Washing and Chopping Pre-treatment Effect of Vetiver Roots on Vetiver Oil Yield and Distillation Time

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Abstract. Vetiver is a type of grass mainly used for its roots to be extracted into vetiver oil. Despite the increasing market demand, the productivity of vetiver oil in Indonesia still consider low. One of the determinant factors of the extraction yield is the pre-treatment before distillation. This study aimed to determine the best pre-treatment method to improve the vetiver oil extraction using water and steam distillation by looking at the yield, distillation rate, and forecasted distillation duration. The distillation process was using water and steam distillation method for 9 hours. The data analysis method used Durbin-Watson autocorrelation analysis. The feasibility test of polynomial regression was modelled with the F test and ANOVA variance test. The result showed that the combination of washing 2-3 times and chopping pre-treatments of vetiver roots with a size of ± 5 cm could significantly increase the extracted vetiver oil by producing the highest yield (0.36% (wet-based) and 0.47% (dry based)), the highest extraction rate (0.057%/h) and the fastest forecasted duration of the distillation (10.5 hours). The combination of washing and chopping pre-treatments of vetiver roots was the best method and could be an economical solution for low productivity problems of vetiver oil in Indonesia.

1. Introduction
Vetiver (Vetiveria zizainoides (L) Nash) is a type of grass that is easily found in Indonesia, especially Garut, West Java. The area of vetiver plantations in West Java reached 2,360 Ha with 71 tons of crude vetiver oil production in 2016 and decreased to 1,620 Ha with 52 tons of crude vetiver oil production in the following year [1]. The derivation in the productivity of vetiver affects the volume of vetiver oil production to be down. Meanwhile, the world demand for vetiver oil annually can reach 300 tons [2]. Vetiver oil is obtained from root extraction by several distillation methods, one of which is water and steam distillation [3]. The vetiver oil industry in Indonesia is dominated by small-medium industries that perform oil extraction with simple methods, so the yield produced is low and oil and does not comply with the required standards. It causes the volume of vetiver oil exports from Indonesia to tend to be low. The characteristics of a good vetiver oil have a brownish yellow colour, slightly thick, and has a sweet, earthy, and woody aroma [4].

Vetiver oil is one of the main ingredients used in the cosmetic, perfumery, pharmaceutical, flavour, and fragrance industries which causes the demand in the global market to be high. One way to increase the volume of vetiver oil is by postharvest treatment. One of the most economical ways to overcome these problems and can be done by small-medium industries. Postharvest treatment of vetiver that can be done is washing. Washing has aimed to remove impurities attached to the roots, in this case is soil [5]. A study stated that washing raw materials could increase the yield and higher oil quality than
unwashed [5]. Another treatment is chopping. There are two types of chopping on the vetiver root, the first is chopping to separate the roots from the bulb, and the second is chopping to reduce the size of the roots. The purpose of chopping is to release the essential oil components from the roots and expand the surface of the vetiver. So, the bulky size can be minimized [7]. Chopping must be done quickly to avoid excessive evaporation of components [5]. This study aimed to determine the best pre-treatment method to improve the vetiver oil extraction using water and steam distillation by looking at the yield, distillation rate, forecasted distillation duration. This was an effort to overcome the low production problem of vetiver oil.

2. Methods
2.1. Tools and Material
Research tools used were aluminum foil, 60 mL reagent bottle, evaporation dish, 10 mL measuring glass, bucket, machete, cardboard, C clamp, baking sheet, oven, distillation set (kettle, stove, dean stark apparatus, and condenser), thermohydrometer, analog scales, and zip locks plastic. The material used were vetiver roots (*Vetiveria zizanoides* (L.) Nash) from Garut, West Java, with a harvest age of ± 12 months, water, and 3 kg LPG gas.

