AJAB

Asian J Agric & Biol. 2019;7(1): 93-102.

Original Article

# Effect of different drying methods on the morphological structure, colour profile and citral concentration of Lemongrass (C*ymbopogon citratus*) powder

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*Received:*
*September 29, 2018*

*Accepted:*
*January 31, 2019*

*Published:*
*March 30, 2019*

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## Abstract

Lemongrass is a well-known aromatic herb due to its strong lemony odour which contributes to several volatile compounds such as citral, β–myrcene and limonene. Volatile compounds of the aromatic herb are consequently difficult to restrain due to high volatility and adverse effect caused by thermal treatment applied during processing. Therefore, this study was conducted to determine the effect of different drying methods on the morphological structure, colour profile and citral concentration of lemongrass powder. Lemongrass powder was prepared by drying the fresh lemongrass stalks with oven drying, vacuum drying and freeze-drying. The yield of lemongrass powder resulted after drying processes were in the range of 9.92-11.09%. The morphology structures of all lemongrass powders were flake-like structure, irregular size, shrunk and appeared of pores. The freeze-dried powder was brighter in colour with L* value of 84.51 ± 1.64 and obtained the highest citral concentration of 321.41 ± 19.97 ppm. This study suggested that freeze-drying was the suitable method for preserving the colour qualities and citral compound of lemongrass powder. The freeze-dried powder of lemongrass has high potential to be applied in the food and beverage products.

**Keywords:** Lemongrass, Freeze dried powder, Drying methods, Colour profile, Citral concentration

## How to cite this:

Hashim MA, Yahya F and Mustapha WAW, 2019. Effect of different drying methods on the morphological structure, colour profile and citral concentration of lemongrass (*Cymbopogon citratus*) powder. Asian J. Agric. Biol. 7(1): 93-102.



## Introduction

Lemongrass is a local perennial grass plant known for the strong lemony odour which comes from *Poaceae* family that grows in tropical and subtropical climate countries such as Southeast Asia including Indochina, Indonesia, Malaysia, Sri Lanka, North and Southern India (Francisco et al., 2011). Volatile composition of lemongrass was dominated by citral compound; a combination between two geometric isomers; neral and geranial which contributed to the lemony odour (Pengelly, 2004). According to Bassolé et al. (2011), neral and geranial consist of 34.6% and 48.1% respectively of the total volatile composition of

lemongrass. In addition, Skaria et al. (2012) stated that aroma of lemongrass also contributed by myrcene (12.75%), geranyl acetate (3.0%), methyl heptanene (2.62%), geraniol (1.85%) and β- elemene (1.33%). Previously, parts of lemongrass have been used in traditional folk medicine for treating nervous and gastrointestinal disturbance and as antispasmodic, analgesic, anti-inflammatory, antipyretic, diuretic and sedative (Santin et al., 2009). Lemongrasses are also commonly used in food flavouring (Katsukawa et al., 2010), fragrance (Katsukawa et al., 2010), pharmaceutical (Shah et al., 2011), and cosmetic (Mohamed Hanaa et al., 2012) industry.

Drying and powdering techniques were recommended for preserving and improving the qualities of the product purposes such as extending the shelf-life (Dirim and Caliskan, 2012), inhibiting of product's spoilage, minimizing thermal stress as well as retaining aroma compounds (Hossain et al., 2010; Kubra and Rao, 2012; Dirim and Caliskan, 2012). Drying methods were significant effected the characteristics of dried product in term of microstructure (An et al., 2016; Gasmalla et al., 2017), colour profile (Nikjooy and Hashemi, 2014) and volatile composition (Díaz – Maroto et al., 2002; Mohamad Hanaa et al., 2012). Studies have been done on the effect of drying method such as oven drying (Mohamed Hanaa et al., 2012; Pirbalouti et al., 2013), freeze drying (Díaz – Maroto et al., 2002; Tajidin et al., 2012) and vacuum drying (Kruma et al., 2011) on fresh herbs such as parsley (Díaz – Maroto et al., 2002), Pandan leaves (Yahya et al., 2010) and lemongrass (Mohamad Hanaa et al., 2012). During oven drying process, aroma preservation should take into consideration as a challenge due to volatile susceptibility which may contributed by high temperature used and prolong exposure to the heat (Nawirska et al., 2009). Meanwhile, vacuum drying caused a minimal shrinkage on the structure of product (Alibas, 2007; Alibas, 2009) as well as inhibited pigment degradation and nonenzymatic reaction (Methakhup et al., 2005; Alibas, 2007). Whereas, freeze-dried product exhibited similar colour profile with the fresh ones such as on Goldenberry (Valdenegro et al., 2013) and retained a greater concentration of aroma compounds of parsley (Díaz – Maroto et al., 2002). Therefore, the purpose of this research was to determine the effect of different drying methods on the morphological structure, colour profile and citral concentration of lemongrass powder.

