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Rendement and Characteristics of Wood Vinegar Produced from Ironwood Delinquent Waste through Clay Kiln Charcoaling Furnace

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ABSTRACT

The study aimed to determine the rendement of charcoal and wood vinegar and to identify wood vinegar chemical compounds made from ironwood deliquent waste. The study was carried out by using charcoaling facilities at the charcoaling center in the village of Asam – Asam. The charcoal and wood vinegar rendements were determined after ironwood delinquent waste was heated for 21 days with a temperature of approximately 400 - 600 ºC. The results showed that the rendement of the charcoal and wood vinegar were 14.02% and 0.05%, respectively. The research with wood vinegar characteristic before purification was pungent, however after purification, the results did not sting; color pH before purification was blackish-brown, however after purification it was pale brown yellow; the specific gravity before purification was 1.012 and after purification, it was 0.97; the pH before purification is 3.63 and after purification 3.41; and acid content before purification was 3.73% and after purification it was 3.66%. As a whole, the physics test of wood vinegar is categorized in Japan standard, however, the color only before purification is not standard. There are 5 (five) chemical compounds identified in wood vinegar both before and after purification.

Keywords: ironwood deliquent waste, wood vinegar, rendement

INTRODUCTION

International world concerns about the phenomenon of global climate change are increasingly high due to rising global temperatures known as global warming. The main cause of warming is a variety of human activities like burning fossil fuels such as petroleum, coal, gas in the form of smoke originating from motor vehicle exhausts and exhaust gases from factory chimneys and furnaces that release carbon dioxide and other gases with greenhouse effect.

Global warming occurs due to increased emissions of greenhouse gases such as carbon dioxide (CO2), methane (CH4), Nitrous oxide (N_2O), and Chloroflourcarbon (CFC). These gases are excessive to the atmosphere so that sunlight reflected to Earth as infrared radiation experiences long waves and ultraviolet which will be transmitted to outer space. Most of sunlight is reflected back to earth by the greenhouse gases formed in the atmosphere so that this further increases the temperature of the earth (Latuconsina, 2010).

Currently the charcoal industry is growing rapidly due to the increasing market demand. However, increased production of charcoal can have negative impacts such as smoke and greenhouse gases (Hadi et al., 2014) and can endanger their survival and environment. To overcome this, it requires efforts to change the gases into liquid smoke (known as wood vinegar) through pyrolysis technology, namely thermal degradation technology for making charcoal (carbonization) without using oxygen in the manufacturing process with a temperature of 400 - 600 ºC.

Liquid smoke (wood vinegar) contains more than 400 chemical compounds in

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which its components vary in number depending on the type of wood, the age of the wood and the conditions of growth (Astuti 2000 in Erawati et al., 2015). Liquid smoke constituent compounds consist of water 11 - 92%, phenol 0.2 - 2.9, acid 2.8 - 9.5%, carbonyl 2.6 – 4% and tar 1.7% (Milly, 2003). According to Girard (1992), the content of compounds making up liquid smoke greatly determines the organoleptic properties of liquid smoke and determines the quality of fumigation. The influencing factors are wood type, pyrolysis temperature, oxygen amount, wood moisture, wood particle size and wood smoke making equipment. Wood vinegar is used as a preservative for various food products and biopesticides to increase agricultural production, deodorize latex and preserve wood against termites.

Liquid smoke which is produced from the pyrolysis process, that in this study the GC-MS method is used, purification of wood vinegar has been done in order to determine the quality grade of it. The quality grade of wood vinegar is specified in three levels by Johansyah (2011) as liquid smoke grade 1 to 3. Wood vinegar grade 3 is not perfectly done due to the tar content. The characteristic of Grade 3 is having dark brown color and pungent. It can be used as preservative for rubber materials.

Liquid Smoke Grade 2 is purer compared to Grade 3 as it is distilled and filtered by putting zeolite. The characteristic is showing yellow brownish color and can be used as food preservative in raw food such as meat, chicken and fish. Grade 1 is the most purified version compared to the other grades, due to fractionation process and filtered by using active carbon. The characteristic is having pale yellow color and can be used for ready-to-eat food preservative such as in noodles and tofu.

