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Characterization Biofuel from Empty Fruit Bunch through Thermal Cracking

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Abstract: Empty Fruit Bunches (EFB) are oil palm waste that has the potential as a source of bioenergy because it contains lignocellulose (cellulose, hemicellulose, and lignin) so that it can be converted into biofuel through thermal cracking, adsorption, and distillation processes. Thermal cracking is the decomposition of the chemical content of biomass by utilizing heat without a mixture of oxygen at a temperature of 200°C–600°C. This study aims to obtain the characteristics of the raw material of EFB in the form of proximate, ultimate, lignin, and biofuel produced. The research was conducted using a thermal cracking reactor designed to control the temperature at 300°C, 350°C, 400°C, and 450°C. The results showed that the raw material characteristics of EFB from proximate were 13.66% water content, 8.74% ash content, 58.66% volatile matter and 18.90% fixed carbon. This water content is relatively high. This is because the drying process on the material has not run perfectly. The ultimate result showed that the EFB had a C content of 54.45%, H content of 5.00%, and O content of 16.27%. The atomic ratio obtained from the ultimate analysis can indicate the amount of calorific value that can be used for certain fuels. The smaller the atomic ratio value contained, the more significant the calorific value contained in a particular fuel. Klason method was carried out to decrease the level of lignin through 4 stages; the lignin content without delignification was resulting into 24.87%, the addition of aquadest was resulting into18.71%, the addition of 5% HCl resulting into 15.34%, and 10% HCl resulting into 14.49%. Delignification of 10% HCl is the pretreatment process before the thermal cracking. The thermal cracking process forms steam; the steam is then condensed to obtain bio-oil. The formed bio-oil was kept to separate the oil from tar. In order to obtain good biofuel quality, adsorption is carried out with zeolite adsorbent, which has been activated with HCL. A comparison of the physical properties of bio-oil before and after adsorption shows a color difference from brownish black to the adsorbed bio-oil, which is distilled to separate the heavy and light fractions. The temperature of 450°C at thermal cracking is close to optimum; when the temperature is increased, the cracking process will be more straightforward and occur optimally. The biofuel produced in this study was tested for its characteristics such as, density (927-1086.68 Kg/m^3), kinematic viscosity (1.17-1.43 mm^2/s), and flash point (66.00-70, 23°C). The biofuel product produced is dominated by C_5-C_{15} compounds (45.07%) according to the results of GC-MS analysis.

Keywords: Bio-oil, Biofuel, Empty Fruit Bunch, Thermal Cracking

1. Introduction

The area of oil palm plantations in 2019 increased by 1.88 % to 14.60 million hectares, with an increase in Crude Palm Oil (CPO) production of 12.92 % to 48.42 million tons [1]. South Sumatra Province is included in the top 6 largest oil palm plantations in Indonesia with an area of 1.22 million hectares, with the plantation area being able to produce around 4.3 million tons of CPO [2]. The increase in the production of oil palm plantations will also increase the amount of waste produced, either in the form of solid or liquid waste. On the other hand, every 1 hectare of oil palm plantation will produce about 1.5 tons of EFB [3]. EFB are one of the most common types of solid waste produced by palm oil mills, reaching 30-

35% of the weight of fresh fruit bunches. However, so far, it has only been used as animal feed, fertilizer, and fed as direct boiler fuel to produce energy [4]. Utilization of this type of palm oil waste is constrained by processing technology that is relatively inexpensive in the preparation of materials, and a process is needed to reduce the water content, which is still quite high, even though the lignocellulose content of EFB is quite high, namely cellulose (41-46.5%), hemicellulose (25.3 -33.8%), and lignin (27.6-32.5%) [5]. Based on these chemical components, EFB has the potential to be used as a biofuel with negligible amounts of nitrogen, sulfur, and ash, which is environmentally friendly [6-7]. The technology of conversion into energy is through a thermochemical process, namely thermal cracking [8-9].

