Redistillation of wood vinegar from peat swamp species

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Abstract. This study aimed to determine the characteristics of wood vinegar (WV) from 3 native peat swamp forest species: gerunggang (Cratoxylon arborescens), mertibu (Dactylocladus stenotachys), and meranti bunga (Shorea teysmanniana) after redistillation. The redistillation was carried out in a laboratory by heating 550 mL of wood vinegar in the distillation flask at a temperature of 100°C for 7 hours. The parameters observed were the yield, the density, the acetic acid content, the pH, and the percentage of hydroxyl scavenging. The results showed that WV of D. stenotachys had the highest values of yield (%), pH, and percentage of hydroxyl scavenging among other WV types. However, its acetic acid content was the lowest when compared with that of S. teysmanniana WV and C. arborescens WV. Besides, both S. teysmanniana WV and C. arborescens WV were considered to have better potential as an antioxidant, an antimicrobial, and a food preservative. The pH levels of all purified WV in this study were classified as very acid indicated by the high quality of WV. Furthermore, D. stenotachys WV and C. arborescens WV fulfilled the requirement of Japanese WV standard. The redistillation of WV from the three species of peat swamp forest successfully produced WV with a clearer, transparent, and pale-yellow color.

1. Introduction
The problems that often appear after the fire in peat swamp forest (PSF) are numerous wood piled up from dead and fallen trees on the forest floor. Accumulation of this wood waste actually can become a potential fuel and, worse, potentially trigger the occurrence of bigger fires in the future [2]. To minimize the risk of the fires, one method that can be attempted is by reducing the volume of dead wood waste to be utilized as the raw material for charcoal and wood vinegar [8].

Wood vinegar (WV), also called pyroligneous acid, is a brown flavorful liquid produced by the distillation of wood in the absence of air condition [22]. In the pyrolysis process, thermal degradation of biomass initiated by evaporation of water vapor followed by hemicellulose decomposition, subsequently cellulose decomposition (at 180°C – 350°C), and terminated by lignin decompositin (at 160°C – 625°C). The decomposition of hemicellulose and cellulose will produce carboxylic acids and carbonyl compounds, whereas the decomposition produces phenolic compounds [20]. The higher the pyrolysis temperature, the volume of WV produced becomes greater [15].

The pyrolysis process produces a liquid which is called by pyrolysis oil, bio-oil, bio-crude, bio-fuel, oil, tar oil, or wood vinegar [8]. Generally, the color of WV is brownish yellow because it still contains tar and other volatile compounds. Furthermore, Fachraniah [6] explain that the yellow and brownish colors in WV are caused by carbonyl compounds. WV has many uses, such as for the food industry and health as well as an insecticide, pesticide, and a crop fertilizer. To remove tar and reduce the strong smoke odor in WV, a purifying process is absolutely necessary. The purpose of WV
purification is to remove tar and to produce WV with prominent functional properties, such as having a clearer color, high acetic acid, lower pH, and less smoke odor [6].

Several experiments to produce WV from three native species of PSF, namely mertibu (*Dactylocladus stenotachys*), meranti bunga (*Shorea teysmanniana*) and gerunggang (*Cratoxylon arborescens*) have been successfully done. However, the WV produced from the pyrolysis process is crude WV with a red brownish color and has a strong smoke odor [2]. Redistillation is a technique for refining WV by separating the solution based on different boiling points. In a redistillation process, some components can evaporate faster than others [17]. The redistillation of WV is carried out by removing unwanted and dangerous compounds, such as poliaromatic hydrocarbon (PAH) and tar. By controlling the boiling temperature, it is expected that a clear liquid smoke which is free of tar and benzopyrene can be obtained. This WV purification process usually uses a temperature lower than the pyrolysis temperature, which is around 100°C depending on the type of wood used for WV material [4].

Because the information about WV from PSF, both crude WV and purified WV, is very limited, hence this current study attempts to find the physical quality of WV from the redistillation of three types of peat swamps forest species: mertibu (*Dactylocladus stenotachys*), meranti bunga (*Shorea teysmanniana*), and Gerunggang (*Cratoxylon arborescens*). The results of this study are expected to give beneficial information to support the development of WV from wood waste to become a more useful product.

