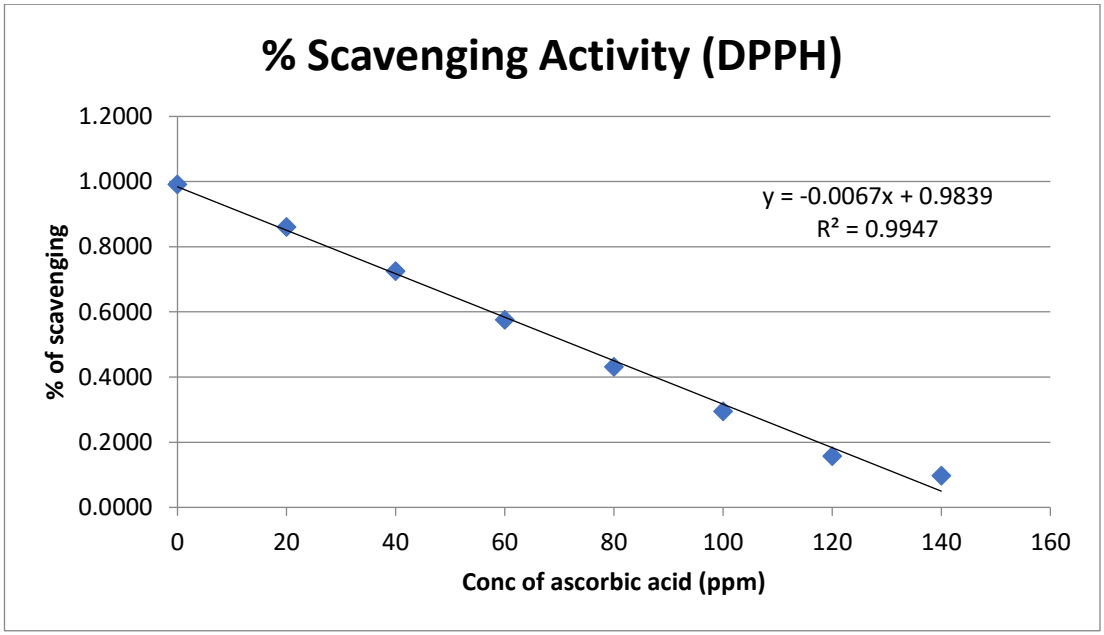


Figure C2 Calibration curve of standard for DPPH assay

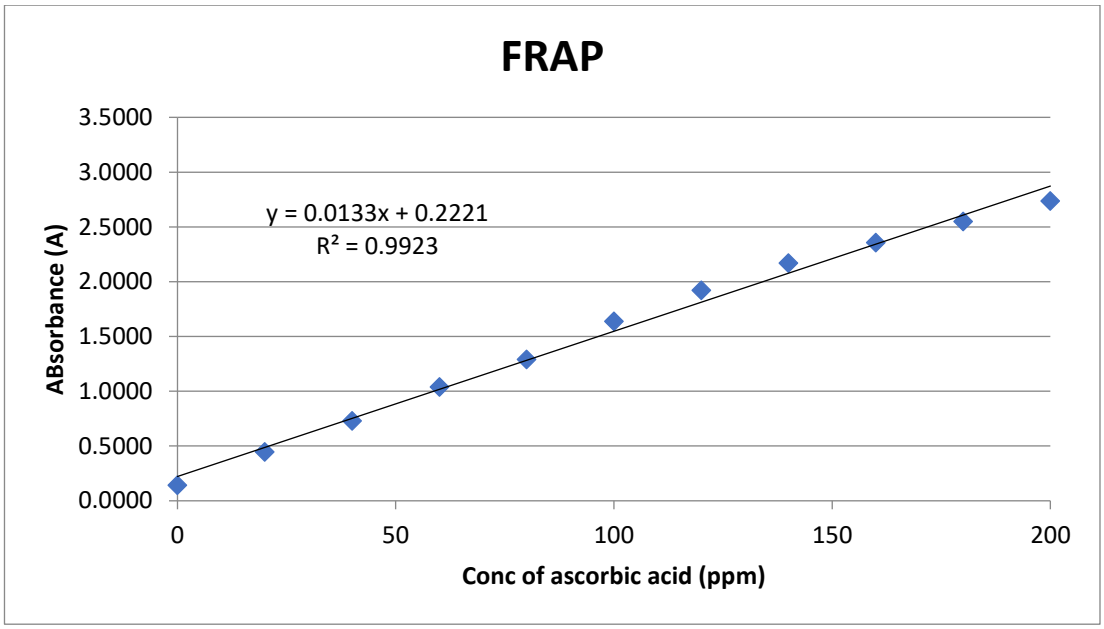


Figure C3 Calibration curve of standard for FRAP assay

Table C1 Calibration curves, linearity range, limit of detection and limit of quantification of three assays (TPC, DPPH, FRAP)

Assay	Linear equation	R^2	Linearity range	LOD	LOQ
TPC	y = 0.0094x	0.9961	0 - 200	0.99	3.02
DPPH	y = -0.0067x + 0.9839	0.9947	0 - 140	0.99	3.02
FRAP	y = 0.0133 x + 0.2221	0.9923	0 - 200	0.99	3.02

APPENDIX D

Gas Chromatography- Mass Spectrometer (GC-MS) method and selected chromatograms

Method

[Comment]

===== Analytical Line 1 =====

[AOC-20i+s]
# of Rinses with Presolvent :4
# of Rinses with Solvent(post) :4
# of Rinses with Sample :0
Plunger Speed(Suction) :Middle
Viscosity Comp. Time :0.2 sec
Plunger Speed(Injection) :High
Syringe Insertion Speed :High
Injection Mode :Normal
Pumping Times :0
Inj. Port Dwell Time :0.0 sec
Terminal Air Gap :No
Plunger Washing Speed :High
Washing Volume :6uL
Syringe Suction Position :0.0 mm
Syringe Injection Position :0.0 mm
Solvent Selection :All A,B,C

[GC-2010]COLUMN: ZEBRON ZB5ms 30 meter x 0.25mm I.D x 0.25um film thickness
Column Oven Temp. :50.0 °C
Injection Temp. :250.00 °C
Injection Mode :Split
Flow Control Mode :Linear Velocity
Pressure :53.5 kPa
Total Flow :9.0 mL/min
Column Flow :1.00 mL/min
Linear Velocity :36.3 cm/sec
Purge Flow :3.0 mL/min
Split Ratio :5.0
High Pressure Injection :OFF
Carrier Gas Saver :OFF
Splitter Hold :OFF
Oven Temp. Program

Rate	Temperature(°C)	Hold Time(min)
-	50.0	1.00
7.00	130.0	12.60

Purge Flow Program

Rate	Flow(mL/min)	Hold Time(min)
-	3.0	13.00

< Ready Check Heat Unit >
Column Oven : Yes
SPL1 : Yes
MS : Yes
< Ready Check Detector(PTD) >
< Ready Check Baseline Drift >
< Ready Check Injection Flow >
SPL1 Carrier : Yes
SPL1 Purge : Yes
< Ready Check APC Flow >
< Ready Check Detector APC Flow >
External Wait :No
Equilibrium Time :0.5 min

[GC Program]

[GCMS-QP2010 Plus]
IonSourceTemp :200.00 °C
Interface Temp. :250.00 °C
Solvent Cut Time :2.50 min
Detector Gain Mode :Absolute
Detector Gain :0.75 kV
Threshold :70

[MS Table]
--Group 1 - Event 1--
Start Time :3.00min
End Time :25.00 min
ACQ Mode :Scan
Event Time :0.30sec
Scan Speed :1111
Start m/z :35.00
End m/z :350.00

Sample Inlet Unit :GC

Figure D1 Gas Chromatography – Mass Spectrometer (GC-MS) methodology

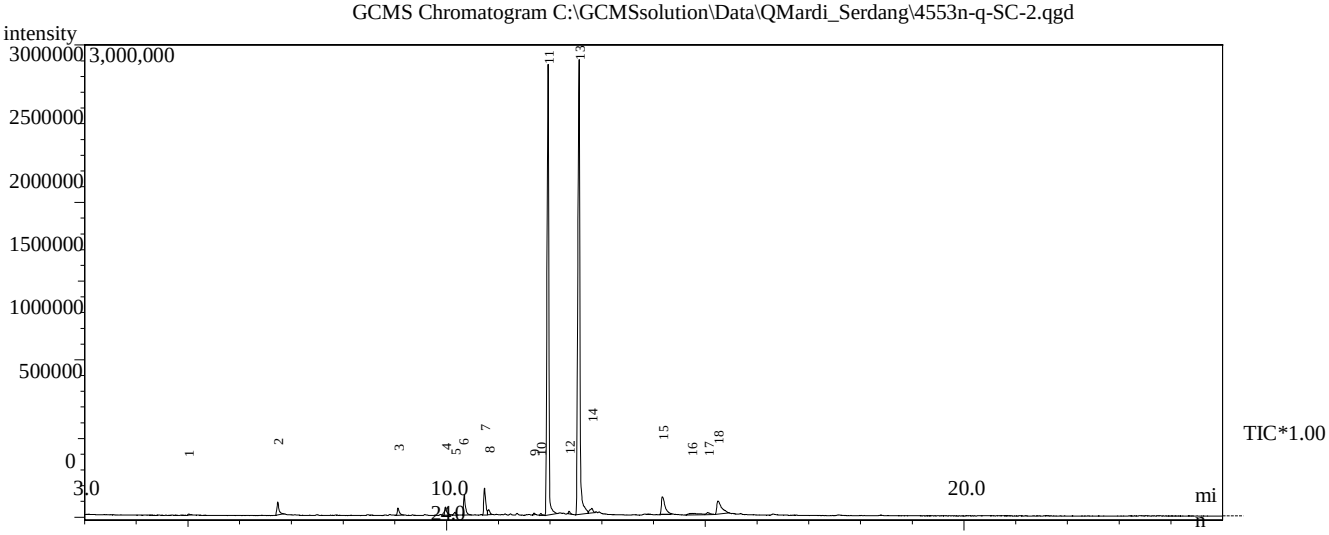
Figure D2 Gas Chromatogram – Mass Spectrometer (GC-MS) result for essential oil of lemon myrtle leaves subjected to HPD at drying air velocity of 2.0 m/s