After wood vinegar is being purified, the characteristics of chemical compounds and obtaining results that are almost the same as grade 3, the process of distillation of wood vinegar in grade 3 to grade 2 aims to separate phenol compounds, acids and compounds that act as anti-bacteria with aromatic polycyclic hydrocarbon compounds (HPA)) called Tar. According to Fachraniah et al (2009), tar has a bad influence because it is carcinogenic so it must be eliminated.

Asam-Asam is a sub-district in Tanah Laut district and situated in Southern coastal of South Kalimantan province, Indonesia. In Asam - Asam, there is a business unit of wood charcoal production from waste of ironwood delinquent. The charcoal processing business uses a furnace made of clay (Clay Kiln) with a total number of 123 furnaces. Charcoal processing is carried out for 21 days. So far, the by-product of the process of charcoaling ironwood waste in the form of liquid smoke has not been utilized at all. If the smoke is not followed up immediately, the smoke from the furnace will pollute the environment. Based on the statement, the research on the physical and chemical properties and distillation of liquid smoke from ironwood waste as a byproduct of the charcoal process is carried out so that this will increase the added value of the charcoal process and minimize the greenhouse gas emissions as a result of making the charcoal.

MATERIALS AND METHODS

Production of Wood Vinegar

The raw material used is the waste of ironwood delinquent from the felling in Asam-Asam village. The raw material is put into a clay furnace to do the charcoal process (carbonization). One clay stove contains

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more than 49.92 tons of ironwood delinquent waste and the charcoal process takes approximately 21 days with a charcoal temperature ranging from 400 - 600ºC, as shown on the below figure.

Figure 1. Charcoaling furnace and wood vinegar catcher

On the third day of the charcoal process, wood vinegar has begun to come out but is still mixed with water. Wood vinegar came out until the twelfth day because the furnace had been closed for the charcoal process.

The wood vinegar obtained was deposited for at least 2 weeks after which the distillation process was carried out to separate the oil and the sediment. The process of purifying wood vinegar from grade 3 to grade 2 is done by adding active charcoal to wood vinegar through the active charcoal column. It is followed by stirring and filtering.

The rendement of charcoal and the rendement of wood vinegar are calculated using the formula (Pireera, 2009):

$$\text{Rendement} = \frac{output}{input} x\ 100\%$$

Where: Input = ironwood delinquent raw material; Output = the weight of charcoal produced or the weight of wood vinegar produced

Purification of Wood Vinegar

Purification of wood vinegar is done with activated charcoal that functions as an absorber so that it is expected to obtain a better quality of wood vinegar to reduce color and odor. How to filter with activated charcoal is done through the activated charcoal column. The charcoal used in this study was activated charcoal by soaking the charcoal into 9% H3PO4 solution and allowed to stand for 24 hours. The purpose of soaking is to enlarge the pores of the charcoal so that it can absorb more organic matter and metal content in wood vinegar. According to Jamilatun, S. dan Salamah (2015), the absorption of charcoal is determined by the surface area of particles and this ability can be increased by

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activating with chemicals or by heating at high temperatures.

Figure 2. Purification of Wood Vinegar

The way to purify wood vinegar is to take 200 ml of wood vinegar which is passed in the active charcoal column and with a 250 gram active charcoal weight. The purification result obtained is 50 ml because tar and some of the compounds present in liquid smoke are absorbed by active charcoal so that this can reduce the carcinogens in wood vinegar.

Quality Testing of Wood Vinegar

Wood vinegar quality testing includes physical components such as odor, color, specific density pH, acid content and chemical testing uses Gas Chromatography-Mass Spectrophotometer (GC - MS). The pH is determined by using the potentiometric test method. The analysing method of GC-MS has combined two instruments that are Gas Chromatography and Mass Spectrophotometer. Gas Chromatography functioned to analyse molecular structure of compound and to dissociate chemical fractions in the compound. Mass Spectrophotometer functioned to analyse the total compound quantitatively (Erliza Noor et al.). The GC-MS tests were completed at the Research & Industry Standardization Centre in Banjarbaru, South Kalimantan.