## Material and Methods

The standard of citral compound (assigned as 99.8% purity) was purchased from Sigma-Aldrich Co. (Malaysia). Fresh lemongrass stalks were purchased from a local market in Kuala Nerus, Terengganu, Malaysia.

**Preparation of lemongrass powder**

Fresh lemongrass stalks with moisture content of 80.0 ± 5.0% (w. b) were cleaned and rinsed using tap water to remove dirt and dust prior to cut into 16 cm lengths and chopped into small pieces. The sample was then divided into three dried portions. Each portion (300.0 ± 5.0 g) was dried using three drying methods i.e. oven drying, vacuum drying and freeze drying.

For oven drying, the sample was spread evenly on an aluminium foil on the oven dryer tray. Then, it was dried at 60.0 ± 2.0°C for 24 h in a conventional oven dryer (UFB 500, Memmert, Germany). The second portion of the sample was dried using vacuum-pump oven dryer (ADP-21, Yamato, Japan) under drying temperature of 60.0 ± 2.0°C for 24 h. The third portion of the sample was frozen in the freezer (MDF-U55V-PE, Panasonic, Japan) for 24 h at - 80 ± 2.0°C prior to freeze drying process at - 47.0 ± 2.0°C. The heating plate of freeze dryer was automated to 20.0 ± 3.0°C on vacuum degree at 0.203 kPa.

After all the drying processes ended, the dried lemongrass was ground into powder form for 30 s by using mill grinder (IKA® Werke Staufen - MF 10.21, Germany) prior to storage at 4.0 ± 1.0°C in zipping lock polyethylene bag prior to further analysis.

**Determination of total yield of lemongrass powder**

Sieving analysis was carried out on modified method from Capariño et al. (2012) by stacking and vibrating the sieve on the sieve shaker (Retsch Model AS 200 digit, Germany) with test sieve (size of 250 μm) at the amplitude of 60 for 20 min. Then, sieved samples were collected, and the final weight was recorded. The total yield of lemongrass powder was calculated by the following equation:

Total yield of lemongrass powder (%) =

$$\frac{\text{Weight of sieved powder (g)}}{\text{Initial weight of chopped lemongrass stalk (g)}} \times 100$$

**Determination of moisture content**

A 2.0 ± 0.5 g of lemongrass powder were weighed into the crucible. The sample was dried using oven drying method (AOAC, 2000) in the conventional oven (BINDER, ED400, Germany) at 105 ± 2.0°C for 24 h. Moisture content of lemongrass powder was determined by the equation below;

Percentage of moisture content (%) =

$$\frac{\text{Weight of powder before drying (g)} - \text{Weight of powder after drying (g)}}{\text{Weight of powder before drying (g)}} \times 100$$

**Determination of colour profile**

The colour profile of the lemongrass powder was determined by using Minolta CR300 chroma meter (Konica-Minolta, Japan) in term of *L**, *a** and *b** values in triplicate analyses as adapted method from Arslan and Özcan, (2008). It was calibrated by using a standard calibration with a white tile. A 25.0 ± 1.0 g of lemongrass powder was packed in the zip-lock polyethylene bag. The chromameter was put directly on the packed of lemongrass powder.

**Determination of morphology structure**

The morphology structure of lemongrass powder was observed under Scanning Electron Microscope (JEOL JSM – 6360, Japan) as adapted from Tonon et al. (2008) to identify the differences occurred by different drying methods. It was coated with 3.5 mA gold/palladium under vacuum condition by using auto fine coater (JEOL JFC – 1600, Japan). Then, it was examined and operated at an accelerating voltage of 5kV under 300× magnification.