MAKMAL GCMS G39 BSF, JABATAN KIMIA, FAKULTI SAINS, UPM, SERDANG, SELANGOR GCMS QP2010 Plus SHIMADZU

GCMS Sample Information

Data Acquired by	: Admin
Acquisiton Date	: 6/29/2018 9:17:05 PM
Sample Type	: Unknown
Level #	1
Sample Name	: Sample C
Sample ID	: AinnaAKahar(MARDI Serdang)
IS Amount	: [1]=1
Sample Amount	5
Dilution Factor	100
Vial #	2
Injection Volume	: 0.2
Data File	: C:\GCMSsolution\Data\QMardi_Serdang\4553n-q-SC-2.qgd
Method File	: C:\GCMSsolution\Data\Qmardi_Serdang\Q-ZB5ms-50130.qgm
Report File	: gcpotrait-zb5ms.qgr
Tuning File	: C:\GCMSsolution\System\Tune1\270618EIF1.qgt
Modified by	:
Admin Final Process & print date	: 7/2/2018 9:21:58 PM

GCMS Chromatogram C:\GCMSsolution\Data\QMardi_Serdang\4553n-q-SC-2.qgd



GCMS Peak Report TIC

Peak#	R.Time	Area	Height	A/H	Mark	Base m/z	Area%	Height%
1	5.008	19905	7907	2.50	MI	43.05	0.11	0.12
2	6.737	235728	81939	2.88		109.10	1.32	1.25
3	9.059	128499	45690	2.81		71.05	0.72	0.70
4	9.976	137657	52760	2.61		69.10	0.77	0.80
5	10.161	44377	16806	2.64	MI	41.10	0.25	0.26
6	10.342	310498	128756	2.41		67.05	1.74	1.96
7	10.732	439624	170311	2.58		81.10	2.46	2.59
8	10.815	90465	33281	2.72	V	84.05	0.51	0.51
9	11.692	22725	10971	2.02	MI	43.05	0.13	0.17
10	11.823	23573	11229	2.10	MI	43.05	0.13	0.17
11	11.963	6945697	2862411	2.43		41.05	38.88	43.55
12	12.365	32399	16101	2.03	MI	41.10	0.18	0.24
13	12.562	8053826	2888893	2.79		69.10	45.08	43.96
14	12.808	118925	27665	4.30	MI	59.05	0.67	0.42
15	14.170	540290	113354	4.77		82.05	3.02	1.72
16	14.745	133971	9229	14.48	MI	69.10	0.75	0.14
17	15.049	60230	12345	4.88	MI	110.10	0.34	0.19
18	15.243	526395	82673	6.37		59.05	2.95	1.26
		17864784	6572321				100.00	100.00

GCMS Library

<< Target >>
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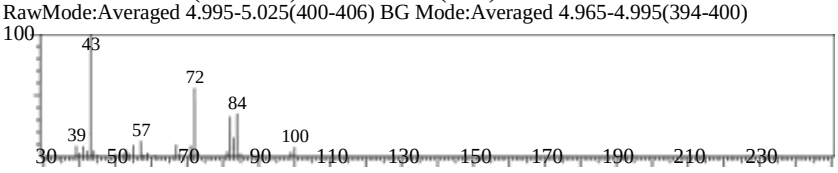

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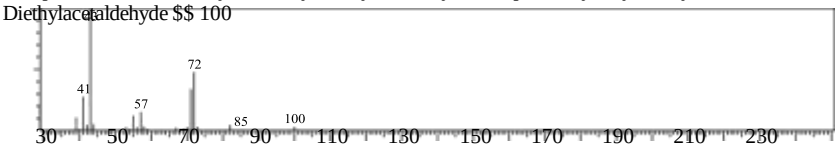

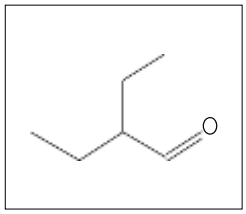

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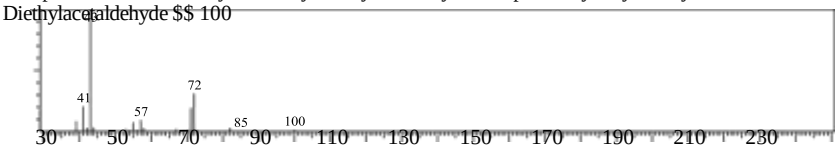

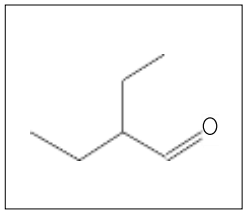

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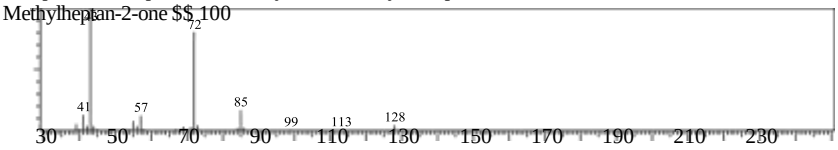

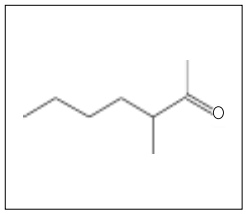

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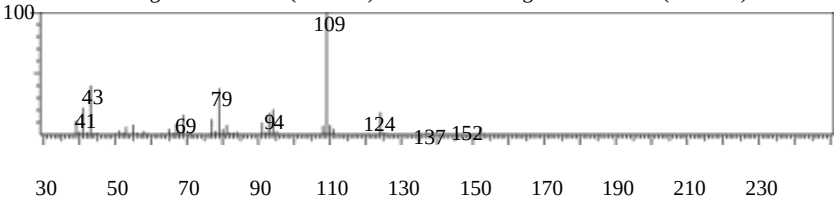

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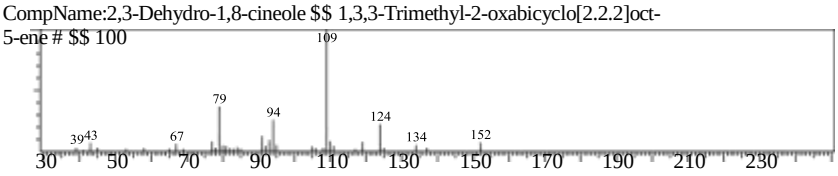

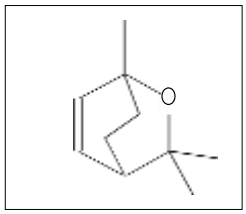

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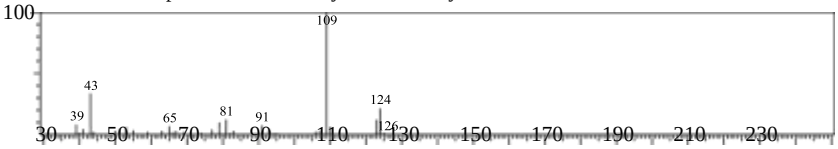

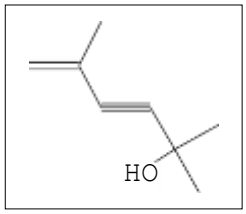

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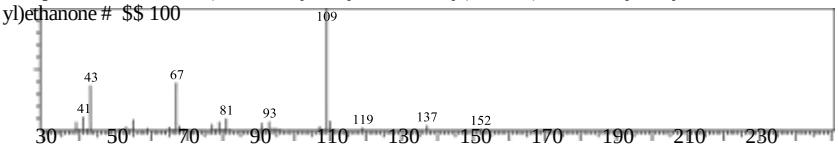
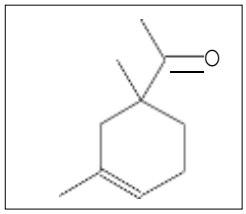
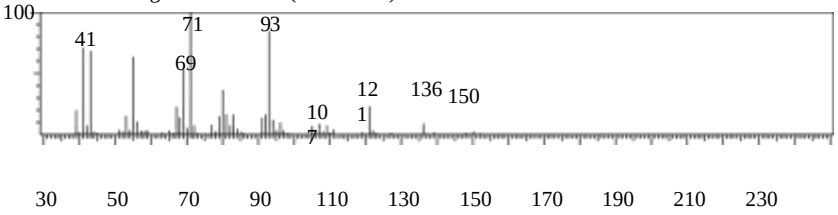
SI:82 Formula:C10H16O CAS:51733-68-7 MolWeight:152 RetIndex:1161
CompName:Ethanone, 1-(1,3-dimethyl-3-cyclohexen-1-yl)- $$ 1-(1,3-Dimethyl-3-cyclohexen-1-yl)ethanone # $$ 100