2.2. Preparation and Pre-treatment of Vetiver Roots
Vetiver roots aged ± 12 months in fresh condition (freshly harvested) were obtained from CV. Sinar Wangi, Jalan Raya Kamojang, Sukakarya Village, Samarang District, Garut Regency. Storage of fresh roots spread out on cardboard. The storage of vetiver is carried out in the SITH ITB wood lab. Room temperature and humidity were measured three times a day using a thermohygrometer.

Washing the vetiver was done by inserting the roots into a bucket then soaking it while repeatedly rinsing so that the soil is released from the roots [5]. The water has been changed 2-3 times in each bucket. The clean vetiver is drained by aerating it on a three stacked rack with the base of each stack spaced apart so that water did not stagnate. The drying process was carried out for approximately seven days after washing. The vetiver chopping was carried out on the same day as the distillation day [5]. Chopping was done with a machete to make the root and bulbs size ± 5 cm.

2.3. Vetiver Root Distillation Process
The process of distillation of the vetiver was done by water and steam distillation. The kettle was cleaned by rinsing with water, then filled with the input water at approximately 12 L for the distillation process. Another distillation set, the dean-stark apparatus, was attached to the boiler pipe connection with the addition of seal tape and rubber at the joint to avoid leakage. The vetiver was weighed 2 kg using a bucket and an analog scale. Then, the vetiver was put into a kettle in which there is already water and a porous aluminum barrier. The kettle was closed, and 12 pieces of C clamps were added to hold the boiler cover. After all the parts were installed, the LPG was attached to the regulator, and the stove was turned on. The distillation process lasts for 9 hours. The obtained oil was recorded every hour to see the effectiveness of the distillation.

2.4. Measuring Moisture Content of Vetiver Roots
Measurement of moisture content was carried out by the gravimetric method. The sample was weighed ±1 gram and placed in an evaporation dish. Sample drying in the oven was carried out for 24 hours. After 24 hours, the sample was weighed again, and the moisture content was calculated [8]. The formula for calculating moisture content is as follows.

\[
\text{Moisture Content} = \frac{\text{total weight} - \text{Dry weight}}{\text{Dry weight}} \times 100\% \quad (1)
\]
2.5. Research Design
The study was conducted experimentally using a Completely Randomized Factorial Design (CRFD) with two factors and two treatment levels. It consists of 3 samples of vetiver for testing the water content. The treatment given was vetiver root with washing and chopping. Each treatment has three replications. The treatment consisted of washing treatment (washed and unwashed) and physical treatment of the roots (chopped and unchopped) by combining these treatments into four combination treatments (washed and unchopped; washed and chopped; unwashed and unchopped; unwashed and chopped). There were 12 units carried out in this experiment.

2.6. Data Processing
The determination of vetiver oil yield was calculated from the oil obtained during distillation compared to the weight of the vetiver roots [9]. The calculation of the yield of vetiver oil is carried out with the following formula.

\[ \text{Yield} = \frac{\text{Total of vetiver oil produced}}{\text{Total of vetiver root}} \times 100\% \]  

The distillation rate of oil is calculated based on the oil yield per time. A simple linear regression can be chosen as a projection model for distillation rate with the following formula.

\[ Y = a + bX \]

Where \( Y \) is the dependent variable, \( X \) is the independent variable, \( a \) is the intercept, and \( b \) is the gradient. Meanwhile, the correlation coefficient formula in simple linear regression is as follows.

\[ r = \frac{n \sum XY - (\sum X)(\sum Y)}{\sqrt{[n \sum X^2 - (\sum X)^2][n \sum Y^2 - (\sum Y)^2]}} \]

The coefficient of determination is done by squaring the value of the correlation coefficient [10]. The initial stage in determining the forecasting model is the interpolation technique. Interpolation is a technique for obtaining a function that can pass through all points of discrete data. In other words, interpolation is a technique of estimating a value between known discrete points [11]. Linear interpolation is the method used to analyze the oil accumulation data obtained for 9 hours. Linear interpolation is formulated as follows.

