Alternative to conventional extraction of vetiver oil:
Microwave hydrodistillation of essential oil from vetiver roots
(Vetiveria zizanioides)

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Abstract. In this study the extraction of essential oil from vetiver roots (Vetiveria zizanioides) has been carried out by using microwave hydrodistillation. In the extraction of vetiver oil using microwave hydrodistillation method is studied the effect of microwave power, feed to solvent (F/S) ratio and extraction time on the yield of vetiver oil. Besides, in this study can be seen that microwave hydrodistillation method offers important advantages over hydrodistillation, such as shorter extraction time (3 h vs. 24 h for hydrodistillation); better yields (0.49% vs. 0.46% for hydrodistillation); and environmental impact (energy cost is appreciably higher for performing hydrodistillation than that required for extraction using microwave hydrodistillation). Based on the analysis using GC-MS can be seen 19 components on vetiver oil that has been extracted using microwave hydrodistillation. In addition, GC-MS analysis showed that the main components of vetiver oil that has been extracted using microwave hydrodistillation method were β-Gurjunene (30.12%), α-Vetivone (20.12%), 4-(1-cyclohexenyl)-2-trimethylsilylmethyl-1-buten-3-yne (13.52%) and δ-Selinene (7.27%).

1. Introduction
Recently there has been an increase in demand for essential oils extracted from plant. This can be attributed to the fact that essential oils have multifunctional properties and hence are playing an increasing role in the food, fragrance, agricultural and pharmaceutical industries [1].

Vetiver grass also known by the Latin name Vetiveria zizanioides or its traditional name Khus, is a perennial grass which is part of the Poaceae family [2]. The root is the most valuable part of the grass as it forms an intertwined network that stops erosion and it contains the majority of the essential oils which has valuable aromatic and biological properties [3]. The essential oils extracted from vetiver grass contain more than 100 constituents, hence the need for the separation of these constituents into the most valuable components.

In developing countries, agricultural grasses are very popular types of crops for rural outreach programmes due to the high value of the essential oils extracted from the grass. Vetiver grass has many uses; it is very easy to grow as it can withstand harsh environmental conditions and does not require large amounts of fertiliser or pruning and therefore it is easily maintained by the user at low cost. It is capable of growing in extreme soil types; this includes sands, shale, gravels, mine tailings and even more toxic soils. It is also very easy to control the spread of vetiver grass as it propagates by root division or slips and is easily removed when no longer required [4].
Currently the extraction of vetiver oil is mostly done using conventional methods such as hydrodistillation, steam distillation and solvent extraction [5]. However, this conventional method requires a long time of extraction, a lot of solvents and requires a lot of energy [6]. However, this method has been controversial for subsequent determination of the oil chemical composition because of the possible transformation of aroma-active compounds by heat, steam, and pH [7]. In addition losses and degradation of some volatile compounds due to long extraction times, degradation of unsaturated or ester compounds through thermal or hydrolytic effects are the principal disadvantages of this extraction method [8]. Thus, developing an alternative rapid, sensitive, safe, and energy conserving extraction method is highly desirable. There is an increased demand for new extraction methods with shortened extraction times and reduced organic solvent consumption, preventing pollution and reducing sample preparation costs. Recently, much attention has been given to the application of microwave dielectric heating in many various processes in the chemical and food industry in order to replace the conventional extraction methods. Extraction processes performed under the action of microwave selective heating [9]. Driven by reducing energy and waste water or solvent, advances in microwave extraction have resulted in some methods such as microwave hydrodistillation and microwave steam distillation [6,9].

Therefore, in this study the extraction of vetiver oil is done using microwave hydrodistillation method. There is no information about studied parameters that affect the extraction of vetiver oil from vetiver roots using microwave hydrodistillation method has been reported in the previous study. In addition, this study will also compare chemical composition, energy consumption and environmental considerations using hydrodistillation method.

### 2. Materials and methods

#### 2.1. Materials and chemicals

The dried vetiver roots used in this study were obtained from Garut, West Java, Indonesia and has size of about 35-60 mesh. The distilled water and anhydrous sodium sulphate used in the study were of analytical grade.