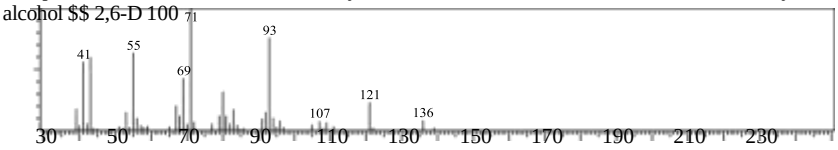


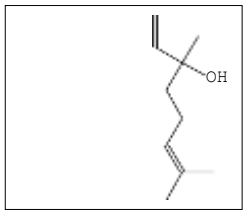


<< Target >>
Line#:3 R.Time:9.060(Scan#:1213)
RawMode:Averaged 9.055-9.065(1212-1214) BG Mode:Calc. from Peak

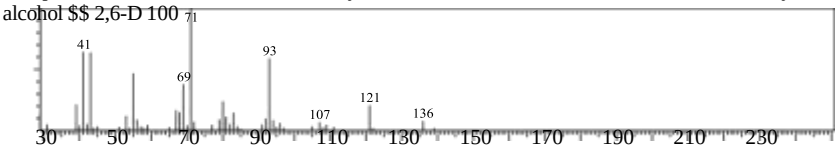


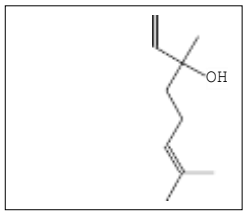
Hit#:1 Entry:17386 Library:NIST08.LIB
SI:96 Formula:C10H18O CAS:78-70-6 MolWeight:154 RetIndex:1082
CompName:1,6-Octadien-3-ol, 3,7-dimethyl- $$ .beta.-Linalool $$ Linalol $$ Linalool $$ Linalyl alcohol $$ 2,6-D 100



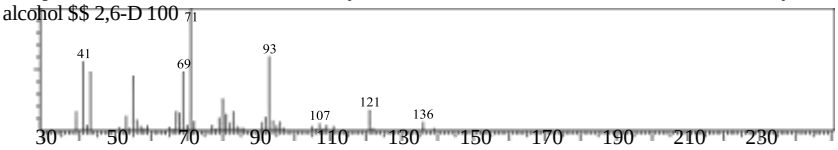


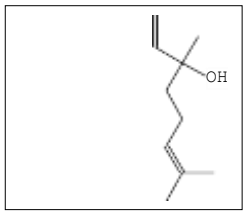
Hit#:2 Entry:9608 Library:NIST08s.LIB
SI:93 Formula:C10H18O CAS:78-70-6 MolWeight:154 RetIndex:1082
CompName:1,6-Octadien-3-ol, 3,7-dimethyl- $$ .beta.-Linalool $$ Linalol $$ Linalool $$ Linalyl alcohol $$ 2,6-D 100



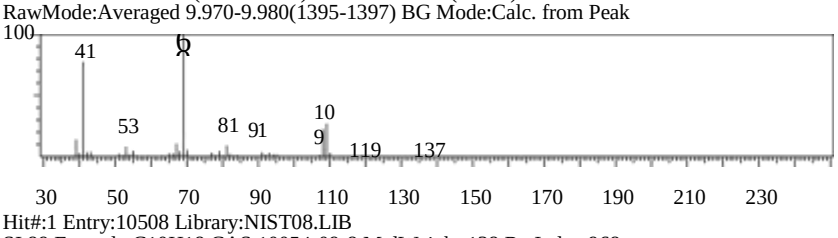


Hit#:3 Entry:9612 Library:NIST08s.LIB
SI:93 Formula:C10H18O CAS:78-70-6 MolWeight:154 RetIndex:1082
CompName:1,6-Octadien-3-ol, 3,7-dimethyl- $$ .beta.-Linalool $$ Linalol $$ Linalool $$ Linalyl alcohol $$ 2,6-D 100

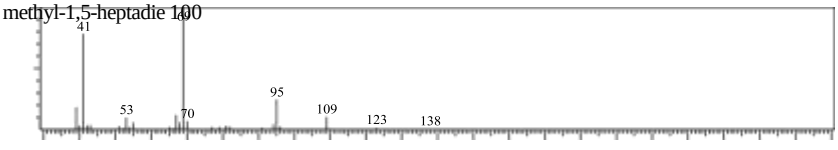


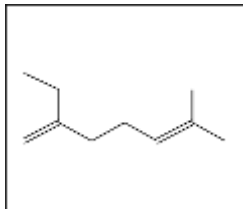


<< Target >>
Line#:4 R.Time:9.975(Scan#:1396) BasePeak:69.10(14008)
RawMode:Averaged 9.970-9.980(1395-1397) BG Mode:Calc. from Peak

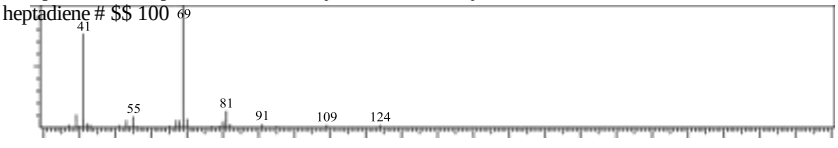


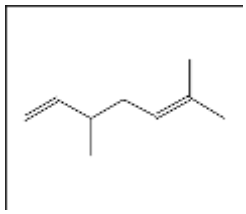
Hit#:1 Entry:10508 Library:NIST08.LIB
SI:89 Formula:C10H18 CAS:10054-09-8 MolWeight:138 RetIndex:968
CompName:2-Octene, 2-methyl-6-methylene- $$ 2-Methyl 6-methylene 2-octene $$ 2-Ethyl-6-methyl-1,5-heptadie 100



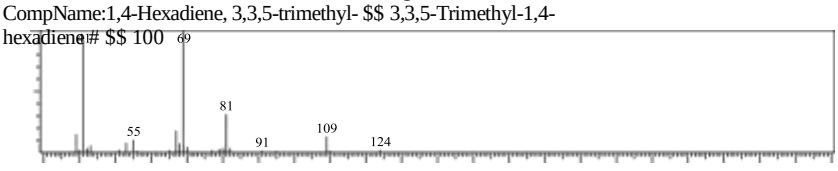


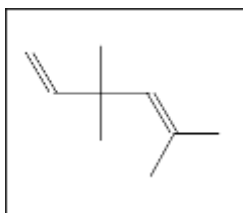
Hit#:2 Entry:6066 Library:NIST08.LIB
SI:88 Formula:C9H16 CAS:34891-10-6 MolWeight:124 RetIndex:827
CompName:1,5-Heptadiene, 3,6-dimethyl- $$ 3,6-Dimethyl-1,5-heptadiene # $$ 100

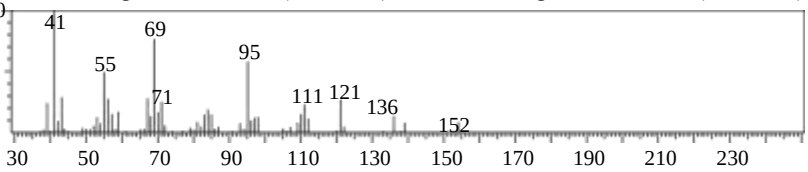


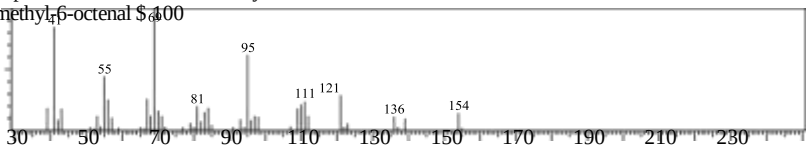


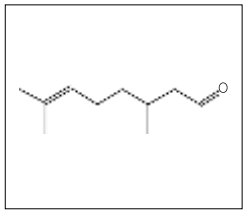
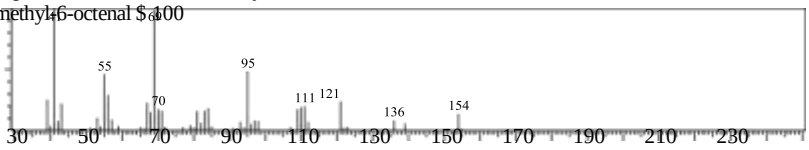
Hit#:3 Entry:6069 Library:NIST08.LIB
SI:88 Formula:C9H16 CAS:74753-00-7 MolWeight:124 RetIndex:807
CompName:1,4-Hexadiene, 3,3,5-trimethyl- $$ 3,3,5-Trimethyl-1,4-hexadiene # $$ 100

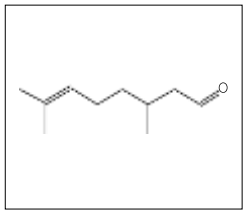
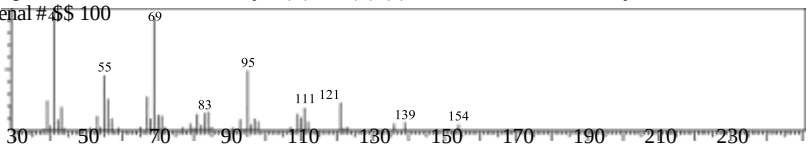


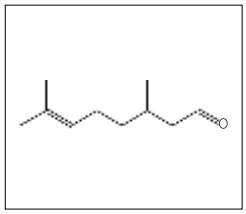
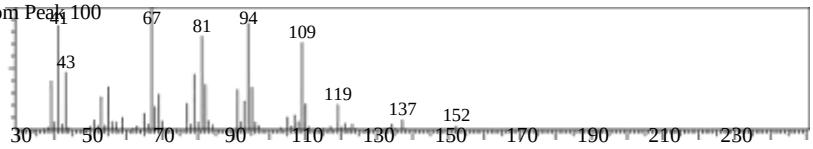