2. Materials and methods

2.1. Production of wood vinegar

Three types of WV were produced at Tumbang Nusa research station, Central Kalimantan Province. The equipment for the WV pyrolysis consisted of a drum with a capacity of 200 L, an iron plate, and a galvanis pipe. The raw materials for WV were some dead woods from 3 species of mertibu (*Dactylocladus stenotachys* Oliv. Family Melastomataceae), gerunggang (*Cratoxylon arborescens* Bl. Family Guttiferae), and meranti bunga (*Shorea teysmanniana* Dyer Ex. Brandis Family Dipterocarpaceae).

The production of WV started by selecting some wood sticks (9 – 20 cm diameter size) and collecting them near the kiln. Then the wood sticks were cut 20-35 cm long and chopped into 4-6 parts. These wood pieces were arranged in a stove with a standing position until the drum kiln was full then closed tightly. The combustion was done with a big flame at a temperature of 400°C for approximately 8 hours. The smoke from this combustion was then captured and channeled through a galvanized pipe which was cooled from a water channel. The smoke from this pyrolysis was collected to make WV [2].

2.2. Purification of wood vinegar (WV)

The purification of WV was carried out in the main laboratory of the Faculty of Mathematics and Natural Sciences, Lambung Mangkurat University. The equipment consisted of a distillation device, a scale, a measuring cup, and a pycnometer, while the required material was WV from *C. arborescens*, *D. stenotachys* and *S. teysmanniana* resulting from the pyrolysis process.

The process of purifying WV began with putting 550 mL of WV into a distillation flask. Then it was heated with a heating mantle at a temperature of 100°C for 7 hours. The steam was formed then flowed into the cooling pipe and collected as the WV distillate.

Afterward, the resulting distillate was analyzed for measuring several parameters, such as the yield, the density, the pH, the acetic acid content, and the percentage of hydroxyl scavenging.
2.3. Analysis procedure

2.3.1. Yield. The yield was obtained by measuring the purified WV (distillate) with a measuring cup. Then the distillate volume was compared with the initial volume of WV before the purification. It was calculated by the following formula:

\[
Yield(\%) = \frac{\text{Purified wood vinegar volume}}{\text{Wood vinegar volume}} \times 100\%
\]  

(1)

2.3.2. Density. The specific gravity was measured by putting the purified WV into a pycnometer until exceeding the mark. Then it was closed to avoid the pressure from the air bubbles and weight. The density of liquid smoke was calculated by the following formula:

\[
Density = \frac{(\text{Pycno weight+sample})-(\text{Empty pycno weight})}{(\text{Pycno weight+distilled water})-(\text{Empty pycno weight})}
\]  

(2)

2.3.3. pH. To check the pH levels, a pH meter was employed to obtain duplicate results.

2.3.4. Acetic acid. The concentration of acetic acid was measured by the titration method. 5 mL of WV was put into a measuring tube, and the water was added until the volume was 100 mL. Next, 10 mL of this liquid was taken and put into an erlenmeyer tube. 2 drops of phenolphthalein solution was added, and the solution was titrated with NaOH 0.1 M. This NaOH solution was used to reddon the solution then recorded.