The working conditions for the GC-MS were: injector temperature was 250°C, detector temperature was 280°C, and separator temperatures was graduated from 60°C up to 280°C with an 5°C increase per minute. Helium was used as mediator gas with flow velocity 0.6 μl per minute. Liquid smoke was dissolved into ether, so that separation could be done between its soluble phase and polar phase. Five μl of the ether phase was then injected to GC-MS with using standard acetate and phenol. The mix of chemical compounds through the gas chromatography then separated to individual chemical components.

Japan Pyroligneous Liquor Association, an industrial body for pyroligneous liquor trader sets 7 parameters in order to regulate the quality of wood vinegar product. This is then known as Japanese standard. The parameters are included a) pH value of around 3.0, b) standard specific gravity

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around 1.010–1.050, c) color should be a pale yellow, bright brown or reddish brown, d) has a marked smoky odor, e) the dissolved tar content should not more than 3%, f) ignition residue should not more than 0.2%, and g) exhibit transparency without suspended solid matter (Wada T., 1997). The GC-MS test results were compared against the set of Japanese standards.

As for the type weight of wood vinegar was calculated by deviding mass by volume, where the mass was the weight of the wood vinegar obtained in grams by Newton's mass unit and volume was in ml units.

RESULTS AND DISCUSSION

Data on the result of research on charcoal and wood vinegar rendement can be seen in the following Table 1.

Table 1. Charcoal and wood vinegar rendement

Product	Input (ton)	Output (ton)	Rendement (%)
Charcoal	49.92	7.00	14.02
Wood vinegar	49.92	0.023	0.05

The wood charcoal rendement obtained in this study was 14.02% and wood vinegar rendement obtained was 0.05%. The low rendement of wood vinegar produced is due to the charcoal combustion process and the condensation system used. This is reinforced by Haji (2012) stating that the formation of wood vinegar requires water as a cooling medium so that the condensation process takes place perfectly and wood vinegar can be produced maximally.

According to Sulaiman (2004), the high and low rendement of wood vinegar is influenced by several factors such as climate, season, age of plants and the content of lignin, cellulose and hemocellulose. This is reinforced by Hendriyadi (2013) stating that high level of ironwood lignin and low level of lignin dissolved in acid will approach the chemical properties of lignin such as the type of needle wood.

Wood Vinegar Physical Charateristics

The quality of liquid smoke depends on the chemical components that contained in it. The quality of the physical characteristics of wood vinegar in the form of odor, color and taste is determined by the group of chemical compounds and that contained in wood vinegar. The results of the analysis of the physical characteristics of wood vinegar from the ironwood delinquent can be seen in Table 2.

The smell of wood vinegar from the ironwood delinquent waste has a distinctive smell of wood vinegar. The transparency is murky and there is sediment. According to Yatagai (2002), good quality of wood vinegar is if it has a smoke odor and does not contain suspension. Furthermore, the quality of wood vinegar varies and depends on the type of raw material and the temperature of charcoaling.

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Table 2. Smell, color, specific density, pH, and acid level of wood vinegar before and after purification

Parameter	Wood vinegar before purification	Wood vinegar after purification	The standard of testing Japanese wood vinegar
Smell	Wood vinegar characteristic	Much reduced	-
Color	Blackish Chocolate	Yellow brown	Yellow brown, reddish pale reddish brown
Weight type	1.012	0.967	>1.005
Acidity (pH)	3.63	3.41	1.5 – 3.7
Acid level (%)	3.73	3.66	1 – 18

The quality of ironwood delinquent waste wood vinegar for Japanese wood vinegar testing standards does not require odor. The odor produced is included in grade 3 which has a strong aroma and is suitable for use in preserving wood and bamboo, clumping latex and replacing Formic acid, antifungal, termite resistant and resistant to termite attack. After purification, the pungent odor has been greatly reduced so that wood vinegar can be used to smoke fish, preserve noodles, chicken, meat and so on.