Thermal cracking is a material decomposition process starting at high temperatures above 300ºC-600ºC and in the absence of O_2 [10]. The feed in this process can be in the form of empty palm oil bunches experiencing bond breaking to form molecules with shorter sizes and structures. The gas obtained is condensed as a hydrocarbon compound known as bio-oil [11]. Bio-oil from thermal cracking is a thick, high-density, brown liquid containing carboxylic acid compounds, alcohols, esters, aldehydes, ketones, benzenoids, and levoglucosan [12-13].

In order to get the functional properties of bio-oil, the bio-oil produced from the thermal cracking process can be fractionated by several methods. One of the bio-oil fractionation methods is bio-oil distillation. Bio-oil distillation is one of the purification methods for bio-oil, which is the process of re-separating a solution based on differences in boiling points [14]. Bio-oil distillation is carried out to eliminate unwanted and harmful compounds, such as tar. Distillation at certain temperature conditions is expected to produce pure biofuel free of tar [15].

Based on information about the previous thermal cracking process for making biofuel, this research will be carried out with the thermal cracking process of empty palm oil bunches and then condensed to produce bio-oil, which after being adsorbed with zeolite adsorbent and the last stage is distillation to obtain a product in the form of biofuel. Based on the data from the Ministry of Energy and Mineral Resources that the consumption of biofuels is increasing year by year; therefore, this study focuses on the final results of biofuels in terms of characteristics.

2. Research Method

2.1. Preparation

EFB are washed and dried for two days to remove bound water content. EFB that have been dried are chopped and dried again. After that, the size is reduced using grinding machine to 1-2 mm.

2.2. Pre-treatment

Pretreatment aims to improve the quality of the raw materials that have been prepared. EFB were treated by the klason method through 4 stages; without delignification, the addition of aquadest, and the use of acids in the form of 5% and 10% HCL [16]. Delignification aims to break the lignin bonds in cellulose in oil palm empty fruit bunches and increase the calorific value [17].

In delignification process, both raw materials and the solution were heated at a temperature of 121ºC for 30 minutes, after it was filtered and washed with water to a neutral pH. The resulting residue was weighed and then dried at 105ºC for 1 hour [18-19]. Fruit bunches are washed and dried for two days to remove bound water content. Dried EFB are chopped and dried again. After that, the size is reduced using grinding to 1-2 mm.

2.3. Thermal Cracking

The raw material that has been delignified with 10% HCL is put into a thermal cracking reactor of 3 kg. Then the reactor is set with temperature variations (300°C, 350°C, 400°C, and 450°C) [20]. The heating process lasts 2 hours; during the process, the valve where the stream flow is closed first until the pressure in the reactor rises. After that, the valve is opened slowly. The steam formed is converted into bio-oil using a condenser. The resulting bio-oil is accommodated [21-22].

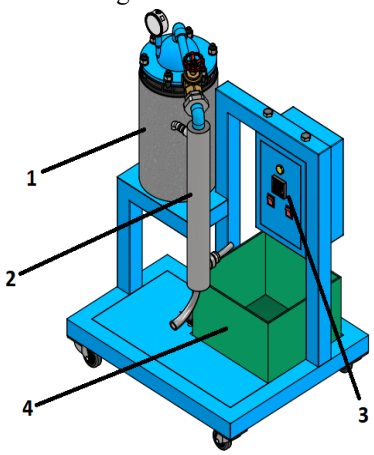

Figure 1. Thermal Cracking Reactor
(1) reactor, (2) condensor, (3) control panel, (4) water tank

2.4. Adsorption using Synthentic Zeolite

Adsorption happens when a substance attracts other substances around it to interact and bind to them [23-24]. The bio-oil obtained was adsorbed using synthetic zeolite, which had been activated using sulfuric acid. The bio-oil and adsorbent were combined in a beaker glass, then heated and stirred at 60°C for 30 minutes, then allowed to stand until the adsorbent and impurities settled, and then the bio-oil was filtered.