2.3.5. Percentage of hydroxyl scavenging. Hydroxyl scavenging is the percentage which states the ability of a compound to scavenge hydroxyl radicals. The hydroxyl radical scavenging activity test refers to the deoxyribose method [7] as follows. Determination of operating time. The solution consisted of 600 \( \mu \)L of deoxyribose solution 2.5 mM, 300 \( \mu \)L of FeCl\(_3\) 1 mM, 300 \( \mu \)L of EDTA 1mM, 300 \( \mu \)L of H\(_2\)O\(_2\) 20 mM, 300 \( \mu \)L of vitamin C 1 mM, and 4500 of buffer fosfat pH 7.4. The solution was incubated for 30 minutes at 37 °C. Next, 1 mL of TCA 5% and 1 mL of TBA 1% were added at a temperature of 80 °C for 30 minutes. After cooling, the absorption of the solution was read at a wavelength of 532 nm. Sample solution. 600 \( \mu \)L of deoxyribose solution 2.5 mM, 300 \( \mu \)L of FeCl\(_3\) 1 mM, 300 \( \mu \)L of EDTA 1mM, 300 \( \mu \)L of H\(_2\)O\(_2\) 20 mM, 300 \( \mu \)L of vitamin C 1 mM, and 4500 buffer fosfat pH 7.4 were put into a closed reaction tube. The solution was incubated for 30 minutes at 37 °C. Then 1 mL of TCA 5% and 1 mL of TBA 1% were added at a temperature of 80 °C for 30 minutes. After cooling, the absorption of the solution was read at a wavelength of 400-600 nm. Control solution. The solution consisted of 600 \( \mu \)L of deoxyribose solution 2.5 mM and the distillated WV from D. stenotachys, C. arborescens, S. teysmanniana as much as 200 \( \mu \)L, 400 \( \mu \)L, 600 \( \mu \)L, 800 \( \mu \)L, and 1000 \( \mu \)L, then 300 \( \mu \)L of FeCl\(_3\) 1 mM, 300 \( \mu \)L of EDTA 1mM, 300 \( \mu \)L of H\(_2\)O\(_2\) 20 mM, 300 \( \mu \)L of vitamin C 1 mM, and the buffer fosfat at pH 7.4 were added to each tube until the final volume reached 6 mL. The mixture was incubated for 30 minutes at 37 °C. Next, 1 mL of TBA 1% and 1 mL of TCA 5% were added at a temperature of 80 °C for 30 minutes. After cooling, the absorption of the solution was read with a maximum wavelength from the optimization results. The result analysis was calculated using following formula in equation 1.

\[
\% \text{ scavenging} = \frac{\text{Absorbance of control solution} - \text{Absorbance of sample solution}}{\text{Absorbance of control solution}} \times 100\%
\]  

(3)

The hydroxyl scavenging analysis was carried out at the Faculty of Medicine, Lambung Mangkurat University.
3. Results and discussion

3.1. Yield (%)
The highest volume of distillate was produced from *D. stenotachys* WV (395 mL) followed by *C. arborescens* (332 mL), whereas the lowest was from *S. teysmanniana* (325 mL) (table 1).

| Type of WV       | Volume (mL) | Yield of distillate (%) | Tar (mL) | Yield of WV (%) |
|------------------|-------------|-------------------------|----------|-----------------|
| *S. teysmanniana* | 550         | 325                     | 59.1     | 240             | 5.87            |
| *C. arborescens*  | 550         | 332                     | 60.4     | 80              | 4.98            |
| *D. stenotachys*  | 550         | 395                     | 71.8     | 320             | 6.12            |

When compared with the yield of WV from the pyrolysis, the distillate yields from the three native species of PSF were much higher. This happened because the purification process was carried out in a closed distillation device and using a low temperature (100 °C). Therefore, only a little gas wasted out from the distillation process. On the other hand, the temperature used was higher (400 °C) in the pyrolysis process. Besides, the resulting products were not only WV but also charcoal, ash, and uncondensed gases (CO, CO₂ and CH₄) [2,15]. Therefore, the WV yield produced from the pyrolysis is generally lower than purified WV.

The WV purification aims to separate tar from WV based on their different boiling points [17]. In a purification process of wood vinegar, the distillate will evaporate more quickly and cool down becoming clearer WV. Meanwhile, substances with a higher boiling point will settle and produce a dark brown liquid called tar. In this research, the highest tar yield was resulted in the purified WV of *D. stenotachys* (320 mL), followed by *S. teysmanniana* (240 mL), and *C. arborescens* (40 mL) (table 1).

| Species       | Content (%) | Specific gravity | Strength grade |
|---------------|-------------|------------------|----------------|
| *S. teysmanniana* | -           | 0.83 (heavy)     | III-II         |
| *C. arborescens* | 53.1 (high) | 0.47 (light)     | III-IV         |
| *D. stenotachys* | 56.2 (high) | 0.53 (light)     | III            |

Sources : [3,13,19]

