CONVERSION OF OIL PALM EMPTY FRUIT BUNCHES USING THE PYROLYSIS INTO BIO-OIL AND ITS CHARACTERIZATION FOR BIOPESTICIDE

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Abstract: Oil palm empty fruit bunches (EFB) is a by-product of processing CPO (Crude Palm Oil) at a palm oil processing factory. The accumulation of Oil Palm EFB waste could cause environmental problems in the future. One of the optimal ways to reuse oil palm EFB waste is to convert it into bio-oil using pyrolysis. Bio-oil contains phenol, acid compounds, and their derivatives with antioxidant and antimicrobial, which makes them suitable for biopesticides. This study aims to produce Bio-Oil by pyrolysis at different temperatures and analyze its characterization for use as a biopesticide. Oil Palm EFB characterization was carried out by measuring the yield and pH and analyzing the Phenol and Acetic Acid compounds in the pyrolysis products at 100oC, 150oC, and 245oC. Yield analysis was carried out by comparing the mass of the Bio-Oil produced with the mass of the Oil Palm EFB raw material used. Phenol was determined using the Folin-Ciocalteu reagent, and absorbance was measured using UV-Vis spectrophotometry, acetic acid was determined using the titrated acid-base method, and pH was measured using a pH meter. The results showed that the parameter of temperature in pyrolysis affected the concentration of the chemical compounds produced. The results showed that the pyrolysis temperature parameter affected the concentration of the chemical compounds produced. Treatment at 150oC- 245oC is the best result due to the parameters that have been analyzed. The characteristics of the Bio-oil produced are a yield of 29.63%-100%, total phenol of 1.04%-3.87%, total acid of 1.12%-2.14%, and a pH value of 4.09 - 4.16. The characteristics of this Bio Oil can be used as a biopesticide that complies with the Indonesian National Standard (SNI).

Keywords: Bio-Oil; EFB, Biopesticide; pyrolysis

Abstrak: Tandan Kosong Kelapa Sawit (TKKS) merupakan produk samping dari pengolahan CPO (*Crude Palm Oil*) pada pabrik pengolahan minyak kelapa sawit. Penumpukan limbah TKKS ini dapat menimbulkan permasalahan lingkungan dimasa depan. Salah satu cara optimal untuk memanfaatkan kembali limbah TKKS adalah mengkonversi TKKS menjadi Bio Oil dengan metode pirolisis. Bio Oil hasil pirolisis TKKS mengandung senyawa kimia fenol dan senyawa asam beserta turunannya dengan sifat antioksidan dan antimikroba sehingga cocok dimanfaatkan sebagai biopestisida. Penelitian ini bertujuan untuk menghasilkan Bio Oil dengan metode pirolisis serta mengetahui karakteristik Bio Oil yang dihasilkan dengan perlakuan suhu yang berbeda untuk dijadikan Biopestisida. Karakteristik TKKS yang diukur adalah rendemen, senyawa Fenol, Asam Asetat dan nilai pH pada perlakuan suhu pirolisis 100 ^{0}C, 150 ^{0}C dan 245 ^{0}C. Analisis randemen dilakukan dengan membandingkan massa Bio Oil yang dihasilkan dengan massa bahan baku TKKS yang digunakan. Kadar fenol ditentukan dengan reagen Folin-Ciocalteu dan diukur absorbansinya menggunakan spektrofotometri UV-Vis, kadar asam asetat ditentukan dengan metode asam tertitrasi dan pengukuran pH menggunakan pH meter. Hasil penelitian menunjukkan bahwa Parameter suhu pirolisis berpengaruh terhadap konsentrasi senyawa kimia yang dihasilkan. Hasil perlakuan terbaik dari parameter yang telah diuji, yaitu pada perlakuan suhu 150 ^{0}C – 245 ^{0}C, menghasilkan rendemen 29,63% - 100%, kadar fenol total 1,04% - 3,87%, kadar asam total 1,12% - 2,14% dan nilai pH sebesar 4,09 - 4,16. Karakteristik Bio Oil ini dapat digunakan sebagai biopestisida yang telah sesuai Standar Nasional Indonesia (SNI).