2.5. Distillation

Distillation is a way to separate volatile components in biofuel. The differences in the boiling points of biofuel functional compounds such as acetic acid, siringol, guaikol, glyoxaldehyde, glyoxal, and methylglyoxal, and carcinogenic compounds such as tar can be separated to match the standard levels for fuel [25].

The adsorbed bio-oil is put into a distillation flask and

heated using an oil batch. The temperature of the bio-oil in the distillation flask is measured, and its value is recorded on the thermometer, wait for the fractions to separate. Biofuel from the distillation was analyzed physically and chemically.

3. Result and Discussion

From the research that has been carried out, the results of observations with temperature variations in the thermal cracking process of oil palm empty fruit bunches are obtained as follows

3.1. Raw Material Analysis

EFB can be used as a raw material in biofuel production according to its proximate and ultimate analysis. The atomic ratio obtained from the ultimate analysis can be used to show the amount of calorific value used for fuel [3].

In addition to proximate and ultimate analysis, to determine the characteristics of EFB, the researchers determined lignin content by delignification using the Klason method.

In the process of delignification of EFB using HCL at a temperature of 121°C, the results of the residual lignin content in EFB are shown in Figure 2.

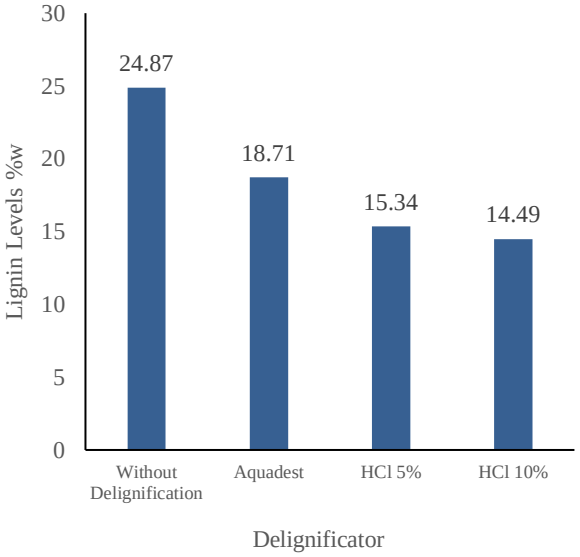

Figure 2. Lignin Levels

Figure 2. shows the lignin levels observed. From the delignification process with HCL, the lignin content in EFB was 15.34% for HCL 5%; 14.49% for HCL 10%; 18.71% in treatment using aquadest, and 24.87% without delignification. This study shows that the greater the concentration of HCL, the lower the remaining lignin content in the raw material. However, in this study, the delignification using aquadest was not far different from the delignification using HCl. This is because the solubility of lignin is limited in acid, or there is still a lot of lignin that is still bound (not soluble in acid). Several studies have shown that the reactivity of the acid catalyst is inhibited by lignin, so it cannot reduce the lignin content significantly and requires a large amount of energy or high temperature (100-230°C) [26]. Lignin is an essential polymeric organic substance widely found in higher plants. The level of lignin in the raw material affects the volume of bio-oil produced, where the higher the lignin content, the smaller the volume of bio-oil.

3.2. Physical Analysis of Biofuel

The physical characteristics of biofuel from the thermal cracking process in the form of color and odor can be seen in Table 1.

Table 1. Physical Analysis of Bio-Oil Before and After Adsorption

Temperature (°C)	Before Adsorption		After Adsorption	
	Color	Odor	Color	Odor
300	Brown Black	Rancid	Brown Black	Rancid
350	Brown Black	Rancid	Brown	Rancid
400	Brown Black	Rancid	Brownis Yellow	Rancid
450	Brown Black	Rancid	Yellow	Rancid

The physical properties of bio-oil before adsorption have the same color and odor at a temperature of 300-450°C and after being adsorbed using synthetic zeolite adsorbents, the bio-oil change at a temperature of 350°C to brown, 400°C brownish yellow and 450°C yellow. The changes were caused by the activated synthetic zeolite attracting tar or impurities during heating and precipitation, so that the color was lighter.