**Identification and quantification of citral concentration**

Quantitative analysis of citral compound of standard solution were performed as method modified from Pellati et al. (2005) by headspace–solid phase microextraction (HS-SPME) accompanied by gas chromatography-flame ionization detector (GC-FID). Stock solution of citral compound (20.0 ± 0.05 ml of 1000 ppm) was prepared by diluted the standard compound with distilled water. Then, the stock solution was diluted with another distilled water for preparing 20 ml of the standard solution of citral compound with series of concentrations of 25, 50, 75, 100 and 125 ppm.

The standard solution of citral compound (1.0 ± 0.05 ml) was measured in 45 ml of headspace vial. Heating block was heated to equilibrate the temperature surrounding the vial. Extraction of citral compound was conducted at 60.0 ± 2.0°C for 30 min by using carboxen/ polydimethylsiloxane (CAR/PDMS) fibre. CAR/PDMS fibre was then immediately inserted into the injection port of the gas chromatography (GC 2010 - Shimadzu, Japan) for 5 min at 250°C. Gas chromatography-flame ionization detector (GC – FID) analyses were carried out by GC 2010 (Shimadzu, Japan) within operation started at 60°C, increased 4°C/min to 150°C, then to 250°C at 20°C/min and equilibrium for 5 min. A BPX-5 capillary column (30 m × 0.25 mm internal diameter, 0.25 µm film thickness) was used in this analysis. The chromatographic conditions were as follows: helium (carrier gas) flow at 30.0 ml/min, the temperature of the injector at 250°C, temperature detector at 250°C with pressure of 100 kPa.

The extraction of citral compound of the lemongrass powders were performed by similar procedure used for standard solution. Fresh lemongrass was used as a control. The two isomers of citral; neral and geranial, were identified by comparing retention times to authentic standards. While concentration of citral compound was expressed as the sum of concentration of neral and geranial obtained from separated standard curve (concentration vs peak area).

**Statistical analysis**

Data of samples were analyzed using MINITAB 14 software (MINITAB Inc., State College, PA, USA) at $p < 0.05$. The one-way analysis of variance (One – way ANOVA) was used to determine the differences between mean values of samples, followed by Fisher's Least Significant Difference (LSD) test to determine the significant differences among samples.

## Results and Discussion

**Total yield of lemongrass powder**

Table 1 shows the total yield of powder, moisture content and colour profile of lemongrass powders prepared by different drying methods. As can be seen in Table 1, the total yield of lemongrass powders from different drying methods were in the range of 9.92-11.09% and the oven drying produced higher yield of lemongrass powder compared to other drying conditions. This result was in contrast with studies done by Kim et al. (2006) and Lee et al. (2012a) on Citrus "*Hallabong*" in which the author and co-workers reported that a higher yield of powder obtained by freeze-dried sample (20.0-22.5%) when

compared to oven dried ones (17.5-22.0%). This study also found that the total yield of freeze-dried was higher when compared to vacuum-dried powder and this result was in good agreement with study done by Rabeta and Lai, (2013) on total yield of *Ocimum tenuiflorum* powder. However, the total yields of lemongrass powder were not significantly affected ($p>0.05$) by different drying methods (Table 1). As expected, similar drying temperature of oven drying and vacuum drying at 60°C as well as constant in particle size of powder (250 µm) might cause an insignificant result of the yield of lemongrass powder.

**Moisture content of lemongrass powder**

Moisture content of lemongrass powders from different drying methods is shown in Table 1. The moisture content of all lemongrass powders and fresh lemongrass were determined by oven drying at 105°C for 24 h. As can be seen, the moisture content of fresh lemongrass was 88.54 ± 3.08% while the moisture content of lemongrass powders was ranged from 8.64-10.26%. As expected, the fresh lemongrass was experienced water removal up to 88% during the dehydration process. It was typical for the fresh plant had up to 80% of water removal as it was in good agreement with study done by Carpenter and Carpenter, (2015) who reported that in the range 50-90% of moisture content in fresh plants was lost during dehydration. This result also in line with 80% of water removal from fresh pandan leaves after drying processes (Yahya et al., 2010).