From the color parameter, ironwood delinquent waste wood vinegar is blackish brown because ironwood contains several chemical components such as cellulose 58.1%, lignin 28.9%, pentosan 20.17%, ash 1% and silica 0.5% (Mariam, 2017). After being distilled, wood vinegar changed color to clearer because the tar content has separated from other compounds that have a low boiling point. Based on the result of the study, the color of ironwood delinquent waste wood vinegar did not fulfill Japanese standard but after purification the color of wood vinegar changed to brownish yellow and fulfilled Japanese testing standard. It is illustrated as seen on the following Figure 3.

Figure 3. Wood vinegar color before and after distillation

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The type weight obtained in this study amounted to 1,012 gr / cm3 measured by a hydrometry device where the result was greater than the Japanese standard> 1,005 gr / cm3 (fulfilling the standard). After purification, the type weight does not fulfill the standard, namely the type weight value of 0.97 gr / cm3. It is suspected that the ironwood tar content is still high. The tar content which is still high can damage its use as plant fertilizer.

Wood Vinegar Chemical Characteristics

The pH (acidity) measurement aims to determine the level of decomposition of raw materials in producing organic acids in wood vinegar. The results of the study of pH obtained were 3.63 using the potentiometric test method (Table 3). The result of this test fulfilled Japanese standard with a pH between 1.5 and 3.7. The pH value affects the durability and shelf life of the product or its organoleptic properties (Wijaya, 2008). According to Swastawati, et al. (2007), wood vinegar can inhibit bacterial growth to pH 4. According to Fengel (1995), the acidity value (pH) is high because it has a high extractive substance content. Ironwood delinquent had a high extractive content because the extractive substances produced compounds such as resin, fat, wax, fatty acids and alcohol. The pH test result obtained after purification (grade 2) is equal to 3.41.

Acid content is one of the important parameters that determine the quality of liquid smoke produced. Organic acids commonly found in wood vinegar are acetic acid obtained from the decomposition of cellulose, hemicellulose and starch. These acids can affect taste, pH, and shelf life (Wijaya, 2008) and greatly affect the total phenol produced. The result of research on acid content in wood vinegar wood waste vinegar showed a value of 3.7%.

Determination of acid content by GC - MS test fulfills Japanese standard. According to Yatagai (2002), high acid levels produce a more pungent odor. Acid content obtained after purification is 3.66%. The result of the GC - MS analysis can be seen in Table 3.

Table 3. Result of GC - MS analysis of wood vinegar

No	Compound Name	%
1	2 –propynoid acid	0
2	Acetic acid	82.87
3	Propanoic acid	2.87
4	2(3H) – Furanone	2.79
5	2 Furanmethanol	1.66
6	2 methoxy – 4 methylphenol	1.32

The most common compound found was acetic acid (82.87%). The chemical component of wood vinegar such as acetic acid is useful for accelerating plant growth and preventing plant diseases. Propanoid acid can inhibit the growth of mold and some bacteria. Methanol accelerates plant growth, while phenol and its derivatives function to prevent pests and diseases or provide antibacterial and antimicrobial effects. In addition, compound such as phenol functions as an antioxidant and extends the shelf life of

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smoked products. According to Komarayati and Gusmailina (2011), wood vinegar can be used as an alternative pesticide to eradicate pests and diseases that so far can only be eradicated using bactericides and fungicides.

CONCLUSIONS

1. The rendement of charcoal from ironwood delinquent waste was 14.02% and wood vinegar rendement was 0.05%;
2. The quality of wood vinegar is included in grade 3 and grade 2 if based on the parameters tested such as odor, specific gravity, pH and acidity so that it fulfills the quality standard of Japanese wood vinegar testing;
3. The GC - MS analysis shows that there are 5 (five) chemical compounds identified; namely acetic acid, propanoic acid, 2(3H) – Furanone, 2 Furanmethanol and 2 methoxy – 4 methylphenol.

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