Essential Oil Extraction From Citronella Stems (Cymbopogon winterianus) By Vacuum Distillation

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Abstract. This research aims to determine the most influential variables among the three variables in extracting essential oils by vacuum distillation, to determine the optimum conditions of the most influential variables, and to determine the characteristics of citronella essential oils according to Indonesian National Standard for citronella oil.

The method used is experimental design by factorial design with two levels with the aim of making a new design to determine the most influential variables. The results of the research that had been carried out using experimental design methods obtained that the most influential variable in distillation of citronella oil through the water vapor distillation process with the assistance of vacuum were the variables of the duration of the oven, and the optimum conditions for the distillation of citronella oil through the steam distillation process with the assistance of vacuum were obtained, namely: 3 hours oven time, with 1 cm of citronella stem size and distillation operating temperature of 150o C. The Geraniol test results through GC-MS showed that geraniol has not met the requirements of Indonesian National Standard 06-3953-1995.

1. Introduction
The essential oil is oil from plants which components are generally volatile, so that most people call it flying oil. Essential oil is also called etherial oil or keteris oil because it has ether-like properties, in international language it is usually called essential oil because it has a distinctive characteristic as an aroma/odor agent.

The ingredients commonly used in essential oils are vetiver (Vetiverazizoniodes), cloves (Eugenia Aromatica), nutmeg (MyristicaFragans), cananga (Cananaga), citronella (Cymbopogon nardus. L) and sandalwood (sandalwood). In this research, the researchers chose to use the alternative of citronella stem as raw material because the yield obtained from the essential oil of citronella stem was 0.4% and the citronellal content was 66-85%, this citronellal component is the most important component in citronella essential oil because it affects the intensity of the odor released by the oil. (erepo.unud.ac.id, 2017)

The used method of essential oil creation was by Water Distillation, Water and Steam Distillation, Direct Steam Distillation. In this research the researcher used the Direct Steam Distillation because it required less water so that could shorten production process time. This steam method can be completed by cohabation system such as condensate water from the separator reentered automatically into the kettle to minimize the water loss. (erepo.unud.ac.id, 2017).

In the research of Sari I.D. et all., 2015 it was only concluded the optimum distillation time of citronella essential oil creation, and the result of the research has not been compared to the Indonesian National Standard of citronella essential oil, as well as has not obtained the optimum...
distillation temperature. Therefore in this research, the researcher would conduct the test as well as compare the obtained result to the Indonesian National Standard of citronella essential oil (SNI 06-3953-1995).

2. Methodology

In this research, the experimental design method was used where a set of data is designed to obtain concrete data to prove a hypothesis. There were several ways of experimental design, one of them was the factorial design method with two levels, namely low level and high level. This method was used in this research. At the low level, the variable duration was 0 hour (without oven), the size of the material was 1 cm, and the distillation temperature was 120°C. While at the high level, the variable duration of the oven was 5 hours, the size of the material was 5 cm, and the distillation temperature was 150°C.

This research used the citronella stems ingredient variable as much as 1,000 grams and 2 litter of waters for 1 hour of distillation and with the assistance of a vacuum compressor which can reduce the pressure in the system by 40 mmHg, so that the pressure in the system will drop to 720 mmHg. After that to the produced oil was conducted the % analysis test for the yield, density, refractive index, total geraniol-citronellal, solubility in ethanol, fat content in accordance with the Indonesian National Standard standard for citronella essential oil (SNI 06-3953-1995).

3. Result and Discussion

| Experiment | Oven Duration P (Hour) | Material Size S (cm) | Distillation Temperature t (°C) | Yield (%) |
|------------|------------------------|----------------------|---------------------------------|-----------|
| 1          | 0 Hour                 | 1 cm                 | 120°C                           | 0.353     |
| 2          | 5 Hours                | 1 cm                 | 120°C                           | 0.386     |
| 3          | 0 Hour                 | 5 cm                 | 120°C                           | 0.202     |
| 4          | 5 Hours                | 5 cm                 | 120°C                           | 0.322     |
| 5          | 0 Hour                 | 1 cm                 | 150°C                           | 0.342     |
| 6          | 5 Hours                | 1 cm                 | 150°C                           | 0.40      |
| 7          | 0 Hour                 | 5 cm                 | 150°C                           | 0.237     |
| 8          | 5 Hours                | 5 cm                 | 150°C                           | 0.315     |
Table 1 showed the largest yield shown in the 6th distillation experiment with a combination of variables that produced the highest yield, namely 5 hours of oven time, 1 cm material size and 150°C distillation temperature.

From the normal probability curve of %P vs I, it was known that the variable farthest from the line was the variable duration of oven (Ip). So, it can be concluded that the variable that has the most influence was the variable duration of the oven (Ip). Furthermore, look for the optimum price of the duration of the oven with the 6th run variable.

In the table and graphic above, from 0 to 3 hours the yield of citronella oil was increasing due to the smaller water content and impurity of the ingredients. However, at 3 to 5 hours the yield value tended to be constant, this was possible because there was no more evaporated water and impurities. The oven on this material was intended for the drying process of the ingredients (citronella) and reducing the moisture content and impurity levels in citronella so that it can increase the % yield obtained.