\[ f_1(X) = f(X_0) + \frac{f(X_1) - f(X_0)}{(X_1 - X_0)} (X - X_0) \]

Description:
\[ X_0 = \text{Initial value} \]
\[ X_1 = \text{End Value} \]
\[ X = \text{Searched value} \]
\[ f(X_0) = \text{Function value of starting point} \]
\[ f(X_1) = \text{Function value of end point} \]
\[ f_1(X) = \text{Function value of searched point} \]

The forecasted duration of the distillation model was carried out by using the data interpolation approach, followed by the model equation from polynomial regression. The polynomial regression is as follows [12].

\[ Y = b_0 + b_1X + b_2X^2 + \cdots + b_nX^n + \varepsilon \]

Where \( Y \) is the dependent variable, \( b_0 \) the intercept, \( b_1, \ldots, b_n \) is the regression coefficient, \( X \) is the independent variable and \( \varepsilon \) is the confounding factors that cannot be explained in the regression model.
2.7. Data Analysis

2.7.1. Classical assumption test. A classical assumption test is used to test data based on whether or not the data is allowed to be tested. One of the classical assumption tests used is the autocorrelation test. Autocorrelation is used to determine whether the data contains autocorrelation or not with the Durbin – Watson statistical test. The data has no autocorrelation if the d value between du and (4 - du) [13]. The time-series data type can be tested for autocorrelation.

2.7.2. Feasibility test of polynomial regression model. Testing the feasibility of the model can be done using the goodness of fit with statistical values. This test can show whether all the variables in the model influence the dependent variable with the test criteria as follows [13].
1. If F value > F table with P-value < 0.05, then this model test is feasible to use for research.
2. If F value < F table with P-value > 0.05, then test this model is not feasible for research.

2.7.3. Analysis of Variance (ANOVA). Determining the effect of chopping and washing on the yield of vetiver oil was carried out through data analysis using Two Ways Factor ANOVA with α = 0.05. Meanwhile, to determine the effect of moisture content on the yield using a simple linear regression test.

3. Result and Discussion

3.1. Moisture Content

The moisture content based on Figure 1 was in the range of 28% - 51%. Vetiver with unwashed-chopped treatment had a higher moisture content than the unwashed-unchopped treatment. However, the moisture content in the washed-unchopped treatment was higher than in the washed-chopped treatment. This is presumably because chopping can expand the surface area of the roots to be distilled.

![Figure 1](image-url)  
**Figure 1.** The moisture content of vetiver with chopped and unchopped roots treatment.

Chopped vetiver has a larger surface area than unchopped vetiver. The root surface has the potential to be attached to more soil when chopped. The type of soil attached to the vetiver is fine-textured so that this type can trap water higher than the coarse-textured soil type [14]. Thus, the more soil attached to the root surface, the more water from the environment is trapped in the soil and results in higher moisture content of the root. The moisture content of the washed-unchopped was higher than the washed-chopped treatment. This is presumably because the water content of the vetiver has not reached the equilibrium moisture content. When the chopping process is carried out at room temperature conditions, water evaporation from the material is still ongoing. Chopping was able to accelerate the evaporation of water.
from the roots because the process of water diffusion from the cell wall to the outside occurs faster than unchopped treatment water diffusion [15].

3.2. Yield of Vetiver Oil
Based on Table 1, wet basis vetiver oil yield with the unwashed-chopped treatment was 0.21%, and the un washed-unchopped treatment was 0.32%. Meanwhile, vetiver with washed-chopped treatment was 0.36%, and washed-unchopped treatment was 0.24%. Washing on vetiver increased the yield compared to without washing because it could remove soil on the root surface [5]. In addition, chopping can facilitate the release of essential oil components from the roots and expand the root surface to minimise the bulky properties [5]. However, the unwashed-chopped treatment had the lowest yield compared to other treatments. It happens because the root has the highest moisture content. The distillation process is a process to separate materials based on differences in evaporation rates. At the same temperature and pressure, the water component has a lighter fraction than the oil component, so that that water will evaporate more quickly than oil. The higher the moisture content, the longer the extraction process will be. In addition, the low oil obtained thought because the oil contains a lot of heavy fraction components that are not evaporated and left on the roots [7]. The yield of unwashed-unchopped treatment was higher than washed-unchopped treatment. It happened because there is cell damage during the process of draining the roots after washing. Cell wall damage may occur in roots being dried and result in evaporation of oil which has a low melting point before the distillation process [16]. The results of the ANOVA test (α = 0.05) showed that the washed-chopped treatment had a significant effect on the yield of vetiver oil.