The different drying methods were not significant influenced ($p>0.05$) on the moisture content of lemongrass powder. But, vacuum-dried powder showed high in moisture content (10.26 ± 1.39%) followed by freeze-dried and the oven-dried powder with 9.44 ± 0.29% and 8.66 ± 1.03% of moisture content respectively (Table 1). It was in good agreement with study done by Wijewardane et al., (2015) on dried pumpkin but in contrast with Neoh et al., (2016) who reported that the moisture content of freeze dried red seaweed was higher (11.19%) compared to oven dried (10.41%) and vacuum dried (10.31%) ones. According to Yousif et al., (1999) and Artnaseaw et al., (2010) drying temperature and time were affected the rate of water evaporation during moisture removal on the heat transfer into the interior of the material. Therefore, no significant effect was observed on moisture content of oven dried and vacuum dried lemongrass powder in where both methods use the same temperature of 60°C as previously reported by Arslan and Özcan, (2010) on *Allium cepa.* L.

**Colour profile of lemongrass powders**

There was a significant difference ($p<0.05$) of the colour profile of lemongrass powder between different drying methods. Freeze-dried lemongrass powder exhibited the highest *L** value with lower *a** and *b** values as compared to oven-dried and vacuum-dried lemongrass powders. It is because of usage of low drying temperature that prevented the enzymatic browning to be occurred (Krokida and Maroulis, 2000), thus produced brighter colour of powder. The freeze-dried lemongrass powder obtained similar effect on the *L** value with various freeze-dried powders from previous studies such as on mango (Capariño et al., 2012), Citrus "Hallabong" (Lee et al., 2012a), chilli (Toontom et al., 2012) and apple (Antal, 2015).

**Table 1: Physical properties (n=3) of lemongrass powders from different drying methods**

| Lemongrass powder | Total yield of powder (%) | Moisture content (%) | Colour profile | | |
|---|---|---|---|---|---|
| | | | L* | a* | b* |
| **Control (fresh)** | - | 88.54 ± 3.08$^{a}$ | - | - | - |
| **Oven dried** | 11.09 ± 1.33$^{a}$ | 8.66 ± 1.03$^{b}$ | 74.57 ± 1.73$^{b}$ | 5.31 ± 0.38$^{a}$ | 20.76 ± 1.74$^{a}$ |
| **Vacuum dried** | 9.92 ± 0.94$^{a}$ | 10.26 ± 1.39$^{b}$ | 76.05 ± 2.66$^{b}$ | 2.62 ± 1.78$^{b}$ | 22.41 ± 0.73$^{a}$ |
| **Freeze dried** | 10.81 ± 1.15$^{a}$ | 9.44 ± 0.29$^{b}$ | 84.51 ± 1.64$^{a}$ | -1.14 ± 0.65$^{c}$ | 17.89 ± 0.91$^{b}$ |

Mean values with different superscript letters in the same column are significantly different at $p<0.05$.

Nevertheless, the *L** values on oven-dried and vacuum-dried lemongrass were insignificantly ($p>0.05$) affected by the drying methods (Table 1). It is due to low heating temperature applied (60°C) during dehydration process which may inhibits Maillard reaction and non-enzymatic reaction from occurred (Artnaseaw et al., 2010). Table 1 also shows the highest *a** value and *b** value of lemongrass powder were obtained from oven drying (5.31 ± 0.38) and vacuum drying (22.41 ± 0.73) treatment, respectively. According to Maskan, (2001), chlorophyll and non-enzymatic reaction are responsible for the alteration of the colour profile of *a** and *b**. Therefore, these results may contribute by loss of green colour due to the degradation of chlorophyll pigments during drying process (Guiné and Barroca, 2012; Sledz and Witrowa – Rajchert, 2012) which may lead to cause another pigment such as carotenoid becomes more visible (Sledz and Witrowa – Rajchert, 2012).

**Morphology structure of lemongrass powders**

The morphological structure of oven-dried, vacuum-dried and freeze-dried lemongrass powder is shown in Figure 1. The structure of oven-dried lemongrass powder was observed to be flake, appeared more shrinkage and collapsed of cellular tissue (Figure 1a). The pointy - edge of small particles with irregular structure was performed. The structure of oven dried (Figure 1a) and vacuum dried (Figure 1b) of lemongrass powder were heat-dried Shiitake mushroom-like structure studied by Tian et al. (2016). According to Lee et al. (2012b), wrinkles were formed on the dried structure due to the increasing of drying temperature which promotes an increase of moisture stress and drying rate. Apart from that, vacuum drying technique caused the formation of small channels on side of the lemongrass powder. The vacuum condition was allowed rapid moisture transfer to the surrounding area by creating high vapour pressure between the product and the drying chamber and causing the cell to swell and formed large channels inside the sample (Kantrong et al., 2014; Tian et al. 2016).