The amount of tar in WV of *D. stenotachys* and *S. teysmanniana* is expected to have a relation with higher wood density of both types of WV than that of *C. arborescens*. Martawijaya [13] stated that wood density is the ratio of weight and volume of wood in dry air condition with the moisture approximately 15%. Woods which have greater specific gravity are generally also stronger and harder [3]. The thermal decomposition biomass process in hardwood will also process more slowly, thus the steam generated from the combustion will be abundant and condensed in WV. However, Komarayati [9] provide a different assumption. They argue that a higher density material will cause the rate of burning becomes longer so that the smoke produced is less. However, several studies have shown that woods with high lignin content generally produce more smoke so that more WV will be produced [5, 9,21].
3.2. Density

The density was measured to determine the mass of volume from each purified WV. The analysis showed that there was no significant decrease in the density of WV before and after purification (figure 1). Similarly, the results of [17]’s study showed that the density of galam (*Melaleuca leucadendron*) WV did not have much difference before and after the purification process.

![Figure 1. Density of wood vinegar before and after redistillation process.](image)

The average density of the WV distillate from 3 species of PSF was 1.6 g/mL, indicating that this density was the highest among the density of galam WV (1.000-1.002 g/mL) [17]; the density of sawdust WV (1.004-1.018 g/mL) [14], the density of coconut shell WV (1.026 g/mL) [6]; the density of nyamplung (*Calophyllum inophyllum*) shell WV (1.009 g/mL) [21], the density of *Shorea* spp. WV (0.9818 g/mL) [16], and the density of ironwood WV (0.967 g/mL) [12].

The density of these three PSF species has fulfilled the density requirements of the Japanese WV standard i.e > 1.001 g/mL [21].

3.3. Acetic acid content

The acetic acid contents in the purified WV from the three PSF species were different. The highest acetic acid level was found in WV of *S. teysmanniana* (1.389 mg/L) followed by the acetic acid level in *C. arborescens* WV (1.157 mg/L), while the lowest level was obtained in *D. stenotachys* WV (0.570 mg/L). The differences in acetic acid contents in the three types of WV indicated that the types of materials and the chemical characteristics of the ingredients affected the yield of acetic acid. Although *D. stenotachys* wood had a high lignin content, but the acetic acid from the resulting WV was actually the lowest among the other types (table 3).

According to Wibowo [21], acetic acid is formed due to the pyrolysis of wood components, such as cellulose, hemicellulose, and lignin. The acid compound is the most dominant compound in WV [1]. The results of Komarayati’s [9] study also revealed that there was a difference in the acetic acid contents in bamboo species. In specific, the acetic acid contents in WV of betung bamboo, black bamboo, and spotted bamboo were 31.37%, 85.39%, and 53.37%, respectively.

The acetic acid from WV has function to accelerate the plant growth and to prevent plant diseases [23], as an antibacterial, and to form the flavor of smoke product [6]. The inhibition process of acetic acid against the bacterial growth occurs by disrupting the synthesis of membrane stability enzymes through an acidification of the cytoplasm of bacterial cells [20]. The result of [10]’s research has shown that the high acetic acid and phenol contents in WV can inhibit the development of bacteria *Eschericia coli* in vitro. In other words, WV has a potential to be developed as an antioxidant and antimicrobial agent for medicine or food preservation.
In this study, the acetic acid content of S. teysmanniana WV was the highest of the other two types, indicating that S. teysmanniana WV has a better potential as an antioxidant, antimicrobial, and food preservative agent. To determine the effectiveness of the antimicrobial of S. teysmanniana WV, it certainly needs to be supported by further research.

3.4. pH
The degree of acidity (pH) is one parameter that can be used to determine the quality of WV. The purified WV in this study was classified as very acid. The pH of S. teysmanniana WV was 1, while pH in each D. stenotachys and C. arborescens WV was 2 (table 3).

| Table 3. The properties of wood vinegar distillate. |
|----------------------------------------------------|
| Parameters                                      | Wood vinegar distillate | Quality standard of WV distillate from Japan [1] |
| - Density (g/mL)                                | D. stenotachys   | S. teysmanniana | C. arborescens | > 1.001   |
| - pH                                            | 1.6163          | 1.6154          | 1.6148          | 1.3 – 3.7 |
| - Color                                         | 2 ± 0           | 1 ± 0           | 2 ± 0           | Colorless – pale yellow |
| - Transparency                                  | Colorless – pale yellow | Colorless – pale yellow | Colorless – pale yellow |
| - Floating matters                               | Transparant     | No floating     | No floating     | Transparant |
| - Acetic acid (mg/L)                            | 0.57 ± 0.043    | 1.389 ± 0.00    | 1.157 ± 0.2     | No floating |
| - hydroxyl scavenging (%)                       | 12.815 ± 0.550  | 12.663 ± 0.368  | 7.786 ± 0.178   | 1 – 18% |