#### 2.2. Microwave hydrodistillation method

In employing microwave hydrodistillation, we used a domestic microwave oven (EMM2308X, Electrolux, maximum delivered power of 800 W) with wave frequency of 2450 MHz. The dimensions of the PTFE-coated cavity of the microwave oven were 48.5 cm x 37.0 cm x 29.25 cm. The microwave oven was modified by drilling a hole at the top. A round bottom flask with a capacity of 1000 mL was placed inside the oven and was connected to the Clevenger apparatus through the hole. Then, the hole was closed with PTFE to prevent any loss of the heat inside.

Some feed to solvent ratio (0.30; 0.40; 0.50 g/mL) were placed in the reaction flask and heated by microwave irradiation with various power (300; 450; 600 W) and various extraction time (60; 120; 180 min). The different densities and their immiscibility required that the water and vetiver oil be separated from each other by separating funnel and the excess water be refluxed to the extraction vessel in order to provide uniform conditions of feed to solvent ratios for extraction. The vetiver oil was collected in amber vials, dried under anhydrous sodium sulphate and stored at 4°C. The extraction yield of vetiver oil was calculated according to the equation given:

$$\text{Extraction yield (\%, w/w)} = \frac{\text{Mass of extracted vetiver oil}}{\text{Mass of dried vetiver roots} \times (1 - \text{water content})} \times 100 \quad (1)$$
2.3. Chemical analysis of vetiver oil components
The components of the vetiver oil were identified by Gas Chromatography-Mass Spectrometry (GC–MS) analysis [6,10,11,12]. An Agilent 6980N gas chromatograph with an Agilent 5973 mass spectrometric detector was used for GC-MS. Gas chromatography-mass spectrometry was incorporated with a chromatography column HP-5.5% phenylmethylsiloxane, 30 m length, 0.32 mm film thickness, 0.25 μm internal diameter). The injection port and the interface were held at 230 and 280°C, respectively. The temperature was programmed from 50°C to 280°C at 10°C per minute with helium as the carrier gas. Most of the compounds were identified according to mass spectra (National Institute of Standards and Technology (NIST) and Wiley spectral library collection).

2.4. Electric consumption
The electric consumption of different extraction methods was calculated based on the influence of power consumption and extraction time. The general equation for the electric consumption described by (Eq. (2))

$$E_C = \frac{P \cdot t}{3600000}$$

(2)

where $E_C$ is electric consumption (kWh), $P$ is power consumption (W) and $t$ is time (s).

Additionally, the relative electric consumption of different extraction methods can be expressed by (Eq. (3))

$$E_{C}^E = \frac{E_C}{m}$$

(3)

where $E_{C}^E$ is relative electric consumption (kWh/g) and $m$ is mass of obtained essential oil (g).

2.5. CO2 emission
The measurements of CO2 emitted were carried out based on the procedures mentioned in the previous studies: to obtain 1 kWh of energy from coal or fossil fuels, 800 g of CO2 will be released into the atmosphere during their combustion [6]. Thus CO2 emission is described by (Eq. (4))
where $E_{CO_2}$ is CO$_2$ emission (kg) and $E_C$ is electric consumption (kWh).

For the relative CO$_2$ emission of different extraction methods was calculated according to (Eq. (5))

$$E^{\prime}_{CO_2} = \frac{E_{CO_2}}{m}$$

where $E^{\prime}_{CO_2}$ is relative CO$_2$ emission (kg/g) and $m$ is mass of obtained essential oil (g).

3. Results and discussion

3.1. Effect of microwave power on the yield of vetiver oil

The microwave power exhibited significant effects on the extraction of vetiver oil (Fig. 2). By microwave hydrodistillation, when the microwave power increased from 300 W to 450 W, the yield of vetiver oil increased very fast. For example, when the microwave power increased from 300 W to 800 W for 180 min, the yield increased from 0.36% to 0.53%. Beyond 450 W, the yields of the vetiver oil did not increase.

![Figure 2](image-url) The effect of microwave power on the yield of vetiver oil (feed to solvent ratio of 0.50 g/mL).

The increase of vetiver oil yield in microwave hydrodistillation is probably due to the high pressure gradient formed inside the plant material (vetiver roots) [13]. Microwave absorption causes fast internal heating thus creating significantly high internal pressures which enhance the extraction of vetiver oil. The cell walls can swell and burst because of internal heating and thus further promotes the release of vetiver oil. Therefore an appropriate increase in microwave power led to rise in yield. Thus, the microwave power of 450 W was chosen as the output microwave power in microwave hydrodistillation.