Furthermore, the freeze-dried lemongrass powder appeared a skeletal-like structure with slightly smooth and flaky with large porous (circled structure of Figure 1c). The microstructure of freeze-dried lemongrass was similar with porous structure on the freeze-dried button mushroom as studied by Argyropoulos et al., (2011). Freeze-dried lemongrass powder experienced less breakage due to the protection effect provided by ice crystals those surrounding the material which was developed during freezing (-80°C) prior to drying in the chamber as explained by Ratti, (2001).

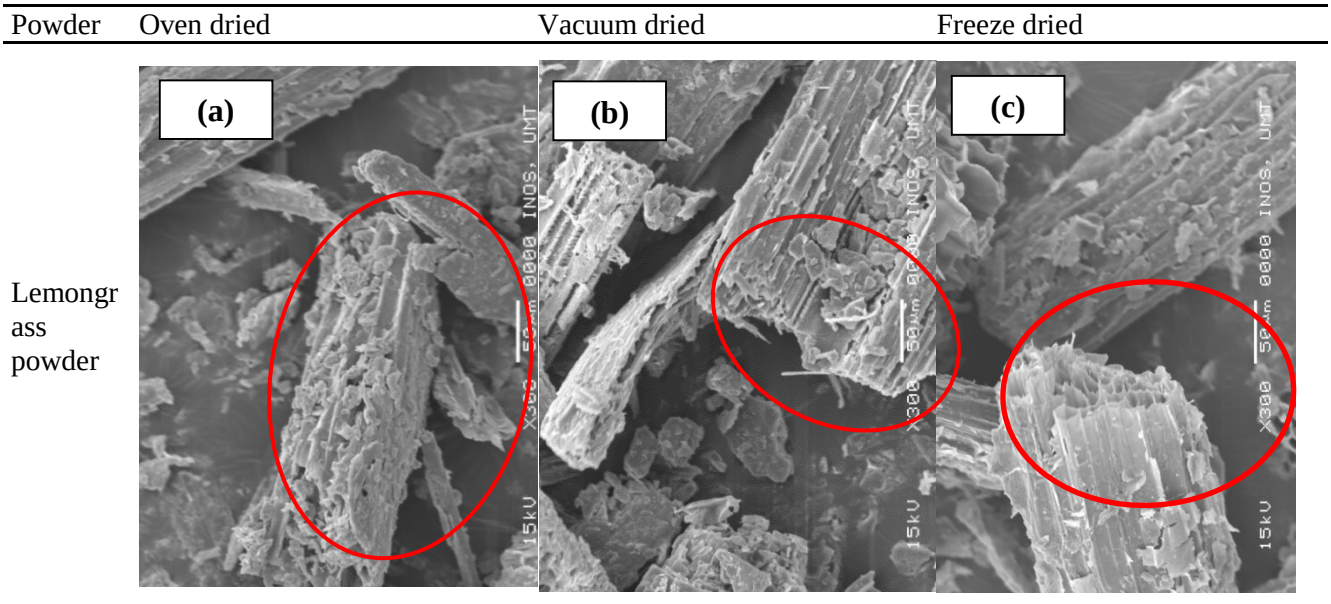


**Fig. 1. Scanning electron microgram of lemongrass powders from different drying methods at 300× magnification (red circle indicated the changes of the microstructure of lemongrass powder)**

**Citral concentration of lemongrass powders**

The gas chromatogram of standard citral compound, citral from fresh lemongrass and citral from lemongrass powders were shown in Figure 2a, Figure 2b and 2c-e, respectively. The isomers of citral compound; neral and geranial were detected at retention time 14.9 min and 16.0 min, respectively. Various peaks were detected after peak detection of neral and geranial of fresh lemongrass (Figure 2b).