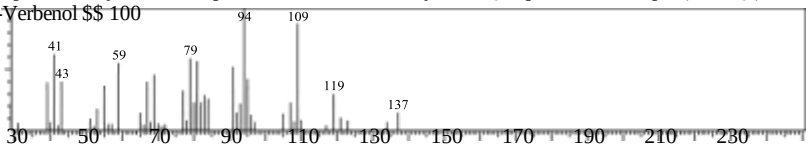
<< Target >>
Line#:5 R.Time:10.160(Scan#:1423)
RawMode:Averaged 10.140-10.185(1429-1438) BG Mode:Averaged 10.090-10.130(1419-1427)
100


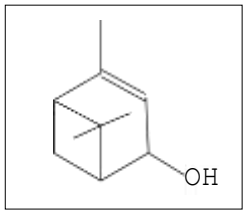
Hit#:1 Entry:9601 Library:NIST08s.LIB
SI:92 Formula:C10H18O CAS:106-23-0 MolWeight:154 RetIndex:1125
CompName:6-Octenal, 3,7-dimethyl- $$ .beta.-Citronellal $$ Citronellal $$ Rhodinal $$ 3,7-Dimethyl-6-octenal $ 100


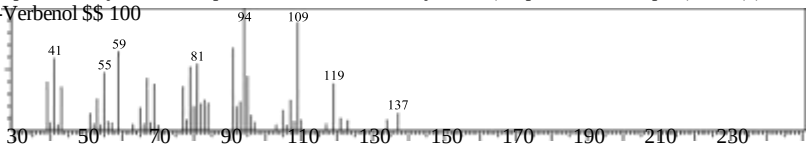

Hit#:2 Entry:9553 Library:NIST08s.LIB
SI:92 Formula:C10H18O CAS:106-23-0 MolWeight:154 RetIndex:1125
CompName:6-Octenal, 3,7-dimethyl- $$ .beta.-Citronellal $$ Citronellal $$ Rhodinal $$ 3,7-Dimethyl-6-octenal $ 100


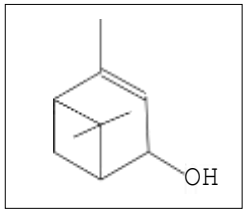

Hit#:3 Entry:17267 Library:NIST08.LIB
SI:91 Formula:C10H18O CAS:2385-77-5 MolWeight:154 RetIndex:1125
CompName:6-Octenal, 3,7-dimethyl-, (R)- $$ (R)-(+)-Citronellal $$ 3,7-Dimethyl-6-octenal # $$ 100


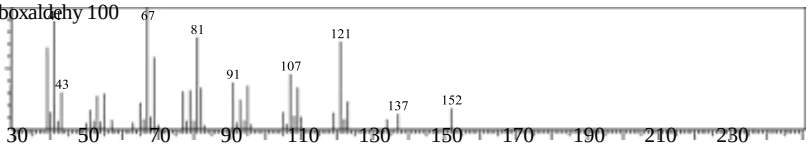

<< Target >>
Line#:6 R.Time:10.340(Scan#:1469) BasePeak:67.05(11383)
RawMode:Averaged 10.335-10.345(1468-1470) BG Mode:Calc. from Peak 100


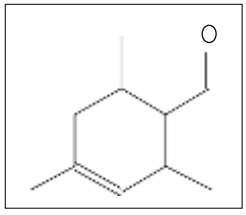
Hit#:1 Entry:9195 Library:NIST08s.LIB
SI:89 Formula:C10H16O CAS:18881-04-4 MolWeight:152 RetIndex:1136
CompName:Bicyclo[3.1.1]hept-3-en-2-ol, 4,6,6-trimethyl-, [1S-(1.alpha.,2.beta.,5.alpha.)]- $$ (S)-cis-Verbenol $$ 100




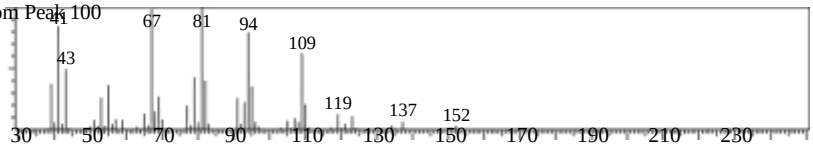
Hit#:2 Entry:16297 Library:NIST08.LIB
SI:89 Formula:C10H16O CAS:18881-04-4 MolWeight:152 RetIndex:1136
CompName:Bicyclo[3.1.1]hept-3-en-2-ol, 4,6,6-trimethyl-, [1S-(1.alpha.,2.beta.,5.alpha.)]- $$ (S)-cis-Verbenol $$ 100




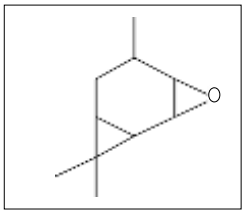
Hit#:3 Entry:16202 Library:NIST08.LIB
SI:87 Formula:C10H16O CAS:1423-46-7 MolWeight:152 RetIndex:1163
CompName:3-Cyclohexene-1-carboxaldehyde, 2,4,6-trimethyl- $$ 2,4,6-Trimethyl-3-cyclohexene-1-carboxaldehy 100




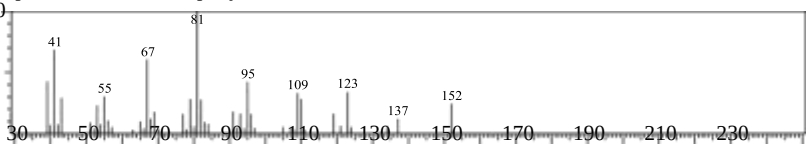
<< Target >>
Line#:7
RawMode:Averaged 10.725-10.735(1546-1548) BG Mode:Calc.
from Peak 100



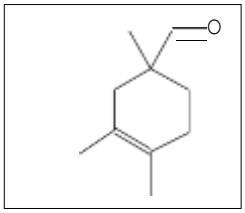
Hit#:1 Entry:16248 Library:NIST08.LIB
SI:89 Formula:C10H16O CAS:6909-20-2 MolWeight:152 RetIndex:948
CompName:Carane, 4,5-epoxy-, trans

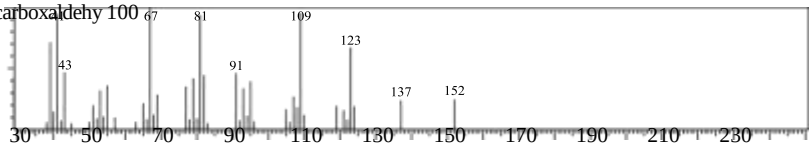


100

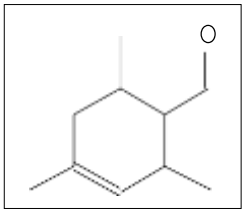


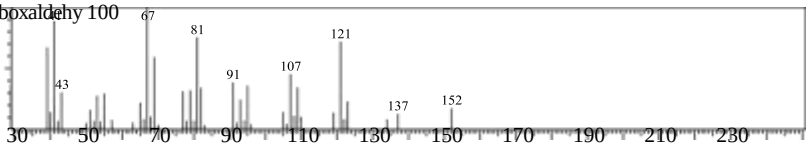
Hit#:2 Entry:16208 Library:NIST08.LIB
SI:88 Formula:C10H16O CAS:40702-26-9 MolWeight:152 RetIndex:1204
CompName:3-Cyclohexene-1-carboxaldehyde, 1,3,4-trimethyl- $$ 1,3,4-Trimethyl-3-cyclohexenyl-1-carboxaldehy 100



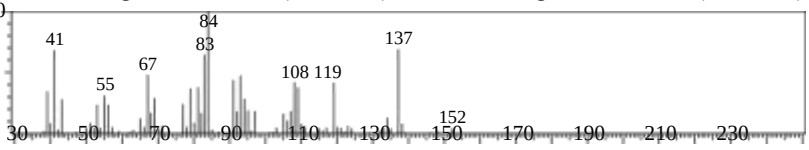


Hit#:3 Entry:16202 Library:NIST08.LIB
SI:87 Formula:C10H16O CAS:1423-46-7 MolWeight:152 RetIndex:1163
CompName:3-Cyclohexene-1-carboxaldehyde, 2,4,6-trimethyl- $$ 2,4,6-Trimethyl-3-cyclohexene-1-carboxaldehy 100

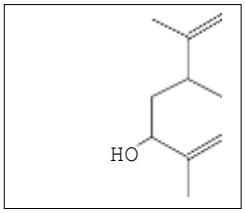


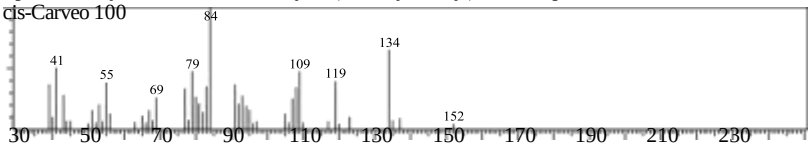


<< Target >>
Line#:8 R.Time:10.815(Scan#:1564) BasePeak:84.05(2344)
RawMode:Averaged 10.810-10.820(1563-1565) BG Mode:Averaged 10.850-10.890(1571-1579)
100