According to Achmadi [1], lower pH value and higher acetic acid level indicate a better quality of WV. A low pH value indicates a high quality of wood vinegar because harmful microbes or bacteria cannot live and reproduce properly at a low pH [5].

The pH value of redistilled WV which was classified as very acid indicated that the WV from the three native peat swamp trees had very high quality. Indeed, pH shows a level of process of breaking down the chemical components of wood into organic acids [17]. Very low pH (very acidic) occurs due to the complete decomposition or the decomposition of chemical components within an airless pyrolysis tube. The pH WV from D. stenotachys and C. arborescens in this research has fullfilled the Japanese standard requirement i.e: 1.3-3.7 [23].

3.5. Hydroxyl scavenging
Hydroxyl scavenging indicates the ability of a compound to scavenge hydroxyl radicals. The greater the value of % scavenging from purified WV, the greater are the hydroxyl radical scavenging activities.

In this research (table 3), the highest hydroxyl scavenging value obtained was in D. Stenotachys WV (12.815 % ± 0.550). The second was obtained in S. teysmanniana WV (12.663 % ± 0.368). Meanwhile, the lowest hydroxyl scavenging value was shown in C. arborescens WV (7.786 % ± 0.178).

The compounds in WV which suppose to have the scavenging activity are the polyphenolic compounds. Generally, the compounds have many hydroxyl clusters that function as a free radical scavenger. According to Li [10], phenolic compounds or polyphenols are an important group of compounds in plants. Polyphenols have at least 8000 different structures including simple phenol, phenolic acids, cumarins, isocumarins, naphtoquinones, xanthones, stilbenes, phlobonoids, and lignins.
In wood vinegar, phenolic compounds are produced from the process of breaking ether bonds and lignin carbon bonds. Polyphenols have a strong ability to clean free radicals, hence it can be used as a reducing agent and an antioxidant [21].

The lowest scavenging activity was found in C. arborescens when compared with other species in this study, and it is actually difficult to explain. This might occur because the flavonoid content in C. arborescens stems is quite low only 3.8 ± 0.4 g/mL of quercetin equivalent (QE), which is much smaller than the flavonoid contents in Mangifera casturi fruits (30.0 ± 1.2 g/mL QE), Setnochlæna palustris fern leaves (14.5 ± 0.7 g/mL QE), and Eurycoma longifolia root (6.1 ± 0.8 g/mL QE) [17].

Although there has been no data regarding the flavonoid content in D. Stenotachys and S. teysmanniana, it is supposed that the flavonoid content in both species are greater. Therefore, the hydroxyl scavenging activities in D. Stenotachys and S. teysmanniana were higher.

4. Conclusion
The redistillation of the wood vinegar from three peat swamp forest species (D. stenotachys, S. teysmanniana, and C. arborescens) produced various characteristics of the wood vinegar. The results of this study indicated that WV of D. stenotachys showed the highest yield value (%), pH, and hydroxyl scavenging (an ability to ward off free radicals). D. stenotachys WV showed the highest yield (71.8%), meaning that it succeeded to remove more tar than the other two types. However, the acetic acid content of D. stenotachys WV was much lower than that of S. teysmanniana and C. arborescens. Therefore, WV from S. teysmanniana and C. arborescens tended to be more appropriate to utilize as an antioxidant, an antimicrobial, and a food preservative. Besides, the three types of purified WV in this study were classified as very acid, indicating that the quality of purified WV was very high. Of the three types of purified WV, Both purified WV of D. stenotachys and C. arborescens have fulfilled the requirement of Japanese WV standard. All in All, the purification of the WV from the three peat swamp species have successfully improved the quality of WV as their reddish brown color became clearer and transparent with pale yellow color.

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