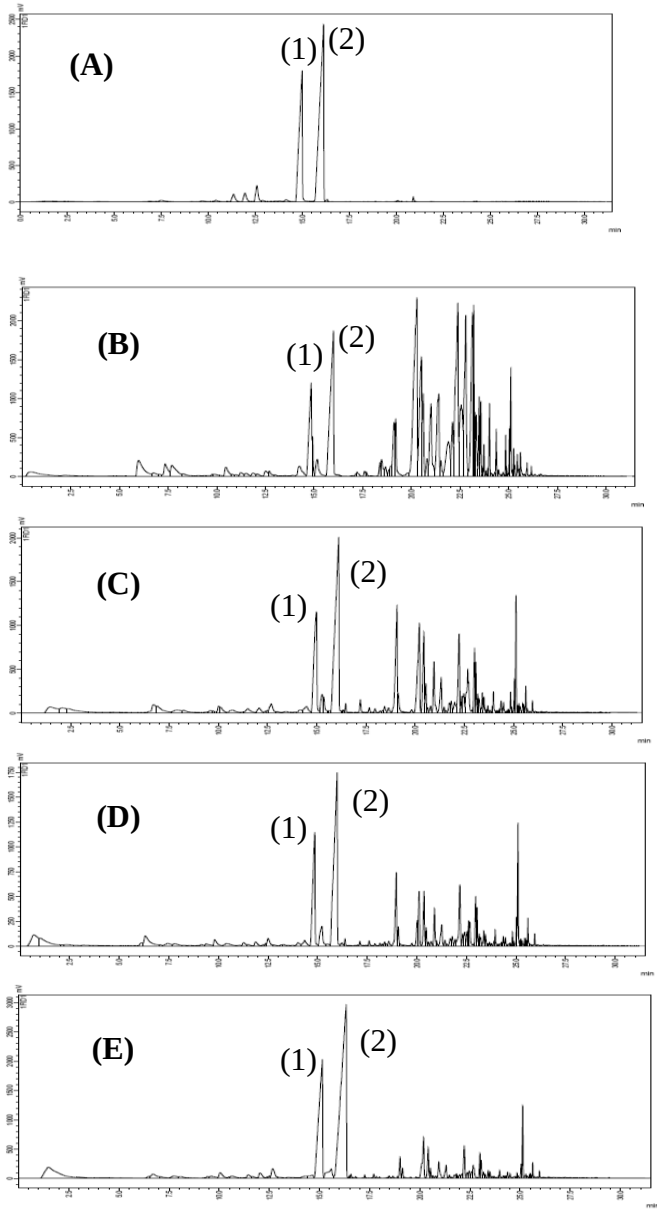


**Fig. 2. Gas Chromatography-Flame Ionization Detector chromatogram of A) Standard Citral, B) Fresh lemongrass, C) Oven dried, D) Vacuum dried and E) Freeze dried of lemongrass powder; Peak identification: (1) neral (*cis* – Citral) and (2) geranial (*trans* - citral)**

The unknown peaks were detected during qualitative analysis which indicated that lemongrass is composed of various aroma compounds such as linalool, β-myrcene, caryophyllene, citronellol, juniper camphor and geranyl acetate as identified by Tajidin et al., (2012). The unknown peaks also showed with different levels of peak height. As can be seen in Figure 2b, the unknown peaks of fresh lemongrass showed higher peak height when compared to height of unknown peaks of lemongrass powders (Figure 2c-e). This observation also indicated that volatile compound of lemongrass had greater of peak height from unknown peaks those detected from oven dried and vacuum dried powder when compared to peak height of freeze dried ones.

Figure 3 showed the citral concentration of lemongrass powders from different drying methods. It showed that citral concentration of lemongrass powders was significantly affected ($p<0.05$) by different drying methods applied. The citral concentration of fresh lemongrass was 97.75 ± 7.29 ppm. The range of citral concentration of lemongrass powders was 123.13 – 321.41 ppm. The fresh lemongrass exhibited lower of citral concentration compared to citral concentration of all lemongrass powders. This result was contradicted with volatile composition of other fresh plant (Díaz-Maroto et al., 2002; Calín-Sánchez et al., 2011). This situation happened because of heat temperature and extraction time of headspace solid phase micro-extraction applied in this study were weak to give affect the parenchyma layer and biological structure of the oil glands for citral to evacuate out during extraction of fresh ones. Lewinsohn et al. (1998) found that isomers of citral compound on fresh lemongrass are entrapped in oil glands under parenchyma tissue cell that restricts the citral from release.