Kata kunci: Bio Oil; Biopestisida; Pirolisis; TKKS.

INTRODUCTION

Based on data released by the Directorate General of Plantations, Ministry of Agriculture of the Republic of Indonesia, Indonesia's palm oil production in 2022 will reach more than 48 million tons. Every oil palm agribusiness production activity produces biomass waste as a by-product. One of them is the waste of Oil Palm Empty Fruit Bunches (EFB) amounting to 20-23% of the total Fresh Fruit Bunches (FFB) produced.

In previous years, Oil Palm EFB waste at factories was used as steam fuel in incinerator machines and the ashes left over from combustion were used as fertilizer or soil fertilizer (Yahaya et al. 2017). However, this process is not environmentally friendly because it produces smoke and dust emissions due to incomplete combustion (Promraksa & Rakmak 2020). Oil Palm EFB is also widely used as organic mulch in plantations, as a substrate for mushroom cultivation (Yahaya et al. 2017) or left to decompose and returned to oil palm plantations as compost to improve soil nutrients and organic matter (Babinszki et al. 2021). However, it takes about one month of fermentation for this Oil Palm EFB to be used as fertilizer. In addition, the traditional composting method of Oil Palm EFB waste also releases nitrogen, large amounts of solid particles, and smoke into the environment which causes environmental problems (Yahaya et al. 2017).

Oil Palm EFB contains lignocellulosic (37.3%-46.5% cellulose, hemicellulose (25.33%-33.8% and 27.6%-32.5% lignin) (Pratama & Sa'diyah, 2022). One the lignocellulosic conversion method can be carried out thermochemically through the pyrolysis (Machado et al. 2022). Pyrolysis is an anaerobic process of the thermal decomposition of the chemical structure of biomass at high temperatures without involving oxygen (Promraksa & Rakmak 2020). One of the products of pyrolysis is Bio-Oil, which is a liquid substance with a color dark and smells like smoke, produced from the decomposition process of the lignocellulose component (Rahman et al. 2015; Yanti et al. 2019). According to Chang (2018), the conversion of Oil Palm EFB waste into Bio-Oil is effective and environmentally friendly in managing Oil Palm EFB waste and overcoming environmental problems caused by Oil Palm EFB waste.

Although relatively rare and still under development, the conversion of Bio-Oil into biochemicals such as olefins and aromatics (Shao et al. 2014), fatty acids (Zhou et al. 2016), as well as into biofuels for transportation such as biodiesel (Sales et al. 2022) been done. Besides, Chang (2018) has conducted research on the potential of Bio-Oil from Oil Palm EFB as an alternative fuel to petroleum. It was reported that Bio-oil from Oil Palm EFB has a lower heat capacity and pH, as well as a higher solids and ash content, but has a lower density, total acid amount and sulfur content compared to petroleum fuel. The resulting Bio-Oil is more susceptible to the formation of carbon deposits, is more acidic and corrosive than petroleum fuels, but emits less sulfur oxide during combustion. The potential of Bio-Oil as an alternative to petroleum fuel or used as a transportation fuel is still limited to research and has not yet been used commercially.

Based on previous research, Bio-Oil can be used as an anti-bacterial (J.Yang et al. 2016), insecticide, and insect repellent activity (Rahmat et al. 2014). Research conducted by Sharip et al (2016) analyze the antifungal activity of Bio-Oil obtained from palm oil mesocarp fiber and found that the resulting Bio-Oil was toxic to Ganoderma boninense, Aspergillus fumigatus, and Trichoderma asperellum. Furthermore Araujo et al (2016) Bio-Oil produced from Eucalyptus urograndis and Mimosa tenuiflora shows high antibacterial and antifungal activity due to other phenolic and furfural compounds. Acids that can affect the taste, pH, and shelf life of smoked products.