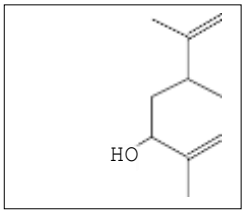


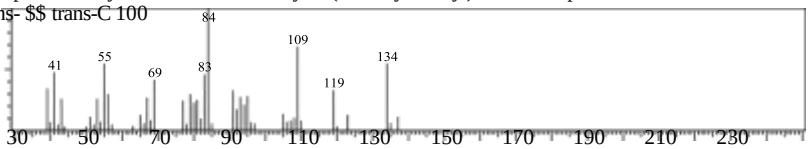
Hit#:1 Entry:16278 Library:NIST08.LIB
SI:87 Formula:C10H16O CAS:1197-06-4 MolWeight:152 RetIndex:1206
CompName:2-Cyclohexen-1-ol, 2-methyl-5-(1-methylethenyl)-, cis- $$ p-Mentha-6,8-dien-2-ol, cis- $$ cis-Carveo 100



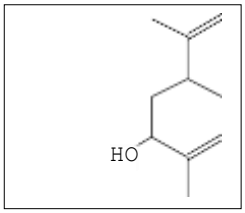


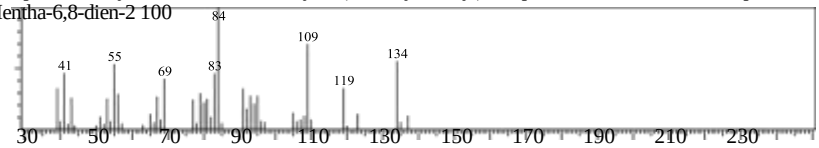
Hit#:2 Entry:9190 Library:NIST08s.LIB
SI:87 Formula:C10H16O CAS:1197-07-5 MolWeight:152 RetIndex:1206
CompName:2-Cyclohexen-1-ol, 2-methyl-5-(1-methylethenyl)-, trans- $$ p-Mentha-6,8-dien-2-ol, trans- $$ trans-C 100

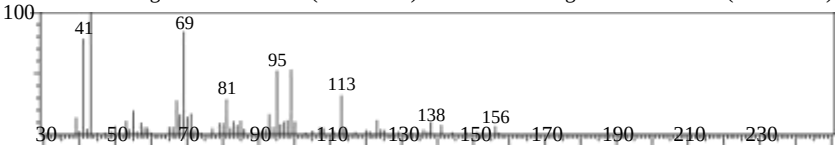


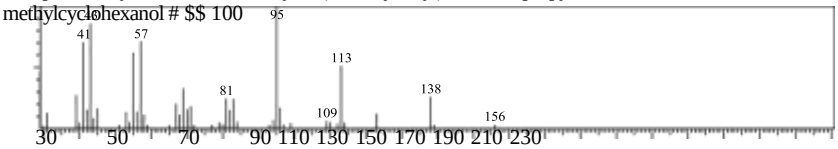


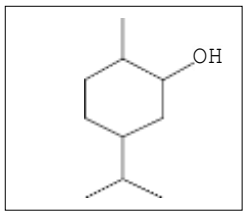
Hit#:3 Entry:9191 Library:NIST08s.LIB
SI:87 Formula:C10H16O CAS:99-48-9 MolWeight:152 RetIndex:1206
CompName:2-Cyclohexen-1-ol, 2-methyl-5-(1-methylethenyl)- $$ p-Mentha-1,8-dien-6-ol $$ p-Mentha-6,8-dien-2 100

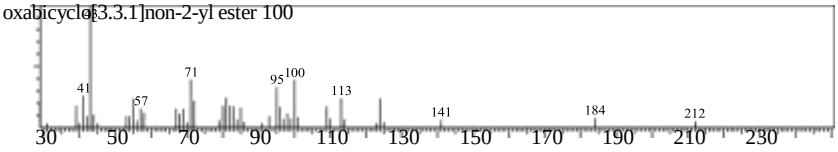


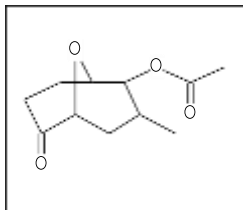


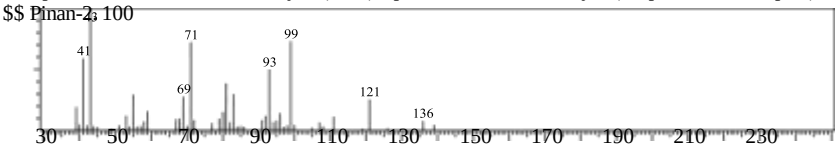
<< Target >>
Line#:9 R.Time:11.690(Scan#:1730)
RawMode:Averaged 11.675-11.710(1736-1743) BG Mode:Averaged 11.645-11.660(1730-1733)


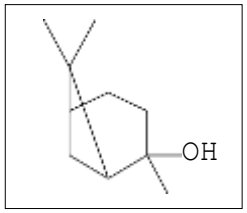
Hit#:1 Entry:9973 Library:NIST08s.LIB
SI:77 Formula:C10H20O CAS:60320-28-7 MolWeight:156 RetIndex:1164
CompName:Cyclohexanol, 2-methyl-5-(1-methylethyl)- $$ 5-Isopropyl-2-methylcyclohexanol # $$ 100


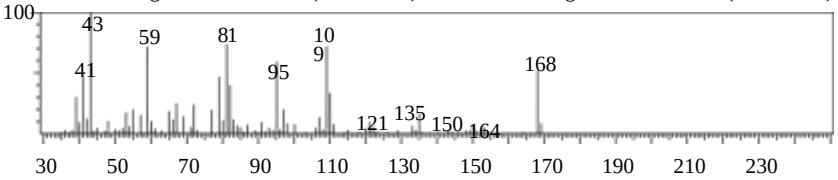


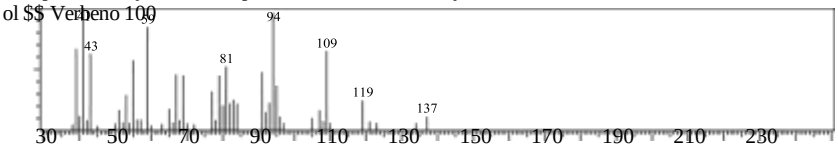
Hit#:2 Entry:50469 Library:NIST08.LIB
SI:76 Formula:C11H16O4 CAS:0-00-0 MolWeight:212 RetIndex:1531 CompName:Acetic acid, 3-methyl-6-oxo-9-oxabicyclo[3.3.1]non-2-yl ester 100


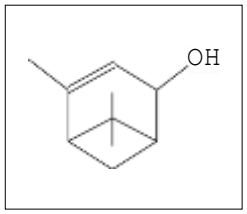


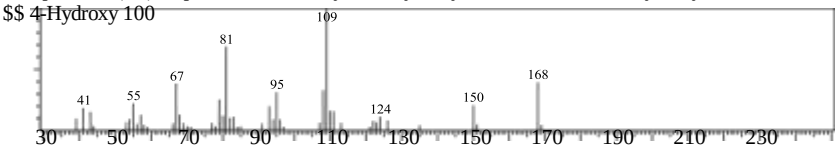
Hit#:3 Entry:17311 Library:NIST08.LIB
SI:76 Formula:C10H18O CAS:4948-29-2 MolWeight:154 RetIndex:1088
CompName:trans-2-Pinanol $$ Bicyclo(3.1.1)heptan-2-ol, 2,6,6-trimethyl-, (1.alpha.,2.beta.,5.alpha.)- $$ Pinan-2-ol 100


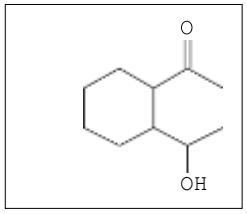


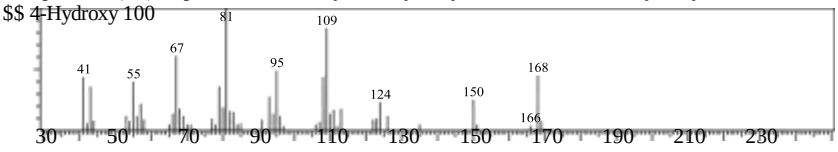
<< Target >>
Line#:10 R.Time:11.825(Scan#:1766) BasePeak:43.05(780)
RawMode:Averaged 11.805-11.835(1762-1768) BG Mode:Averaged 11.780-11.790(1757-1759)


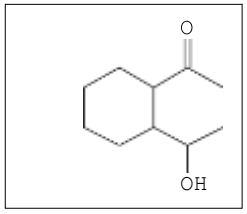
Hit#:1 Entry:9123 Library:NIST08s.LIB
SI:71 Formula:C10H16O CAS:473-67-6 MolWeight:152 RetIndex:1136
CompName:Bicyclo[3.1.1]hept-3-en-2-ol, 4,6,6-trimethyl- $$ d-Verbenol $$ Berbenol $$ 2-Pinen-4-ol $$ Verbeno 100




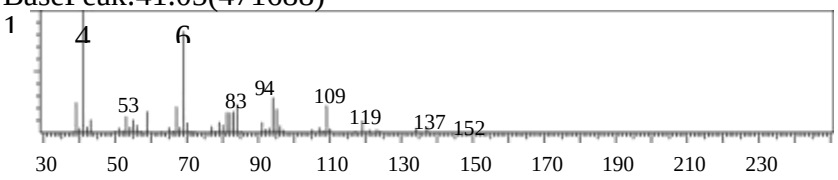
Hit#:2 Entry:24309 Library:NIST08.LIB
SI:70 Formula:C10H16O2 CAS:21766-50-7 MolWeight:168 RetIndex:1461
CompName:1(2H)-Naphthalenone, octahydro-4-hydroxy-, trans- $$ 1-Keto-4-hydroxy-trans-decalin $$ 4-Hydroxy 100