Freeze-dried lemongrass powder showed the highest of citral concentration (321.41 ± 19.97 ppm) followed by oven-dried (155.44 ± 16.22 ppm) and vacuum-dried lemongrass (123.13 ± 17.69 ppm) powder. This result can be explained that freeze drying method might preserved the citral compound of lemongrass powder by minimizing degradation of the compound. It was in good agreement with Ebadi et al., (2015) who reported that the composition of citral compound on lemon verbena was well –preserved after freeze-drying at 64.7 % compared to oven drying (60$^{o}$C) and vacuum drying (60$^{o}$C) at 56.7% and 55.5% respectively.

In addition, freezing caused a minor cellular disturbance with inhibiting release of aroma compounds (De Ancos et al., 2000). The enzymatic reaction is preventable and volatile compounds are restricted from release to the surrounding at low temperature (Palacios, 2014). Furthermore, the effect from freezing caused solid phase micro-extraction become more efficient toward the monoterpene of citral adsorption as agreed by Díaz-Maroto et al., (2002) on extracting sesquiterpenes compounds of spathulenol and β-eudesmol from parsley.

Figure 3 also showed that citral concentration of oven-dried and vacuum-dried lemongrass powder was insignificant ($p>0.05$) to each other. Thermal drying leads to oxidation, rearrangement and degradation reaction in the presence of oxygen causing a decrement of the volatile components (Ding et al., 2012). The decrement of citral compound in this study had a similar effect on decrement on monoterpenes of limonene and 1, 8- cineole under heat-oven drying under drying temperature at 60°C (Khangholil and Rezaeinodehi, 2008).

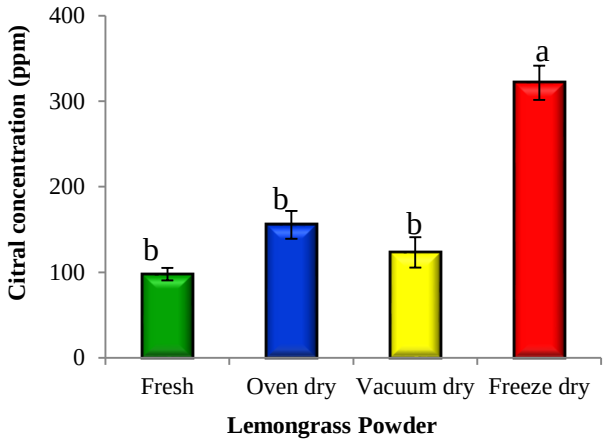


**Fig. 3. Citral concentration (n=3) of lemongrass powder from different drying methods.**
Bar with different superscript letters is significantly different ($p < 0.05$).
F: Fresh lemongrass stalk; OD: Oven dried lemongrass powder, VD: Vacuum dried lemongrass powder, FD: Freeze dried lemongrass powder

The citral concentrations were found to be affected by the microstructure of lemongrass powders. The thermally dried powders were exhibited of collapsed fibre with stress-wrinkle structure. Small pores were detected during microscopic observation on the lemongrass powders (Figure 2). It is possible for volatile compounds to release from the plant's structure to the atmosphere due to the microstructure changes from drying such as through plant's cell expansion (An et al., 2016) as previously observed on the cell structure of the freeze-dried parsley (Díaz – Maroto et al., 2002) and spearmint (Díaz – Maroto et al., 2003).

## Conclusion

In conclusion, this study showed that moisture content and total yield of lemongrass powders were not significantly ($p<0.05$) affected by different drying methods. Freeze drying was able to retain the colour of the lemongrass powder with highest L* value and lowest in both a* and b* values. The scanning electron microscopy images revealed that microstructure of the thermal-dried of lemongrass powders lead to deform and collapse. Freeze dried lemongrass powder obtained the highest concentration of citral compound. This study suggested that freeze dried lemongrass powder potential to be applied in food and beverages product.

## Acknowledgement

The authors are grateful to Universiti Malaysia Terengganu for facilities and financial support from Research Acculturation Collaborative Effort (RACE) grant (Vote No. 56035) to conduct the research project.

## Contribution of Authors

Hashim MA: Conducted experiment, analysed the data and drafted the manuscript (master student for this research project)
Yahya F: Planned the experimental design, read and approved the final draft of manuscript (Leader of this research project/supervisor)
Mustapha WAW: Planned the experimental design, read and revised the manuscript (co-researcher /mentor of this research project)

**Disclaimer:** None.
**Conflict of Interest:** None.
**Source of Funding:** Research Acculturation Collaborative Effort (RACE) grant (Vote No. 56035).

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