3.2. Effect of feed to solvent (F/S) ratio on the yield of vetiver oil

As shown in Fig. 3, the yield of vetiver oil was found to increase with the increase of feed to solvent (F/S) ratio and then fall down at the optimum ratios. The smaller feed to solvent ratio means a larger concentration difference which favors mass transfer. But in microwave-assisted extraction, a higher solvent volume may give lower yield [14]. As found in Fig. 3, the yield of vetiver oil increased with the increase of feed before the feed to solvent (F/S) ratio reached 0.40 g/mL, and then they fell down. The phenomena were probably due to the inadequate stiring of the solvent when the microwave was applied at larger feeds. Hence, the feed to solvent (F/S) ratio of 0.40 g/mL seems to be appropriate for microwave hydrodistillation.
3.3. Effect of extraction time on the yield of vetiver oil

Fig. 4 shows the effect of extraction time on the yield of vetiver oil over the range 60-180 min under a fix microwave power of 450W. From the Fig. 4, the amount of yield does not change significantly after 120 minutes where the yield obtained up to this period was 0.50%, 0.64% and 0.45% for feed to solvent ratio (F/S) of 0.30, 0.40 and 0.50 g/mL, respectively. The rate of extraction was high at the beginning of the extraction but get slow gradually by time. The effect of this parameter in microwave hydrodistillation has been studied by number researches on various plant materials [15,16,17] and almost all of them obtained the same configuration as illustrated in Fig. 4. These results confirmed the Fick’s second law of diffusion which stated about the final equilibrium achieved by the solute concentrations in plant matrix and in the solvent after a certain time. This cause into no significant improvement in vetiver oil yield when prolonging the extraction time.

Figure 3. The effect of feed to solvent (F/S) ratio on the yield of vetiver oil.

Figure 4. The effect of extraction time on the yield of vetiver oil (microwave power of 450 W).
3.4. **Electric consumption and environmental considerations**

The reduction of the extraction time is clearly advantageous for the proposed microwave hydrodistillation method in terms of cost and energy. In this study, a comparison between extraction of vetiver oil using hydrodistillation and microwave hydrodistillation methods has been performed. The extraction of vetiver oil using hydrodistillation method is based on previous research by Leite (2012) [5]. Leite (2012) [5] has done the extraction of vetiver oil with feed to solvent (F/S) ratio of 0.0167 g/mL for 24 hours using heating mantle (Glas Col STM1001 230 V/600 W) and obtained yield of 0.46%. While as a comparison then in this study selected the operating conditions from the extraction of vetiver oil using microwave hydrodistillation method that can be obtained yield almost equal to the extraction of vetiver oil using hydrodistillation method. So in this study for extraction of vetiver oil using microwave hydrodistillation method selected operating conditions as follows: microwave power of 300 W, feed to solvent (F/S) ratio of 0.40 g/mL and extraction time of 3 h. This is because in these operating conditions obtained yield almost equal to the extraction of vetiver oil using hydrodistillation method that is 0.49%.

The energy requirement needed to perform the extraction methods, based on the power consumptions of the hot plate (in hydrodistillation) and the microwave oven (in microwave hydrodistillation), considering the total periods of full extractions, was 14.40 kWh for hydrodistillation and 0.90 kWh for microwave hydrodistillation (Table 1). Kusuma and Mahfud (2017a) [6] showed that for the energy requirements, microwave hydrodistillation needs less than 1.30 kWh for normal performance but conventional methods expend more than 6.50 kWh. Relative electric consumption for the production of 1 g vetiver oil in hydrodistillation and microwave hydrodistillation was 294.63 and 2.72 kWh/g vetiver oil, respectively (Table 1). This indicates a substantial saving in the extraction cost when using microwave hydrodistillation instead of hydrodistillation. In addition Kusuma and Mahfud (2017a) [6] has also reported that the energy required for the extraction of essential oil from patchouli leaves (*Pogostemon cablin* Benth) was 6.55 kWh per gram of patchouli oil in hydrodistillation, but 1.62 kWh per gram of patchouli oil in microwave hydrodistillation.