3.3. Quantitative and Qualitative Analysis of Biofuel

Biofuel obtained from the thermal cracking, adsorption, and distillation process can also be observed in the form of volume obtained. The comparison of the weight of the condensate product obtained with the number of EFB used as raw material can be seen in Figure 3.

The difference in bio-oil volume before and after adsorption at a temperature of 300°C is the most adsorbed compared to the others, which is 65 ml. This is because the temperature of 300°C is the initial stage of the thermal cracking process. The biomass in the reactor experienced a slight decrease in weight, such as breaking bonds, the emergence of free radicals, and the formation of carbonyl groups with the release of a bit of water (H_2O), carbon monoxide (CO), and carbon dioxide (CO_2) so that when adsorption was carried out the volume of biofuel decreased quite a lot between another.

Figure 3a-3b. shows the effect of temperature (300, 350, 400, and 450°C) on the volume of biofuel. It can be seen that temperature significantly affects the volume of biofuel. The higher the temperature, the more steam is produced. This is

because the solid raw material will evaporate and turn into steam, reducing the weight of the solid raw material.

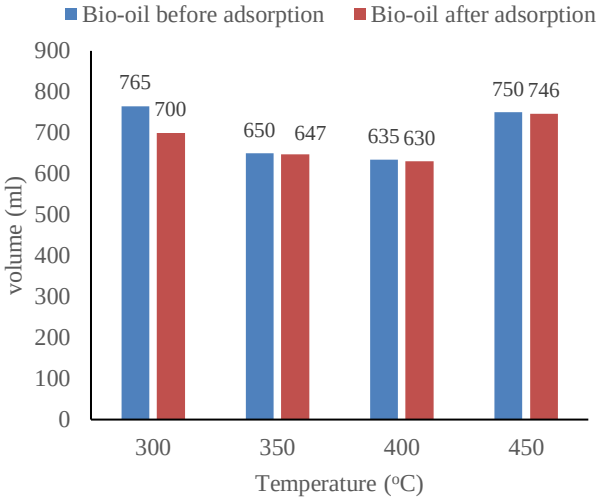

Figure 3a. The Relationship between the use of Adsorbent and Thermal Cracking Temperature on Product Volume

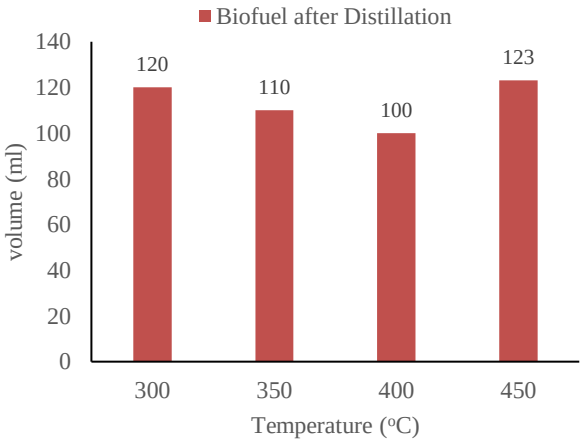

Figure 3b. Biofuel Volume

3.3.1. The Relationship between the use of Adsorbent and Thermal Cracking Temperature on Product Density

Density is one of the physical properties tested to determine the closeness of the characteristics of commercial liquid products to liquid products resulting from thermal cracking [27]. It can be stated that the density is based on the number of carbon chains; the shorter the number of carbon chains, the smaller the density obtained. The results of calculating density data against temperature are shown in Figure 4.

The compounds in the liquid product primarily determine the physical properties of the liquid product resulting from thermal cracking. Increasing or decreasing a compound's concentration will affect the liquid product's characteristics. Based on the research, it is known that the higher the temperature used, the lower the density of the resulting product. High deoxygenation temperatures will increase the occurrence of secondary reactions that lead to the formation of light products. Density itself is influenced by the carbon chain of the compound formed; the higher the density, the longer the number of carbon atoms formed, and vice versa. Most of these compounds are members of the paraffin, naphthenic, or aromatic hydrocarbon classes; each class has different chemical and physical properties.

