Analysis of virgin coconut oil (VCO) components after heating and adding Cymbopogon nardus as the essential oil

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Abstract. Cymbopogon nardus has the potential to be an essential oil, used for kitchen spice, cosmetics (aromatherapy), and cancer medicines which contains citrate, eugenol, citronellol, citronellal, geraniol, citronellyl acetate, geranyl acetate, beta-caryophyllene, limonene, and methyl eugenol. This study aimed to analyze the components of Virgin Coconut Oil (VCO) after heating and adding Cymbopogon nardus compared to commercial products. The method of making coconut oil was enzymatic and heated for about ten minutes. Analysis of VCO components after being heated and added citronella was carried out by using ultimate size difference, microscopic analysis, and Gas Chromatography-Mass Spectrometry compared with the commercial VCO product. The results showed that there was an intensity change in the similarity of ion fragments as well as microscopically and carbon content of more than 70%, hydrogen 10%. Separation of VCO components resulted in good separation among ethyl esters such as ethyl caprylate, ethyl laurate, and ethyl myristate detected for 30 minutes by gas chromatography and fragment ion similarity in mass spectrometry. Chemical components in commercial oils were lauric acid, ethyl laurate, glycerol tricaprylate, and vinyl decanoate.

1. Introduction
Virgin coconut oil (VCO) is only one, but it comes with various trademark, allowing the content of different chemical compounds due to different manufacturing procedures and techniques. Virgin coconut oil or pure coconut oil is produced from old coconut (Cocos nucifera L) and cooked through mechanical and natural means, either by heating or without heating. Traditionally VCO is made by heating at temperatures that are generally not controlled and through a simple and easy process with the technology available in the household.

VCO analysis resulted in a content of about 40% lauric acid, 24% myristate, 11% palmitate, 7% caprylate (octanoate), 6% oleate, 3% stearic, as well as 0.8 and 0.5 respectively as linoleic and caproic [1]. The essential oil can provide a distinctive aroma obtained from secondary metabolites in plants, as a complex mixture of terpenoid hydrocarbons, oxidized terpenes, and sesquiterpenes [2]. Plant parts such as vetiver root (Vetiveria zizanioides), peterbri wood stems (Cordia trichotoma), leaves such as citronella (Cymbopogon nardus), flowers such as Lavenders (Lavandula officinalis), and fruits such as lemon, orange (Citrus spp.) and others [3].
Chemical components of essential oil are oxygen-containing monoterpenes such as citronellal, geranial, geraniol, citronellol, and mineral, which have the potential as an antifungal in the range of 250-1000 µg / mL [4]. The chemical components of *Cymbopogon nardus* analyzed by GC-MS are citronellal and geraniol with the highest percentage area of 30%, citronellol (13%), limonene and elemol respectively about 4%, and other compounds less than 2% [5]. Sesquiterpenes as markers identified in essential oils by GC-MS [6]. The citronellal was detected higher in the first fraction of *Cymbopogon nardus* about 25% by GC-MS [7].

The GC-MS of VCO components in the publication varies greatly as well as the essential oils in *Cymbopogon nardus*, so that in this study developed a method for making VCO with and without heating and addition of essential oils from various *Cymbopogon nardus* particles was carried out. VCO components with the addition of essential oils were expected to increase the content of volatile compounds so that they could maintain the characteristics of the oil. The results of the analysis were compared to commercial VCO.

**2. Materials and Methods**

2.1 Material

The leaves of *Cymbopogon nardus* and coconuts were collected and bought in the traditional market, in the morning at the Kesiman Village in Denpasar Bali. Ethanol p.a (Merck), product VCO, aquadest, Ethyl Palmitate, laureate, dan Oleate standards from Merck.

2.2 Extraction of *Cymbopogon nardus*

Fragrant *Cymbopogon nardus* L. as much as 500 g, cut into thin pieces to obtain small pieces and then aerated until small and dry *Cymbopogon nardus* L. is obtained, then boiled for 2-3 minutes and put in an oven about 8-9 hours at 55-65°C, then cooled, blended, and sifted with an 80 mesh sieve and obtained about 55 g of *Cymbopogon nardus* L. powder. *Cymbopogon nardus* L. is also made fresh only by blending and chopping so that the resulting blender and chopped fresh *Cymbopogon nardus* L. are obtained.