Bio-Oil which is formed from the pyrolysis process of hemicellulose produces furfural, which is a derivative of furan, as well as a long series of

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carboxylic acids and acetic acids. Pyrolysis of cellulose will produce acetic acid and small amounts of furans and phenols and pyrolysis of lignin produces phenols and phenol ethers (Kan et al. 2016). The acid compounds produced can affect the taste, pH, and shelf life of Bio-Oil products. Phenol compounds cause Bio-Oil to have a distinctive smoke aroma. Phenolic compounds and acetic acid contained in liquid smoke function as antimicrobials and bacteriostatic. Phenol compounds and their derivatives function to denature proteins in bacterial cells and destroy cell membranes, in order suitable for use as biopesticides.

Several commercial biopesticides, such as pyrethrum products from Tanacetum cinerariifolium, neem products from Azadirachtaindica, and rotenone from Derris and Lonchocarpus spp (Sola et al. 2014), have been traded globally. Biopesticides are a potential alternative to reduce the use of chemical/synthetic pesticides which can damage the environment and are dangerous for human consumption and cause damage to soil microbiology (Faisal et al. 2018).

Based on this description, it is necessary to conduct research on the potential of Bio-Oil from Oil Palm EFB waste as a Biopesticide. The purpose of this study was to convert Oil Palm EFB into Bio-Oil using pyrolysis and determine the characterization of the Bio-Oil due to Biopesticide with different temperature treatments.

METHOD

Oil Palm EFB Pyrolysis

Tools and Materials

The tool used in the pyrolysis is a set of pyrolysis tools. While the material used is Oil Palm EFB waste. The series of pyrolysis tools used is shown in the Figure 1.

Figure 1. Series of Pyrolysis Equipment
Caption: 1. stove, 2. thermometer, 3. reactor, 4. chimney, 5. condenser pipe, 6. container filled with ice water

Procedures

Before the pyrolysis start, Oil Palm EFB is chopped into small pieces to increase the surface area, then soaked for 24 hours to reduce the ash content. After that, the Oil Palm EFB is dried in the sun with the aim of removing the water in the sample.

Pyrolysis was carried out 3 times with different temperature-100°C, 150°C and 245°C. Pyrolysis is carried out by burning Oil Palm EFB in the reactor, then the resulting smoke is flowed to the condenser so that the smoke condenses to produce Bio-Oil (Pamori et al. 2015). The pyrolysis process was stopped until the Bio-Oil no longer came out of each condensate.

The next process is sedimentation to separate the tar contained in the Bio-Oil by leaving the Bio-Oil in the bottle for 7 days, then filtration using Whatman filter paper to produce Bio-Oil which does not contain tar compounds.

Characterization of Bio-Oil

Yield Analysis

Weigh the empty bottle, then fill it with Bio-Oil. Then the bottle containing Bio-Oil was weighed carefully. The yield is calculated with the formula.

yield (%b/b)= x/y ×100%

Information :

X = Weight of Bio-Oil-Mass of the filled bottle–mass of the empty bottle (grams)

Y = dry weight of Oil Palm EFB raw material (grams)

Qualitative Analysis of Phenol Compounds

Dropped 0.5 mL of Bio-Oil, dissolved in 5 mL of distilled water, and heated to boiling. 1% $FeCl_3$ was added to determine the presence of phenol and its derivatives in Bio-Oil. A positive test for phenol indicated by the formation of dark green, red, purple, blue, or black colors

Quantitative Analysis of Phenol

Determination of the phenol quantitatively in Bio-Oil was carried out using the Folin-Ciocalteu (FC) method with pure phenol as a standard. This was done because of the ability of the FC reagent in alkaline conditions to oxidize the hydroxyl groups (OH) of phenol group compounds.

50 µL Bio-Oil is diluted using distilled water up to 20 mL. From 20 mL of diluted sample, 0.5 mL was pipetted and placed in a test tube. 0.5 mL of Folin Ciocalteu reagent was added and allowed to stand for 3 minutes, then shaken on a vortex shaker. 1 mL of 5% Na_2CO_3 was added, and shaken. The absorbance was measured using UV-Vis

spectrophotometry at the maximum wavelength.