Table 1. The yield of vetiver oil on a wet basis and a dry basis with washing treatment and variations of chopped and unchopped roots.

| Basis       | Unwashed | Washed |
|-------------|----------|--------|
|             | Unchopped| Chopped| Unchopped| Chopped |
| Wet basis   | 0.32%    | 0.21%  | 0.24%    | 0.36%   |
| Dry Basis   | 0.43%    | 0.32%  | 0.35%    | 0.46%   |

The yield of wet basis obtained from distillation for 9 hours is in the range of 0.21 – 0.35%. The yield of vetiver oil commonly obtained by farmers in Garut Regency is 0.27 – 0.35% for 12-14 hours of distillation [5]. It proves that the yield will increase if the root is pre-treated, even with a shorter distillation. The calculation of the dry basis yield was done to determine the yield obtained based on the conditions of the initial moisture content of different materials. The yield obtained for all treatments was higher than that of the wet base. In calculating the dry base yield, the initial dry root mass was the root mass that no longer contains water so that it can be known the actual vetiver oil content in the ingredients.

3.3. Correlation of Moisture Content to Yield of Vetiver Oil
The correlation between the moisture content with dry base and wet base to vetiver oil yield presented in Figure 2. Based on Figure 2, the relationship between vetiver oil yield and moisture content was inversely proportional.
Figure 2. The correlation between the moisture content with dry base and wet base to vetiver oil yield.

It stated that the higher the moisture content, the lower the oil yield. The moisture content in the roots would affect the amount and quality of distilled oil. If the vetiver contains high water, the part that would evaporate first is the cell’s cavity containing water. Water evaporation from vetiver with high water content will take longer than vetiver with low water content. Thus, the possibility of the oil not being extracted optimally will be high. In addition, vetiver in fresh conditions with slow-drying would cause the roots to become mouldy and rot [6]. The rotten vetiver will reduce the quality and yield of vetiver oil.

3.4. Distillation Rate

The distillation rate can be seen from the gradient on the curve. The gradient value is a measure of the change in the vertical and horizontal distances of the curve. Based on Figure 3, the gradient of the vetiver distillation with the unwashed-unchopped treatment was 0.055, and the chopped variation was 0.040. It means that every one-hour oil refining will produce an average of 0.055% oil yield for the unchopped variation and 0.04% oil yield for the chopped variation. Likewise, in Figure 4, the gradient of the washed-unchopped variation is 0.042, and the chopped variation is 0.057. The speed of steam determines the oil extraction rate from the roots into contact with the oil. Essential oils can be distilled with water vapour when the steam is in contact with the oil so that the steam penetration process into the cell membrane to push the oil out would occur through hydro diffusion [17].

Figure 3. The yield of vetiver oil per time in the unwashed treatment.
Figure 4. The yield of vetiver oil per time in the washed treatment.

The highest distillation rate was washed-chopped treatment because the chopping process can accelerate the release of oil and expand the root’s surface so that the possibility of steam contact with the material is high. Chopping can cause damage to the roots so that the oil glands will open. When the oil glands were open, steam penetration into the roots will be more accessible, and the hydro diffusion process will be faster [17]. The highest distillation speed in the treatment without washing was the variation without chopping. It was presumably because the soil that inhibits the oil diffusion process in the unchopped treatment was less than the chopped vetiver. Chopping can expand the root’s surface and positively increase the number of impurities attached to the root, in this case, soil. Thus, the contact of steam with oil would be inhibited due to the high amount of impurities.