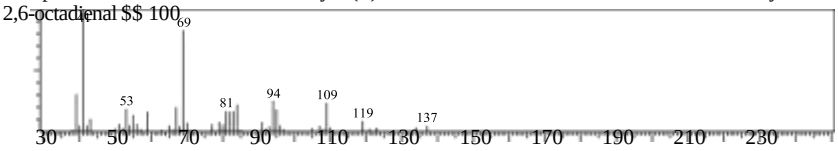
Hit#:3 Entry:11890 Library:NIST08s.LIB
SI:70 Formula:C10H16O2 CAS:21766-50-7 MolWeight:168 RetIndex:1461
CompName:1(2H)-Naphthalenone, octahydro-4-hydroxy-, trans- $$ 1-Keto-4-hydroxy-trans-decalin $$ 4-Hydroxy 100


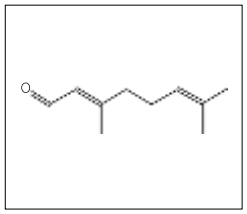


<< Target >>
Line#:11 R.Time:11.965(Scan#:1794)
BasePeak:41.05(471688)

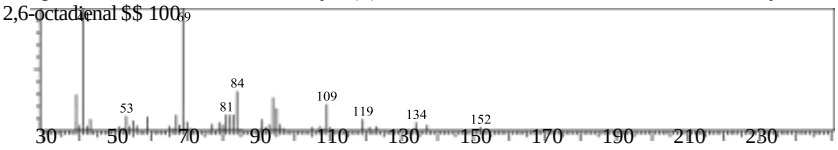


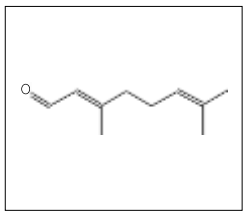
Hit#:1 Entry:9118 Library:NIST08s.LIB
SI:98 Formula:C10H16O CAS:106-26-3 MolWeight:152 RetIndex:1174
CompName:2,6-Octadienal, 3,7-dimethyl-, (Z)- $$ .beta.-Citral $$ cis-Citral $$ cis-3,7-Dimethyl-2,6-octadienal $$ 100



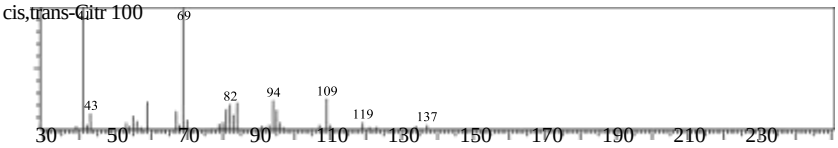


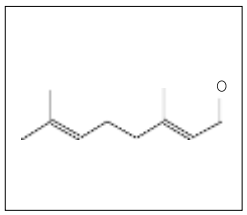
Hit#:2 Entry:9150 Library:NIST08s.LIB
SI:95 Formula:C10H16O CAS:106-26-3 MolWeight:152 RetIndex:1174
CompName:2,6-Octadienal, 3,7-dimethyl-, (Z)- $$ .beta.-Citral $$ cis-Citral $$ cis-3,7-Dimethyl-2,6-octadienal $$ 100



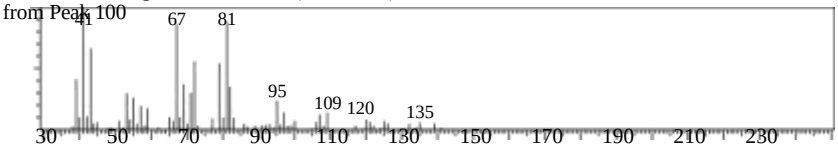


Hit#:3 Entry:9121 Library:NIST08s.LIB
SI:93 Formula:C10H16O CAS:5392-40-5 MolWeight:152 RetIndex:1174
CompName:2,6-Octadienal, 3,7-dimethyl- $$ Citral $$ 3,7-Dimethyl-2,6-octadienal $$ Citral,c&t $$ cis,trans-Citr 100

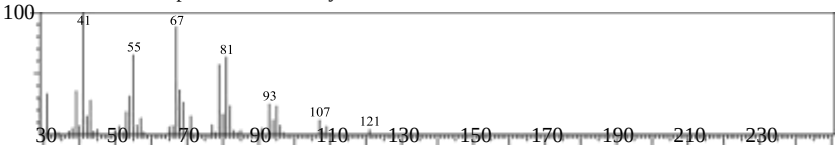


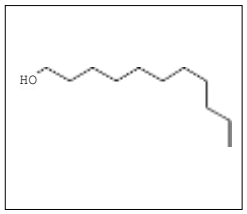


<< Target >>
Line#:12 R.Time:12.365(Scan#:1874) BasePeak:41.10(1580)
RawMode:Averaged 12.360-12.370(1873-1875) BG Mode:Calc. from Peak 100

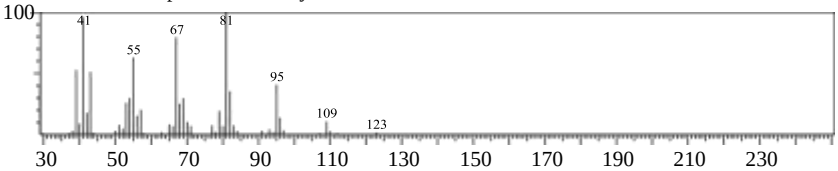


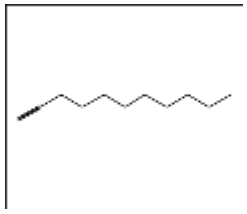
Hit#:1 Entry:11910 Library:NIST08s.LIB
SI:82 Formula:C11H20O CAS:2774-84-7 MolWeight:168
RetIndex:1355 CompName:10-Undecyn-1-ol



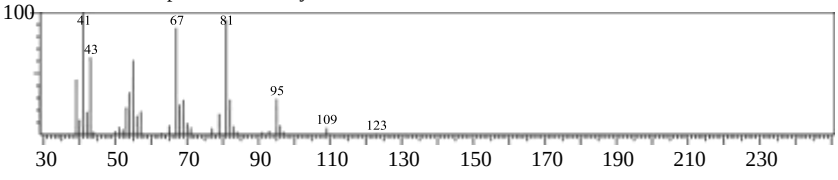


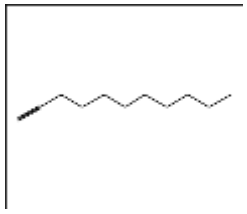
Hit#:2 Entry:9243 Library:NIST08s.LIB
SI:82 Formula:C11H20 CAS:2243-98-3 MolWeight:152
RetIndex:1112 CompName:1-Undecyne $$ 1-C11H20 $$



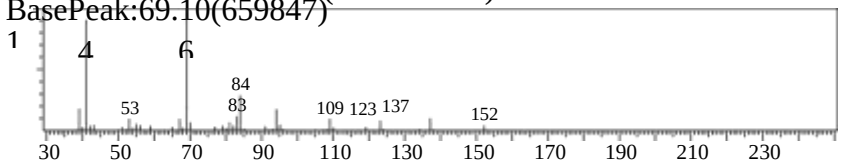


Hit#:3 Entry:9233 Library:NIST08s.LIB
SI:82 Formula:C11H20 CAS:2243-98-3 MolWeight:152
RetIndex:1112 CompName:1-Undecyne $$ 1-C11H20 $$

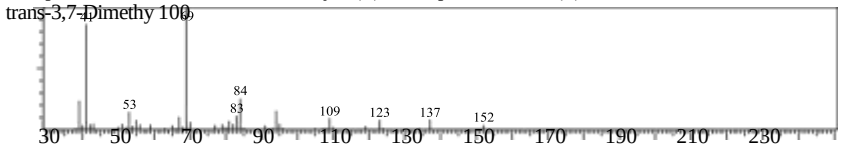


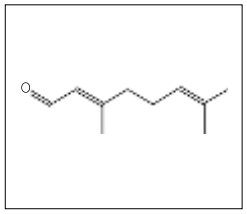


<< Target >>
Line#:13 R.Time:12.560(Scan#:1913)
BasePeak:69.10(659847)

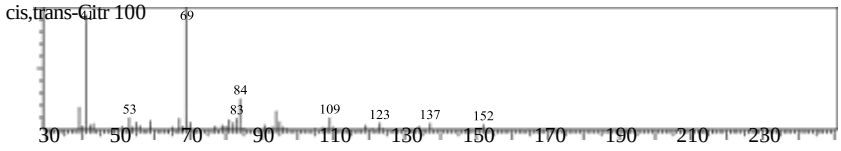


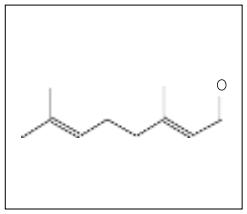
Hit#:1 Entry:9151 Library:NIST08s.LIB
SI:98 Formula:C10H16O CAS:141-27-5 MolWeight:152 RetIndex:1174
CompName:2,6-Octadienal, 3,7-dimethyl-, (E)- $$ .alpha.-Citral $$ (E)-Citral $$ trans-Citral $$ trans-3,7-Dimethy 100