The phenol standard curve was prepared by weighing 0.01 gram of phenol and dissolving it using distilled water to a volume of 100 mL. Phenol was obtained with a concentration of 100 ppm. From a solution of 100 ppm then pipette 0.1 mL; 0.2mL; 0.3mL; 0.4 mL and 0.5 mL and placed in a test tube. For a solution that has not reached 0.5 mL, then add distilled water 0.4 mL, 0.3 mL, 0.2 mL, 0.1 mL and 0 mL. So that the phenol concentrations obtained were 20 ppm, 40 ppm, 60 ppm, 80 ppm and 100 ppm. Then 0.5 mL of Folin Ciocalteu reagent was added and allowed to stand for 3 minutes, then shaken on a vortex shaker. 1 mL of 5% Na_2CO_3 was added, shaken and the absorbance was measured using UV-Vis spectrophotometry at the maximum wavelength.

The total phenolic compounds in Bio-Oil are determined based on the regression equation that has been obtained on the phenol standard curve. Based on the equation that has been obtained, it can be known the value of x or total phenolic concentration by entering the absorbance value of the average Bio Oil sample to -y.

The total phenol content is determined by the following equation.

$$\text{Phenol Content} = \text{Total Phenol Concentration} \times \frac{\text{Initial Bio Oil Volume}}{\text{Final Bio Oil Volume}}$$

Quantitative Analysis of Acetic Acid

Determination of total acetic acid was carried out by acid-base titration. 0.1 N NaOH which was calibrated primarily first using oxalic acid. The titration begins by taking 0.2 mL of Bio-Oil and placing it in a volumetric flask and adding distilled water to a volume of 100 mL. Then 3 drops of phenolphthalein indicator were added and titrated with 0.1 N NaOH solution until the end-point of the titration, which is when the sample color changes to purplish-red and is stable (does not change when homogenized) (Luditama, 2014). Calculation of total acetic acid content is carried out using the following equation.

$$\text{Acetic Acid Content} = \frac{V.NaOH \times N.NaOH \times \text{Acetic acid molecular weight}}{\text{Sample weight (gram)} \times 1000} \times 100\%$$

pH test

Measurement of the pH of Bio-Oil using a pH meter is carried out by dipping the pH meter electrode into 10 mL of Bio-Oil sample. Then read the scale on the pH meter after the constant pointer needle (Anisah, 2014).

RESULTS AND DISCUSSION

Pyrolysis Process

The raw material used in the manufacture of Bio-Oil using this pyrolysis is Oil Palm EFB. Raw materials were chosen because apart from their abundant availability Oil Palm EFB contains lignocellulosic (37.3% - 46.5% cellulose, hemicellulose 25.33% - 33.8% and 27.6% -32.5% lignin) (Pratama & Sa 'diyah, 2022). In thermochemical terms, one of the ways to convert lignocellulose through pyrolysis. According to Machado et al (2022), in the pyrolysis process lignin will decompose to form Bio-Oil while hemicellulose and cellulose will decompose to form pyrogenic acid (liquid smoke).

Pyrolysis was carried out by heating 300 grams of Oil Palm EFB which had previously been chopped into small pieces and soaked in water for 24 hours to remove the ash content. Dried EFB is pyrolyzed at 100ºC, 150ºC and 245ºC. When the material is burned in the reactor, heat transfer occurs by conduction from the heat source to the reactor. In the process of heat transfer by conduction, the metal molecules of the reactor will hit the molecules that are nearby and provide heat to the environment, so that the EFB in the reactor will receive heat through a metal intermediary (reactor).