3.5. Forecasting Model of Distillation Time

Forecasting distillation time was done to determine the time when the oil can no longer be extracted, in other words, the oil component in the material has run out. Before the determination of the forecasting model, the classical assumption was tested with an autocorrelation test on the data on the accumulation of vetiver oil recovery during distillation. Autocorrelation testing was carried out to determine the feasibility of time series data [13]. Based on the results of the Durbin-Watson statistical test, the data on the accumulation of vetiver oil acquisition during distillation for all variations of the pre-treatment did not show any autocorrelation so that the data could be used as a distillation forecasting model.

Figure 5. Forecasting model of vetiver oil distillation with unwashed treatment.
Figure 6. Forecasting model of vetiver oil distillation with washed treatment.

The selection of the regression equation model was done after the data is interpolated. The forecasting model was selected by reviewing the highest coefficient of determination and the results of the F-Test. The third-order polynomial regression equation model is the most suitable equation model for estimating the accumulation curve of vetiver oil recovery based on the distillation time because the coefficient of determination of the third-order polynomial regression model is the best. Based on the results of the F test, the third-order polynomial regression model is feasible to use for all variations of the initial treatment. Figures 5 show a graph of oil recovery during a 9-hour refining time and the resulting equation. The third-order polynomial regression equation for the treatment of vetiver unwashed with chopped or unchopped variations is as follows.

\[ Y = -3E-08x^3 + 3E-05x^2 + 0.0032x - 0.4023 \]  
\[ R^2 = 0.9973 \]  

\[ Y = -4E-08x^3 + 4E-05x^2 + 0.0064x - 0.6491 \]  
\[ R^2 = 0.998 \]

Based on the forecasting results, the estimated end time of distillation in the unwashed-chopped treatment was 780 minutes (13 hours) with the estimated recovery of vetiver oil was 4,678 mL. There was an increase in the acquisition of vetiver oil after 9 hours of distillation as much as 0.478 mL. Meanwhile, for unwashed-unchopped treatment was 630 minutes (10.5 hours) with the estimated recovery of vetiver oil was 6.493 mL and the addition of vetiver oil recovery after 9 hours distillation was 0.16 mL. The difference in the end time of refining in the two treatment variations was caused by differences in the speed of oil refining. Based on Figure 5, the distillation rate in the washed-unchopped treatment was faster than the washed-chopped variation. Similar studies have been carried out with the results obtained that when the refining rate is large, the amount of oil obtained is more. The use of a distillation rate of 0.13 mL/min produces 0.65% oil and a distillation rate of 11.7 mL/min produces 1.42% oil [18].

In addition, other factors that were thought to affect the end time of distillation on chopped roots were due to the increased amount of soil adhering to the root surface. Soil could affect the vapor diffusion process in the roots during distillation. One of the types of impurities found in vetiver roots is fine-textured soil [14]. Figures 6 show the graph of the oil recovery during the 9-hour distillation time and the model of the third-order polynomial regression equation that was formed successively for the treatment of washing with variations chopped and unchopped is as follows.

\[ Y = -3E-08x^3 + 3E-05x^2 + 0.0032x - 0.4023 \]  
\[ R^2 = 0.9973 \]

\[ Y = -4E-08x^3 + 4E-05x^2 + 0.0064x - 0.6491 \]  
\[ R^2 = 0.998 \]

Forecasting results show that the estimated time for completion of washed-chopped treatment was 630 minutes (10.5 hours) with the estimated recovery of vetiver oil is 7,467 mL. There was an increase in the acquisition of vetiver oil after distillation of 9 hours as much as 0.3 mL. Meanwhile, distillation time for the washed-unchopped treatment was 660 minutes (11 hours) with the estimated recovery of...
vetiver oil was 5.049 mL and the addition of vetiver oil recovery after 9 hours of distillation was 0.249 mL. The completion time of distillation in the chopped treatment was faster than the unchopped treatment, presumably because if the material was not chopped the distillation time only depended on the relatively slow diffusion process [7].