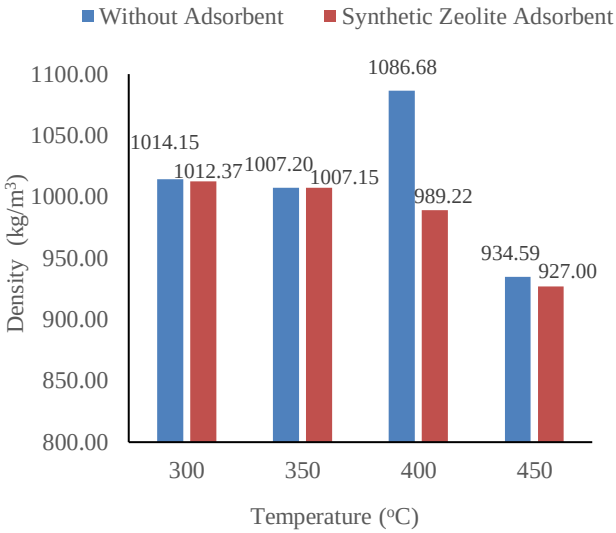

Figure 4. The Relationship between the use of Adsorbent and Thermal Cracking Temperature on Product Density

Based on Figure 4, the density of the thermal cracking liquid product for a temperature of 300-450°C is in the close range, namely the value of 927-1068.68 kg/m^3. This value is almost close to the density of water, which is 1000 kg/m^3 and is still more significant than the density value of kerosene density (835 kg/m^3). The closeness of the density value of the liquid product to the density of water proves that this thermal cracking liquid product still contains water.

3.3.2. The Relationship between the use of Adsorbent and Thermal Cracking Temperature on Product Viscosity

Viscosity describes how fast or slows the liquid flows. The kinematic viscosity of biofuel can be seen in Figure 5.

The increase in kinematic viscosity is proportional to the chain length of the hydrocarbon compounds. The product's viscosity is strongly influenced by the number of paraffinic hydrocarbons formed; the shorter the chain, the more value. Figure 5. shows that the viscosity value decreases when the heating temperature is higher. The increase in viscosity value at higher temperatures is due to the cracking of the bonds in the molecule. The smaller the viscosity, the lighter the fraction contained in the product.

This research shows that the product's kinematic viscosity is in the range of 1.16 – 1.79 mm^2/s. This difference in viscosity is because when testing the resulting biofuel product, the ball's fall was not hampered by the viscosity of the biofuel.

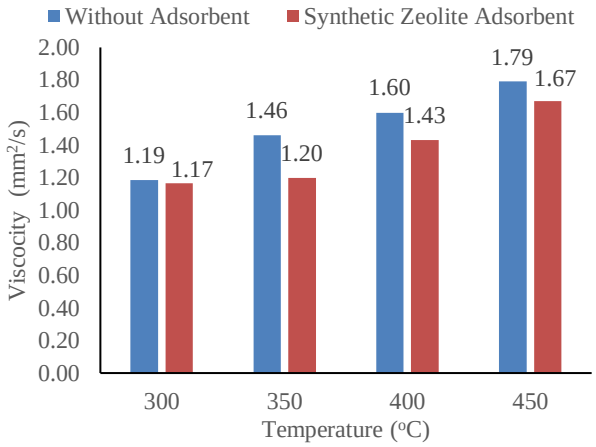

Figure 5. The Relationship between the use of Adsorbent and Thermal Cracking Temperature on Product Viscosity

Based on the analysis of the physical properties of biofuel by looking at the results of density and viscosity analysis, the biofuel obtained still contains water. The presence of this water indicates the formation of water during the reaction. The reaction for the formation of water occurs because the expulsion of oxygen is less than perfect, so there is still a large amount of oxygen. This large amount of oxygen allows normal combustion reactions to produce water and carbon dioxide. As a result, the reaction that occurs is a combustion reaction, not thermal cracking. This error is found in the procedure for removing oxygen from the reactor. The condition of the reactor that is free of oxygen cannot be measurably identified.