Meanwhile, the convection process occurs due to thermal expansion and conduction. So as a result, the specific gravity of the Oil Palm EFB particles decreases, then the lighter particles will be pushed up (hot air) while the cold air above them will fall to replace them. The heat contained in the material due to the convection process causes the substances in the material to evaporate (evaporate) in the form of smoke, then flows into the pipe that connects the reactor and the condenser due to pressure and temperature expansion in the convection process so that the smoke enters the condenser. Inside the condenser, the smoke will be cooling until it reaches the dew point and then there will be a change from the gas phase into liquid. The liquid phase of the pyrolysis product is Bio-Oil. Pyrolysis was stopped until Bio-Oil no longer came out of the condenser. In this study, the pyrolysis process took 2 hours.

The next process is sedimentation which is carried out for 7 days. This process is carried out to precipitate Tar compounds. The precipitated tar then filtered using whatman filter paper, so that this process is expected to reduce the tar content in Bio-Oil.

Potential of Bio-Oil as Biopesticide

In the pyrolysis, various reactions occur- the decomposition of hemicellulose and cellulose to produce acetic acid and its derivatives, and the decomposition of lignin to produce phenolic compounds-. The results of lignocellulosic degradation (acetic acid and phenolic compound groups) have antimicrobial to control microbial infections in the environment (Sharip et al. 2016). In this study, characterization were conducted due to potential of Bio-Oil as a Biopesticide - yield, phenol, acetic acid and pH-. The results were compared with the Indonesian National Standard (SNI).

Yield Analysis

Yield is the percentage of the amount of Bio-Oil produced in the pyrolysis which is obtained by comparing the mass of the Bio-Oil for each mass of raw material (EFB).

Table 1. The Effect of Temperature on the Yield of Bio-Oil

Temperature (^{0}C)	Yield (%)		Total (%)
	Condensate 1	Condensate 2	
100	4	-	4
150	3,70	25,93	29,63
245	47,83	52,17	100

According to Budaraga et al., (2016) the yield percentage produced depends on the raw material and pyrolysis temperature. Based on the research, the yield data shows that the higher temperature produced more bio-oil.

Pyrolysis at a temperature 100°C produces the lowest yield. This can be seen from the charcoal that has not burned completely in the reactor. It indicates pyrolysis at 100°C has not been able to burn the material optimally. Yield more is produced at a temperature of 245°C due to the complete decomposition of lignin compounds and increases the yield (Mustafiah, 2016). Budaraga et al (2016) stated that the material contains more lignin fraction, which will increase the yield.

Qualitative Analysis of Phenol Compounds

A qualitative Analysis of phenolic compounds was carried out to ensure the presence of phenolic compounds in Bio-Oil. A positive test for phenolic compounds is indicated by the formation of dark green, red, purple, blue, or black colors (Harborne, 1987). The result of the qualitative analysis is described in Table 2.

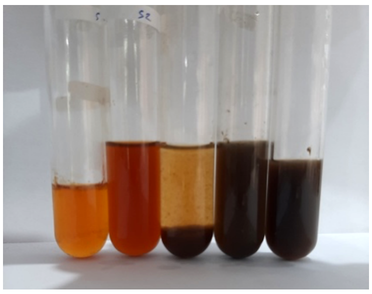

Figure1. Discoloring of Bio-Oil

A positive analysis for phenolic compounds is indicated by the formation of dark green, red, purple, blue, or black colors (Harborne, 1987).

Table 2. Qualitative Analysis of Phenol Compounds

Temperature (^{0}C)	Discoloring		Results
100		Orange	-
150	Condensate 1	Reddish brown	+
	Condensate 2	Brown and has sediment	-
245	Condensate 1	Black	+
	Condensate 2	Black	+

Note: (+) indicates a positive presence of phenol content

Table 2 shows the positive results of phenol compounds at 150^{o}C condensate 1 and at 245^{o}C condensate 1 and 2 which produced a dark black color after adding 1% $FeCl_3$.

Quantitative Analysis of Phenol Compounds

Phenol is an important parameter to know in determining the quality of Bio-Oil. Identification of phenol in Bio-Oil is expected to meet the quality criteria requirements for biopesticides. The results of the total phenol of Bio-Oil EFB in different temperatures are presented in Table 3.