2.3 Preparation of Virgin Coconut Oil

A total of 14.40 kg of grated coconut added with water in a ratio of 1:2 so that the coconut milk was obtained and then allowed to stand for 3 hours. The cream formed in the upper layer called blondo and then filtered as Virgin Coconut Oil. Furthermore, with and without heating for about 10 minutes was applied.

2.4 Preparation of Virgin Coconut Oil and *Cymbopogon nardus* the mixtures

There are 3 forms of fragrant *Cymbopogon nardus* L., each powder weighing 1 g added to each 100 g of VCO with and without heating which was is placed in a glass bottle and stored for 24 hours. The VCO soaked fragrant *Cymbopogon nardus* L. was filtered and stored again in another bottle. Furthermore, the heating VCO and addition of *Cymbopogon nardus* and without both treatments were analyzed microscopically, using ultimate sample analysis, and Gas Chromatography-Mass Spectrometry.

2.5 Analysis of Essential Oil from the leaves of *Cymbopogon nardus* and Virgin Coconut Oil

2.5.1 Ultimate analysis

An analysis to determine the content of elements of carbon, hydrogen, oxygen, and nitrogen (CHON).

2.5.2 Microscopic analysis

Microscopic analysis of all samples using Nikon Eclipse Ni

2.5.3 Gas Chromatography-Mass Spectrometry

Gas Chromatography-Mass Spectrometry analysis was performed using a Shimadzu GC-QP 2010 Ultra, fitted with a DB-5MS capillary column 5% diphenyl-polydimethylsiloxy (30m x 0,25 mm,
film thickness 0.25µm-catalog 122-5532 with serial number USC638033H. Helium was used as the carrier gas at a column flow of 1.19 mL/min and linear velocity 40 cm/sec, purge flow 3mL/min and split ratio 100.0. The oven temperature was programmed at 70-300°C (15º/min, 29 min), injection temperature 250°C, and ion source temperature 220°C, interface temperature 250°C, MS start m/z 35 and the end m/z 500. The identification of the essential oil components was based on the comparison of acquired mass spectra with reference spectra of the NIST08.LIB and Wiley7.LIB mass spectral library. Relative amounts of essential oil from *Cymbopogon nardus* in the mixture with VCO components were calculated based on the chromatogram peak area normalization method.

3. Result and Discussion

Virgin Coconut Oil, with a clear color and smell of coconut, was produced as much as 1.48 kg with a yield of 10.28%.

3.1 Ultimate analysis

Analysis of carbon, hydrogen, oxygen, and nitrogen in VCO with and without heating and the addition of the ultimate fragrant lemongrass shown in Figure 1.

![Figure 1](image1.png)

**Figure 1.** Presented of Carbon and hydrogen by ultimate analysis detected in a mixture of VCO and *Cymbopogon nardus*

3.2 Microscope analysis

Microscopic VCO results showed that the particle size of about 4 µm is the smallest size after VCO is treated by heating and the addition of fragrant lemongrass powder. This can be caused by damage to oil due to heating. Also, fragrant lemongrass needed to obtain good and homogeneous characteristics to obtain VCO with good lemongrass. Results were shown in Figure 2.

![Figure 2](image2.png)

**Figure 2.** Sighting of VCO microscopically (a) VCO (b) VCO-Powder of *C. nardus* (c) VCO-heated (d) VCO- heated and added *C. nardus*
3.3 Analysis of Essential Oil in a mixture of VCO and Cymbopogon nardus by GC-MS

The analysis of all VCO in Figure 3, the VCO control without heating using Gas Chromatography detected propane-1,1,3 triethoxy and ethyl esters such as octanoic acid, tetradecanoic acid, hexadecanoic acid, and octadecanoic acid (Figure 3a). After heating VCO detected more ethyl ester such as ethyl tridecanoate and pentadecanoic acid with small peaks (Figure 3b).

The addition of Cymbopogon nardus to the VCO which was analyzed by gas chromatography was detected in addition to the ester as well as Figure 3b and more components such as dodecanoic acid 1,2,3-propanetriol ester (CAS) but increased peak area (Figure 3c) at the end of the separation there were still many compounds analyzed as impurities with still not good separation and resolution are shown in Table 1. The commercial product of VCO was also detected lauric acid, ethyl laurate, glycerol tricaprylate, and vinyl decanoate can show in Figure 4.