3.3.3. The Relationship between the use of Adsorbent and Thermal Cracking Temperature on Product Flash Point

Flash point is one of the essential practical parameters; the higher the flash point, the higher the safety during handling, transportation, and storage. The flash point of biofuel can be seen in Figure 6.

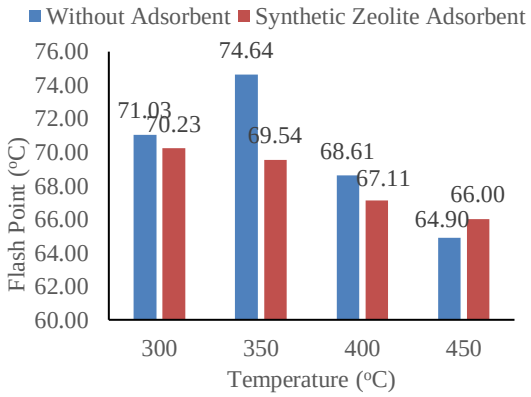

Figure 6. The Relationship between the use of Adsorbent and Thermal Cracking Temperature on Product Flash Point

3.3.4. The Relationship between the use of Adsorbent and Thermal Cracking Temperature on Product Moisture Content

Water is a molecule that is always present in all materials and is a determinant of the quality of a material. Moisture content is a compound that must be reduced or eliminated. The highwater content in fuel will inhibit combustion [29]. The results of the water content analysis are in Figure 7.

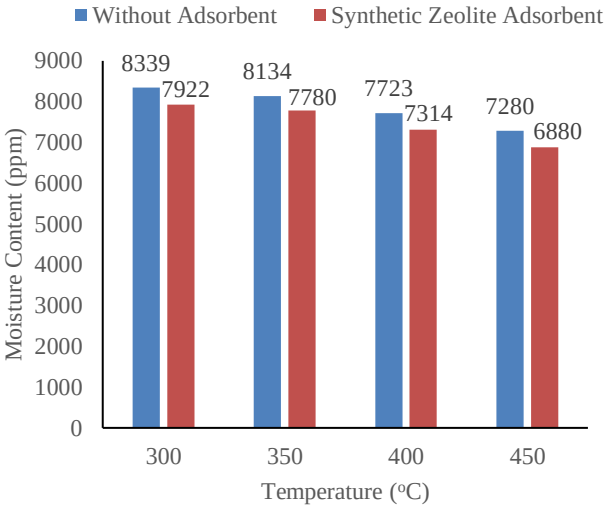

Figure 7. The Relationship between the use of Adsorbent and Thermal Cracking Temperature on Product Moisture Content

In Figure 7 the higher the temperature of the water content, the smaller the water content; from the research results, the value of the range of 6880-7922 ppm is still more significant when compared to the value of the water content of the fuel (500 ppm). The water content proves that this thermal cracking liquid product still has a lot of water content. This water content indicates the formation of water during the reaction; this occurs at the time of storage of biofuel, where during the condensation process, the water content is incorporated into the biofuel.

3.4. GC-MS Analysis Result

GC-MS (Gas Chromatography - Mass Spectroscopy) is an instrument consisting of two analytical methods. In this study, the identification of liquid products was carried out using the GC-MS method. Identification is made to determine the distribution of compound components in biofuel resulting from thermal cracking.

The chromatography peaks show the types of components contained in biofuel; it can be seen that the number of components identified reached 51 types of components, as indicated by the formation of 51 peaks. The following is the result of the chromatography breakdown of GC-MS against biofuel thermal cracking, which is shown in Figure 8.