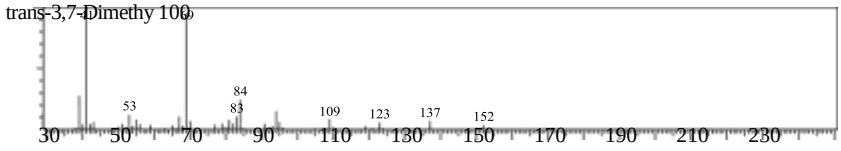


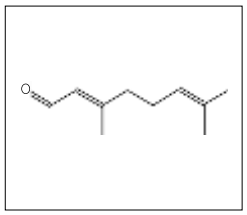
Hit#:2 Entry:9148 Library:NIST08s.LIB
SI:97 Formula:C10H16O CAS:5392-40-5 MolWeight:152 RetIndex:1174
CompName:2,6-Octadienal, 3,7-dimethyl- $$ Citral $$ 3,7-Dimethyl-2,6-octadienal $$ Citral,c&t $$ cis,trans-Citr 100



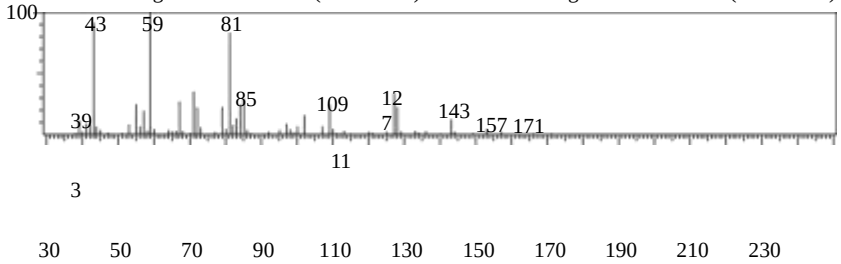


Hit#:3 Entry:16177 Library:NIST08.LIB
SI:96 Formula:C10H16O CAS:141-27-5 MolWeight:152 RetIndex:1174
CompName:2,6-Octadienal, 3,7-dimethyl-, (E)- $$ .alpha.-Citral $$ (E)-Citral $$ trans-Citral $$ trans-3,7-Dimethy 100

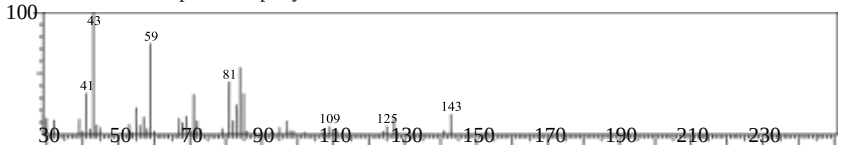


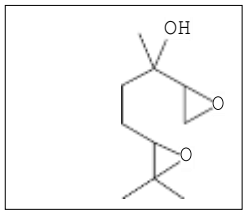


<< Target >>
Line#:14 R.Time:12.810(Scan#:1963) BasePeak:59.05(2345)
RawMode:Averaged 12.805-12.815(1962-1964) BG Mode:Averaged 12.745-12.755(1950-1952)
100

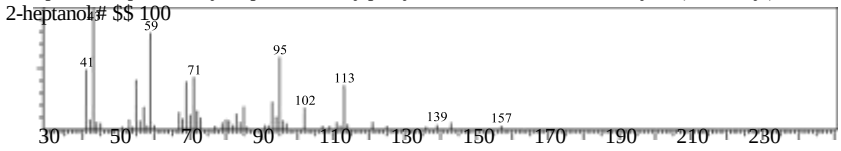


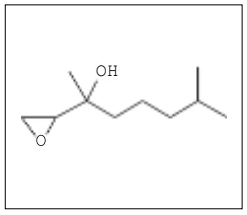
Hit#:1 Entry:34351 Library:NIST08.LIB
SI:84 Formula:C10H18O3 CAS:0-00-0 MolWeight:186
RetIndex:1224 CompName:Epoxy-linalooloxide
100



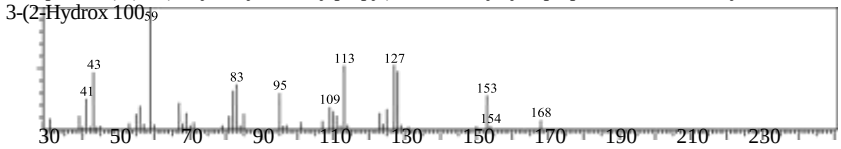


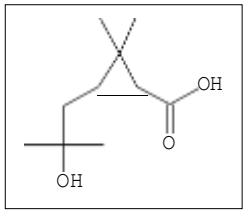
Hit#:2 Entry:26633 Library:NIST08.LIB
SI:72 Formula:C10H20O2 CAS:107358-56-5 MolWeight:172 RetIndex:1133
CompName:.alpha.-Methyl-.alpha.-[4-methylpentyl]oxiranmethanol $$ 6-Methyl-2-(2-oxiranyl)-2-heptanol# $$ 100



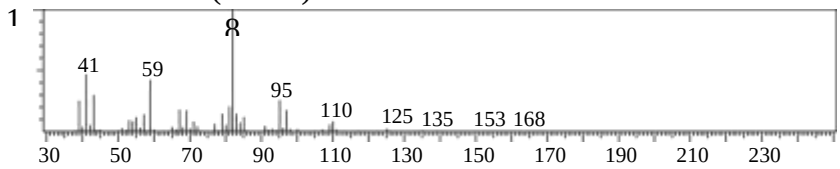


Hit#:3 Entry:34384 Library:NIST08.LIB
SI:71 Formula:C10H18O3 CAS:56941-78-7 MolWeight:186 RetIndex:1363
CompName:(+-)-3-(2-Hydroxy-2-methylpropyl)-2,2-dimethylcyclopropane-trans-1-carboxylic acid $$ 3-(2-Hydrox 100

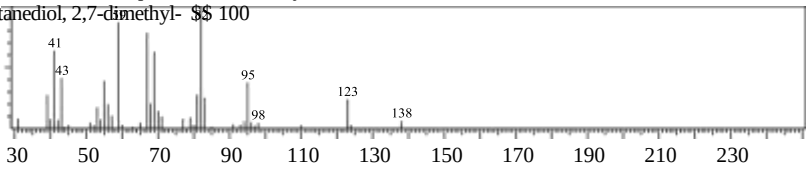


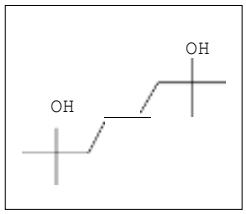


<< Target >>
Line#:15 R.Time:14.170(Scan#:2235)
BasePeak:82.05(20256)

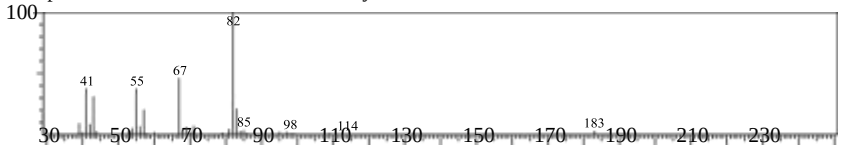


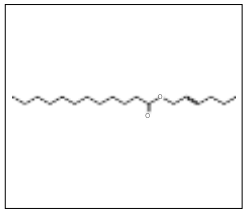
Hit#:1 Entry:27681 Library:NIST08.LIB
SI:82 Formula:C10H22O2 CAS:19781-07-8 MolWeight:174
RetIndex:1199 CompName:2,7-Dimethyl-2,7-octanediol $$ 2,7-Octanediol, 2,7-dimethyl- $$ 100



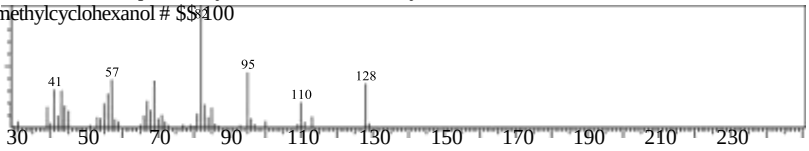


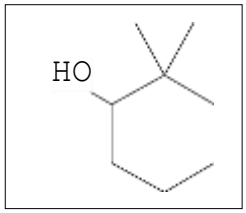
Hit#:2 Entry:99191 Library:NIST08.LIB
SI:81 Formula:C18H34O2 CAS:0-00-0 MolWeight:282 RetIndex:1986
CompName:Dodecanoic acid, 2-hexen-1-yl ester



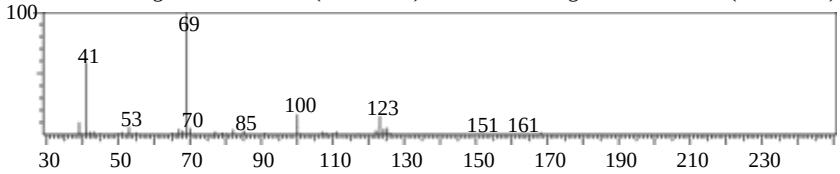


Hit#:3 Entry:7372 Library:NIST08.LIB
SI:81 Formula:C8H16O CAS:1193-46-0 MolWeight:128
RetIndex:1042 CompName:Cyclohexanol, 2,2-dimethyl- $$ 2,2-Dimethylcyclohexanol # $$ 100