3.6. Vetiver oil extracted over distillation time
Figure 7 showed oil fluctuation values every 1 hour. Vetiver oil appeared the fastest after 2 hours of distillation. The water and steam distillation methods require time to boil the water located at the bottom 1/3 of the kettle so that the extraction process begins when the water boils, i.e., after the first 2 hours of distillation [19]. In addition, the oil extraction process will occur after the water in the cell cavity has evaporated. The steam formed at the beginning of the distillation, still at a low temperature to condense the roots. It takes place until the temperature of all materials was the same as the boiling point according to a certain pressure used [7].

Unwashed-chopped treatment was known to come out more in the 2nd hour of distillation compared to unwashed-unchopped treatment. It is presumably because chopping can damage the cell walls of the root so, the oil glands are open, and the surface of the root becomes wide. Essential oils can be distilled with water vapor when the steam was in contact with the oil so that the steam penetration process to the cell membrane will push the oil out through, and hydro diffusion will occur. Chopping can accelerate the hydro diffusion process so that the oil comes out faster [17].

The oil yield in the treatment of the chopped root increased from the 2nd to the 3rd hour of distillation time. Meanwhile, the increase in oil recovery occurred at the 2nd hour to the 6th hour of distillation time for unchopped root. Oil recovery increase occurs when the steam begins to contact the oil, and a diffusion path is formed so that the amount of oil extracted is high. Meanwhile, the oil gradually decreased at the 4th hour of the chopped treatment and the 7th hour of refining the unchopped treatment. The decline occurs until the end of distillation. In this phase, it was suspect that the oil content in the root is decreasing so that the diffusion between steam and oil will be slower. The extraction rate will be fast at the beginning of the distillation and slow down until the end of the distillation. The phase of decreasing oil recovery earlier in the chopped treatment was thought the low vapor diffusion process due to impurities on the surface of the vetiver. So, the steam penetration process on the root does not run perfectly [9].

Figure 7. Forecasting the extraction of vetiver oil over time of distillation in the unwashed treatment.

Figure 8 showed that vetiver oil appeared after 2 hours of distillation time. The yield in the chopped root variation increased from the 2nd hour to the 4th hour of distillation time. Meanwhile, for unchopped
treatment, the increase in oil recovery occurred at the 2nd hour to the 3rd hour of distillation time and increased again at the 6th hour of distillation time.

The occurrence of two stages of increase is thought to be due to the low fraction of oil being spread over the cortex of the vetiver root and mixed with the high fraction oil. Thus, the amount of oil obtained during the refining hours fluctuates. Oil with a low boiling point was extracted at the beginning of the distillation, and at the end of the process, the amount of oil obtained decreases because the remaining oil with a weight fraction/high boiling point remains [7]. The extraction gradually decreased at the 5th hour of distillation on the chopped variation and the 7th hour of distillation on the unchopped variation.

4. Conclusion
Washing and chopping pre-treatment of vetiver roots with water and steam distillation method affected vetiver oil recovery, distillation rate, and forecasting duration of distillation. The highest yield of vetiver oil based on wet basis and dry basis as follows 0.36% and 0.46% with washed-chopped treatment. The highest distillation rate was in the unwashed-unchopped treatment with a yield value of 0.055%/hour and the washed-unchopped treatment had a yield value of 0.057%/hour. The forecasting model showed that the fastest distillation time with the highest vetiver oil volume was washed-chopped treatment, and the estimated distillation time was 10 hours 30 minutes with a final volume was 7.46 mL from 2 kg of roots. The best treatment that can be used as a solution to increase the extraction of vetiver oil based on yield, distillation rate, and the forecasting of distillation time was washed-chopped treatment.

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