The Analysis Result and Comparison with Indonesian National Standard

| No. | Test Type               | Requirement                                      | Research Result | Result Suitability |
|-----|------------------------|--------------------------------------------------|-----------------|--------------------|
| 1   | Color                  | Pale yellow until brownish yellow                | Pale yellow     | Suitable           |
| 2   | Specific Gravity       | 0.880 – 0.922                                    | 0.8474          | Unsuitable         |
| 3   | Refractive index       | 1.466 – 1.475                                    | 1.4469          | Unsuitable         |
| 4   | Total Geraniol, weight/weight | Min 85                                           | 13.358          | Unsuitable         |
| 5   | Citronella, weight/weight | Min 35                                           | 44.002          | Suitable           |
| 6   | Solubility in Methanol 80% | 1:2 clean So on clear                         | Clear           | Suitable           |
| 7   | Foreign substance: Fat | Negative                                         | Negative        | Suitable           |

The colors obtained in the citronella essential oil were pale yellow, the produced color has been in accordance with the required standard. The yellow color in the essential oil was caused by the yellow substance contained in the oil or called as carotene.

The specific gravity of the tested oil was still not in accordance with the National Standard for Citronella Oil (SNI 06-3953-1995). According to Kawiji (2010), high or low specific gravity of essential oils can be influenced by components that have high boiling points. These components required a sufficient temperature to distill in comparison with low boiling components. This indicated that the operating conditions with a temperature of 150°C have not been able to distill components that have a high boiling point, this can be seen through the results of GC-MS analysis of the tested oil.
which showed that the geraniol component which has a high boiling point has not been distilled optimally.

The refractive index of the obtained essential oil was not in accordance with the predetermined standards. According to Feryanto (2006), heating with a suitable temperature will cause a number of long-chain compound components that have a high boiling point to be distilled with oil. This component can affect the density level of the oil medium so that it will affect the level of the oil refractive index value. Through the GC-MS analysis results of the tested oil, it showed that the geraniol components which have a high boiling point have not been distilled optimally. In the analysis, there was no conversion of oil to fat because the heating was carried out at no more than 160°C. At heating temperatures above 160°C, the oil can hydrolyze into unsaturated fats which are difficult to dissolve in water.

The solubility in ethanol obtained a clear mixture, it meant that when mixing with ethanol with a ratio of 1: 2 the resulting oil was completely soluble in ethanol. This was in accordance with the theory because essential oils should dissolve 100% in ethanol.

Picture 3. The Chromatogram Curve of GC-MS analysis
### Picture 4. Table of GC-MS analysis result

| No | RT    | Height   | Area       | IT  | FT  | % Area | Component                                              |
|----|-------|----------|------------|-----|-----|--------|--------------------------------------------------------|
| 1  | 3.644 | 4.45E+08 | 14374496   | 3.609| 3.689| 2.662% | Bicyclo (3.1.1) hept-2-ene, 3,6,6-trimethyl-        |
| 2  | 3.724 | 2.88E+09 | 76007952   | 3.689| 3.774| 14.074%| 1R-α-Pinene                                           |
| 3  | 3.934 | 8.34E+08 | 46423548   | 3.844| 4.034| 8.596% | Hexylene Glycol                                       |
| 4  | 4.925 | 3.59E+08 | 14474118   | 4.89 | 5.135| 2.680% | 3-Carene                                               |
| 5  | 5.305 | 4.86E+08 | 16144491   | 5.27 | 5.375| 2.989% | Limonene                                               |
| 6  | 7.981 | 3.59E+09 | 23768544   | 7.856| 8.171| 44.002%| (R)-(+)/=Citronellal                                   |
| 7  | 9.937 | 8.72E+08 | 62852060   | 9.857| 10.117| 11.638%|(R)-(+-)-β-Citronellal                                 |
| 8  | 10.547| 8.91E+08 | 72139672   | 10.467| 10.712| 13.357%| Guaniol                                                |

Based on the results of the analysis using the Gas Chromatography-Mass Spectro method, it can be seen that the citronellal content was seen at an height of 7.981 points, while for geraniol point it was seen at an height of 10.547. Based on the analysis, the resulting citronellal content was 44.002% while geraniol content was 13.358%. Based on the Indonesian National Standard which has determined the minimum content of citronellal is 35%, this result is appropriate for the analysis because the temperature used is already above the boiling point temperature of citronellal. Whereas for geraniol, only 13.358% levels were obtained, this is not in accordance with the Indonesian National Standard which requires a minimum level of 85%.

### 4. Conclusion

The most influential variable to the citronella oil distillation through the water steam distillation process with the assistance of vacuum was the oven duration variable. The optimum condition of citronella oil distillation through the water steam distillation process with the assistance of vacuum was in the oven duration for 3 hours, with the 1 cm citronella stem size and 150°C distillation operation temperature. It showed that in the operation condition with temperature of 150°C has not been able to distillate the component with high boiling point.
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