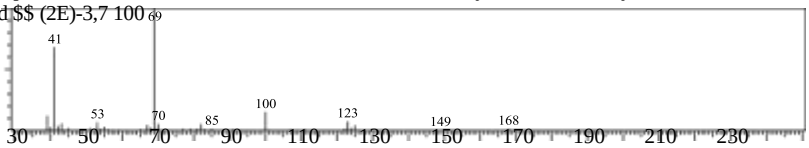


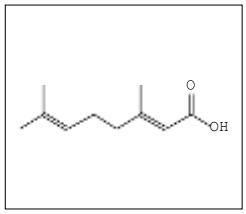


<< Target >>
Line#:16 R.Time:14.745(Scan#:2350) BasePeak:69.10(2943)
RawMode:Averaged 14.665-14.805(2334-2362) BG Mode:Averaged 14.560-14.625(2313-2326)

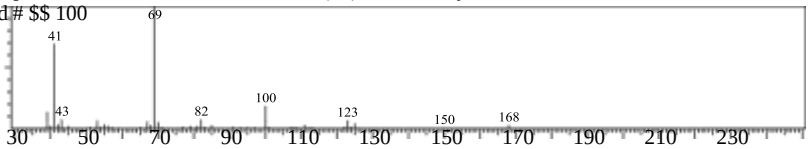


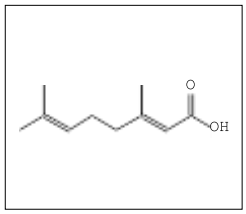
Hit#:1 Entry:24217 Library:NIST08.LIB
SI:93 Formula:C10H16O2 CAS:459-80-3 MolWeight:168 RetIndex:1342
CompName:Geranic acid $$ 2,6-Octadienoic acid, 3,7-dimethyl- $$ 3,7-Dimethyl-2,6-octadienoic acid $$ (2E)-3,7 100



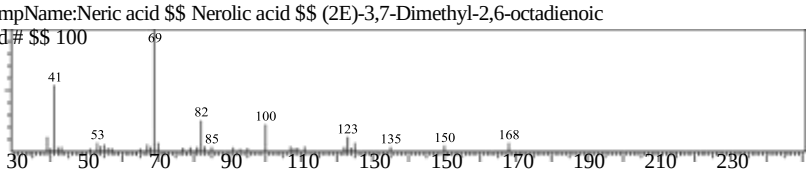


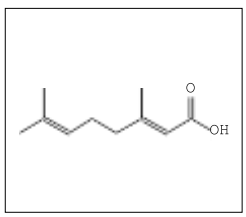
Hit#:2 Entry:24216 Library:NIST08.LIB
SI:92 Formula:C10H16O2 CAS:4613-38-1 MolWeight:168 RetIndex:1342
CompName:Neric acid $$ Nerolic acid $$ (2E)-3,7-Dimethyl-2,6-octadienoic acid # $$ 100



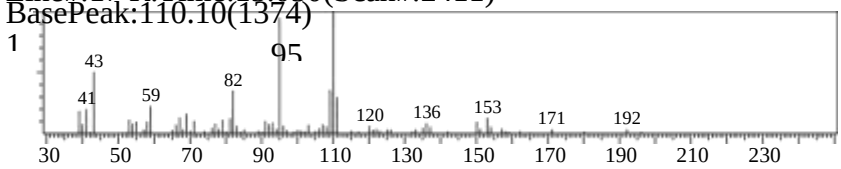


Hit#:3 Entry:11886 Library:NIST08s.LIB
SI:89 Formula:C10H16O2 CAS:4613-38-1 MolWeight:168 RetIndex:1342
CompName:Neric acid $$ Nerolic acid $$ (2E)-3,7-Dimethyl-2,6-octadienoic acid # $$ 100

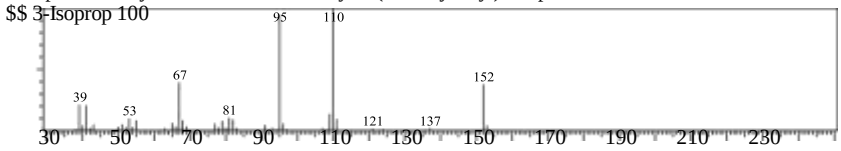


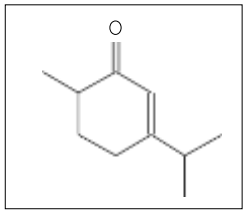


<< Target >>
Line#:17 R.Time:15.050(Scan#:2411)
BasePeak:110.10(1374)

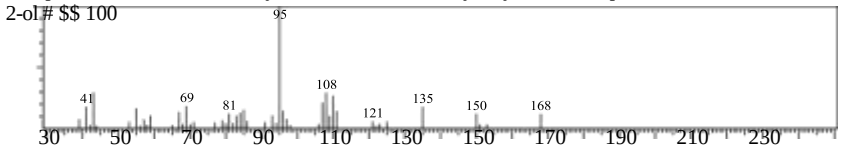


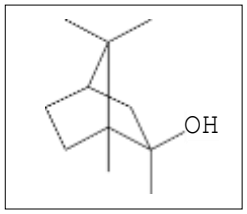
Hit#:1 Entry:16346 Library:NIST08.LIB
SI:74 Formula:C10H16O CAS:499-74-1 MolWeight:152 RetIndex:1158
CompName:2-Cyclohexen-1-one, 6-methyl-3-(1-methylethyl)- $$ p-Menth-3-en-2-one $$ Carvenone $$ 3-Isoprop 100



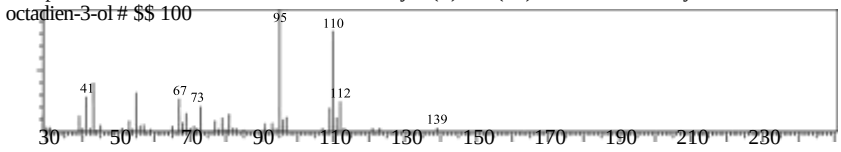


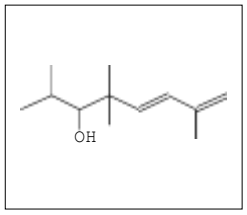
Hit#:2 Entry:24459 Library:NIST08.LIB
SI:74 Formula:C11H20O CAS:91278-70-5 MolWeight:168 RetIndex:1161
CompName:2-Bornanol, 2-methyl- $$ 1,2,7,7-Tetramethylbicyclo[2.2.1]heptan-2-ol # $$ 100



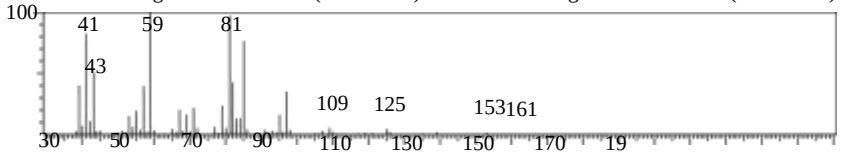


Hit#:3 Entry:32405 Library:NIST08.LIB
SI:73 Formula:C12H22O CAS:77142-78-0 MolWeight:182 RetIndex:1203
CompName:5,7-Octadien-3-ol, 2,4,4,7-tetramethyl-, (E)- $$ (5E)-2,4,4,7-Tetramethyl-5,7-octadien-3-ol # $$ 100

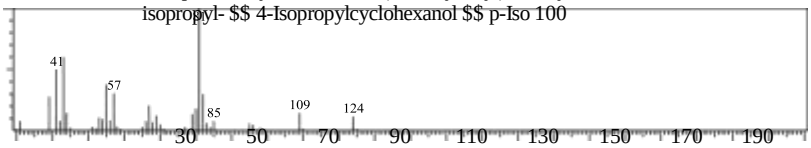


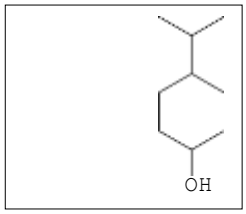


<< Target >>
Line#:18 R.Time:15.245(Scan#:2450) BasePeak:59.05(7456)
RawMode:Averaged 15.220-15.300(2445-2461) BG Mode:Averaged 15.175-15.210(2436-2443)

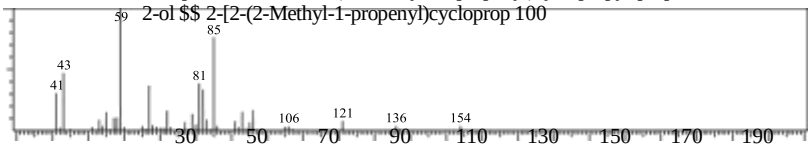


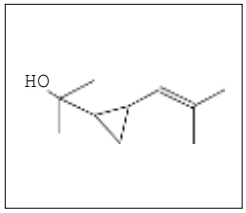
Hit#:1 Entry:12230 Library:NIST08.LIB
SI:81 Formula:C9H18O CAS:4621-04-9 MolWeight:142 RetIndex:1103
CompName:Cyclohexanol, 4-(1-methylethyl)- $$ Cyclohexanol, 4-isopropyl- $$ 4-Isopropylcyclohexanol $$ p-Iso 100





Hit#:2 Entry:17342 Library:NIST08.LIB
SI:81 Formula:C10H18O CAS:38996-80-4 MolWeight:154 RetIndex:1056
CompName:trans-2-[2'-(2''-Methyl-1''-propenyl)cyclopropyl]propan-2-ol $$ 2-[2-(2-Methyl-1-propenyl)cycloprop 100





Hit#:3 Entry:5087 Library:NIST08s.LIB
SI:80 Formula:C8H16O CAS:4534-74-1 MolWeight:128 RetIndex:1068
CompName:4-Ethylcyclohexanol $$ 4-Ethylcyclohexanol,c&t $$ Cyclohexanol, 4-ethyl- $$ trans-4-Ethyl cyclohex 100