Figure 3. Separation of the VCO compounds using Gas Chromatography (a) VCO control (b) VCO after heating (c) VCO after adding Cymbopogon nardus powder (CNP)

Figure 4. One of the commercial VCO is also detected lauric acid, ethyl laurate, glycerol tricaprylate, and vinyl decanoate other than above ester
Table 1. The Retention time (tR) and peak area resulted of VCO components in VCO control, VCO after heating, and VCO after adding Cymbopogon nardus powder

| tR  | Area VCO x 10^4 | Components                                      |
|-----|-----------------|-------------------------------------------------|
| 7.2 | 22 7            | Propane, 1,1,3- triethoxy                        |
| 8.8 | 192 82 64       | Octanoic acid ethyl ester                       |
| 8.9 | 64 67 50        | Benzoic acid-2 hidroxy methyl ester (CAS)       |
| 10.9| 128 100 99      | Tetradecanoic acid ethyl ester (CAS)            |
| 13.2| 81 167 130      | Tetradecanoic acid ethyl ester (CAS)            |
| 17.1| 97 76 61        | Tetradecanoic acid ethyl ester (CAS)            |
| 19.8| 133 24 19       | Hexadecanois acid ethyl ester (CAS)             |
| 21.1| 14 18 35        | Dodecanoic acid 2,3-dihydroxypropyl ester       |
| 22.1| 6  16           | 9,12-Octadecanoic acid                         |
| 22.2| 6  34 307       | 9 Octadenoic acid ethyl ester                  |
| 22.6| 49             | Octadecanoic acid ethyl ester                  |
| 29.4| 31 736          | Octanoic acid, 4 tri decyl ester               |
| 29.5| 97 170          | Octanoic acid-1,2,3 propaneryl ester (CAS)      |
| 31  | 37 88           | Decanoic acid 2 hydroxyl-1 hidroxymethyl       |
| 32.8| 34 27           | Dodecanoic acid-1- hidroxymethyl               |
| 32.7| 45             | Octadecanoic acid-2,3-bis-1 osotetradecyl      |
| 32.8| 53             | Dodecanoic acid-1,2,3-propaneryl ester         |
| 33.3| 97             | Dodecanoic acid-1,2,3-propaneryl ester         |

\( t_R \) retention time (minutes)  
C control  
H heating  
A\textsubscript{cp} adding Cymbopogon nardus powder

Fatty acid ethyl esters obtained in VCO was by the results of Asy’ari and Cahyono [1] with the highest of lauric acid content followed by other esters [8]. The results of the esters obtained in VCO vary widely. The percentage of lauric acid is greater by centrifugation in making VCO [9]. In addition to the method of fermentation can also be done, which by hot extraction processes better yield than cold methods [10].

About 22 chemical compounds were detected in C. nardus essential oil, one of the citronellal was the highest percentage, and the least was citronella [11-13]. The age of Cymbopogon nardus leaves affects the physical and chemical quality of the essential oil extracted [14, 15]. The Extraction process and quality of coconut can result in good an antioxidant and high oil yield contents [16]. The fermentation processes were also found the highest phenolic compounds and α tocopherol contents [17]. More than sixty compounds were detected in oil, sesquiterpenes, and monoterpenes as the chemical profiles by GC-MS, compounds containing nitrogen and sulfur were found in the determined oils [18].

Ion fragmentation (m / z) on mass spectrometry at each peak in each treatment above that VCO was spread between m / z 35 to 250, but VCO with the addition of Cymbopogon nardus and then heated resulted in m / z spread from 35 to 500, the result as shown in Figure 5. VCO Commercial as a positive control could be detected five peaks using GC-MS with the same system and condition above. The maximal result of ethyl esters such as ethyl caprylate, ethyl decanoate, ethyl laurate, ethyl myristate, and ethyl hexadecanoate, respectively, shown in Figure 6.
Figure 5. Relative intensity (%) for fragment ion (m/z) (a) virgin coconut oil (VCO) control, (b) VCO after heating, (c) VCO after adding Cymbopogon nardus, and (d) Combination of (a), (b), and (c).

Figure 6. Peaks of ethyl ester in VCO Commercial by Gas Chromatography

4. Conclusion
Separation of VCO components resulted in good separation among ethyl esters such as ethyl caprylate, ethyl laurate, and ethyl myristate detected for 30 min by gas chromatography as well as fragment ion similarity in mass spectrometry. Chemical components in commercial oils lauric acid, ethyl laurate, glycerol tricaprylate, and vinyl decanoate.

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