**Table 1.** The electric consumption and environmental impact (CO₂ emissions) for the extraction of vetiver oil using hydrodistillation and microwave hydrodistillation methods

| Extraction methods     | Electric consumption (kWh) | Relative electric consumption (kWh/g vetiver oil) | CO₂ emission (kg) | Relative CO₂ emission (kg/g vetiver oil) |
|------------------------|-----------------------------|-----------------------------------------------|------------------|-----------------------------------------|
| Hydrodistillation (Leite, 2012) | 14.40                       | 294.63                                       | 11.52            | 235.70                                  |
| Microwave hydrodistillation | 0.90                        | 2.72                                         | 0.72             | 2.17                                    |

Regarding the environmental impact of pollution, the calculated quantity of CO₂ emitted in the atmosphere was higher in the case of hydrodistillation (11.52 kg CO₂) than microwave hydrodistillation (0.72 kg CO₂) (Table 1). Kusuma and Mahfud (2017a) [6] found that the amount of CO₂ which was released into the atmosphere was higher in hydrodistillation (5.36 kg CO₂) than microwave hydrodistillation (1.01 kg CO₂). Relative amounts of CO₂ emissions that result from the production of 1 g vetiver oil were higher in hydrodistillation (235.70 kg CO₂/g vetiver oil) than in microwave hydrodistillation (2.17 kg CO₂/g vetiver oil) (Table 1). This finding further indicated that there was a significant difference between hydrodistillation and microwave hydrodistillation in terms of the amount of CO₂ released into the atmosphere for the production of 1 g vetiver oil. Also, waste water was lower in microwave hydrodistillation than hydrodistillation. In addition Kusuma and Mahfud (2017a) [6] has also showed that the amount of CO₂ released into the atmosphere was dramatically higher in
hydrodistillation (5.24 kg CO₂/g patchouli oil) than that in microwave hydrodistillation (1.30 g CO₂/g patchouli oil).

3.5. Gas Chromatography-Mass Spectrometry (GC-MS) analysis
To find out the components contained in vetiver oil analysis is done using Gas Chromatography-Mass Spectrometry (GC-MS). The results of the GC-MS analysis can be seen from the obtained chromatogram (Fig. 5). From the chromatogram can be seen that on the vetiver oil that has been extracted using microwave hydrodistillation method there are 19 components. Based on the results of GC-MS analysis also showed that the main components of vetiver oil that has been extracted using microwave hydrodistillation method were β-Gurjunene (30.12%), α-Vetivone (20.12%), 4-(1-cyclohexenyl)-2-trimethylsilylethyl-1-buten-3-yne (13.52%) and δ-Selinene (7.27%). The results of the GC-MS analysis for vetiver oil that has been extracted using other methods showed different results. It can be seen from the results of the GC-MS analysis for vetiver oil that has been extracted using hydrodistillation method that has been done by Leite (2012) [5]. The main components of vetiver oil produced by hydrodistillation method were nootkatone (22.64%), khusimol (9.33%), pogostol (5.70%) and vetiselinolen (4.66%).

Figure 5. The chromatogram of vetiver oil that has been extracted using microwave hydrodistillation method.

4. Conclusion
The extraction of essential oil from vetiver roots (*Vetiveria zizanioides*) using microwave hydrodistillation offered important advantages over hydrodistillation: shorter extraction times (3 h vs. 24 h for hydrodistillation); better yields (with microwave hydrodistillation the obtainable vetiver oil quantity is higher than in case of hydrodistillation); it is environmentally friendly (the amount of CO₂ emission – a result of the extraction process of vetiver oil – was dramatically higher in hydrodistillation than that of microwave hydrodistillation); has reduced cost; and has less energy consuming compared to hydrodistillation. Therefore it can be used to confirm the efficiency of microwave hydrodistillation method and to explain how microwave hydrodistillation speed up the extraction process of vetiver oil.
In addition, based on the results of composition analysis for vetiver oil components using GC-MS, it can be seen that 19 components can be detected in the vetiver oil that has been extracted using microwave hydrodistillation method. The main components of vetiver oil that has been extracted using microwave hydrodistillation method were β-Gurjunene (30.12%), α-Vetivone (20.12%), 4-(1-cyclohexenyl)-2-trimethylsilylmethyl-1-buten-3-yne (13.52%) and δ-Selinene (7.27%).

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