Table 3. Total Phenol in Different Temperature

Temperature (^{0}C)	Phenol (%)	
	Condensate 1	Condensate 2
100	0,67	-
150	1,04	1,37
245	1,57	3,87

Table 3 shows that the total phenol in Bio-Oil ranges from 0.67% to 3.87%. More Phenol was produced at a temperature of 245^{o}C and it is almost the same as that of another study conducted by Rasi & Yulius (2016), which obtained a phenol of 3.88% at a temperature of 300–500^{o}C in Bio-Oil from coconut shell pyrolysis. In addition, Sitanggang & Sigalingging (2018) using Bio-Oil as raw material from rice husks produced an average phenol content 0.0051-0.0074%.

The presence of phenol in the Bio-Oil makes Bio-Oil as a biopesticide. Based on the Indonesian National Standard (SNI), the recommended phenol content is only a maximum of 2% in a pesticide. This finding shows that the Bio-Oil has met the SNI quality standards for Biopesticides.

Analysis of Acetic Acid

The total acetic acid in the bio-oil determines the quality of the bio-oil produced. The presence of acetic acid with phenol indicates good bio-oil quality, because phenolic compounds are also antimicrobial.

Table 4. Total Acetic Acid of Bio-Oil

Temperature	Total Acetic Acid **(%)**	
(^{0}C)	**Condensate 1**	**Condensate 2**
100	0,3	-
150	1,12	2,9
245	1,83	2,14

Table 4 shows that the highest total acid is found at the pyrolysis temperature of 150^{o}C-245^{o}C, because at a temperature of 200^{o}C-260^{o}C is the phase where the hemicellulose component decomposes become acidic compounds such as acetic acid compounds.

Based on the Indonesian National Standard (SNI), the recommended acetic acid content in Bio-Oil is 1.10% - 7.99%. While the results for the acetic acid content of Bio-Oil EFB were 1.12% - 1.83%. Its shows that the Bio-Oil has met the SNI quality standards for Biopesticides.

pH value

The pH value is an important parameter in determining the quality of the Bio-Oil. The pH measurement on Bio-Oil to determine the level of acidity in Bio Oil. The lower the pH value show good quality of the Bio-Oil and vice versa. In addition, the measurement of the pH of Bio-Oil to determine the level of decomposition of the raw materials in the pyrolysis (Diatmika et al. 2019).

Table 5. pH Value of Bio-Oil

Temperature	**pH**	
(^{0}C)	**Condensate 1**	**Condensate 2**
100	4,00	-
150	4,09	4,34
245	4,21	4,16

Table 5 shows at all the temperatures, all condensate has low pH and indicates good quality of Bio-Oil. The Bio-Oil is ready to inhibit the rate of activity of harmful microorganisms. The pH has a relationship with the total phenol in Bio-Oil. Phenol compounds can release H^+ ions and make low pH or make more acid (Diatmika et al. 2017).

Based on the Indonesian National Standard (SNI), the recommended pH in Bio-Oil ranges from 2.76 - 4.50. and it means that Bio-Oil of EFB has met the SNI quality standards for Biopesticides.

CONCLUSION

The results showed that Oil-Palm EFB has potential as a raw material for making Bio-Oil. The characterization of the Bio-Oil produced contain organic compounds such as acetic acid and phenolic compounds which are anti-bacterial due to use as alternative biopesticides.

The temperature during pyrolysis affects the concentration of chemical compounds produced. The higher temperature produces more yield, phenolic compounds, and acetic acid, although does not imply a pH. The best results at 150°C – 245°C, produce yield 29.63% - 100%, total phenol 1.04% - 3.87%, total acid 1.12% - 2.14%, and a pH value of 4.09 - 4.16. The characteristics of this Bio-Oil can be used as a biopesticide that complies with the Indonesian National Standard (SNI).

The utilization of pyrolysis technology can convert biomass, such as Oil Palm EFB which was previously not utilized optimally can be converted into value-added products which in this case produce Bio-Oil for Biopesticide which is more environmentally friendly than synthetic pesticides.

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