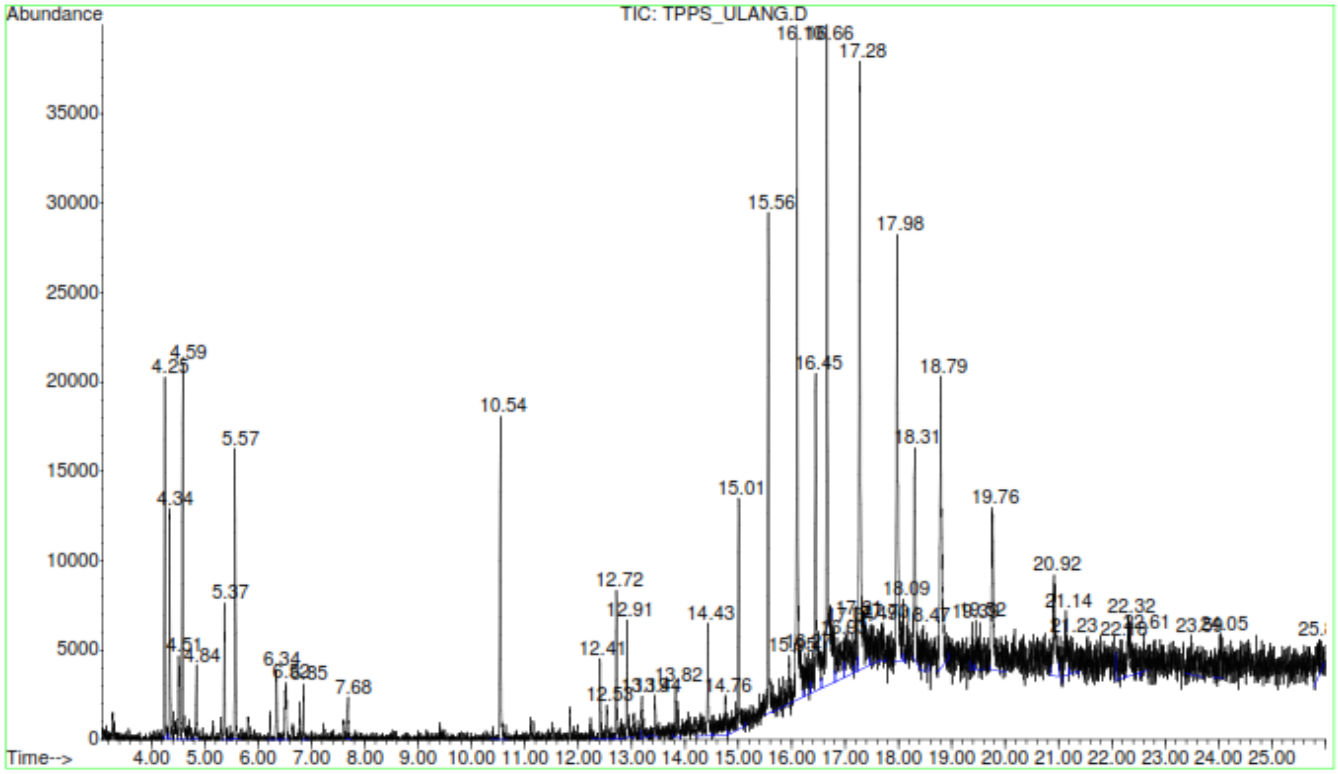

Figure 8. EFB Biofuel Chromatography

The results of identifying these components are tabulated in Table 2.

Table 2. Fraction and Chemical Compound Composition of Biofuel

Fraction	Component	Compound Composition (%)
$<C_5$	Gas	0.36
C_5-$C1_5$	Gasoline, Kerosene, Naphtha	45.07
C_{15}-C_{18}	Diesel	13.42
C_{18}-C_{20}	Lubricant	9.52
$>C_{20}$	Residue	14.61
-	Other	17.02
	Total	100.00

Table 2 shows that the main components of the thermal cracking product liquid are phenol, acid compounds, oxygenated compounds, and some aromatic compounds. Phenol and its derivatives are the compounds with the most extensive composition. These phenolic compounds presence can increase the liquid product's flammability. However, because this liquid product still has a high content of water and oxygenate compounds, the combustion of this liquid product is still relatively long. The presence of these acidic compounds also causes the liquid product to be corrosive to many metals except stainless steel.

After carrying out the characteristics of the fractions and chemical compounds of the liquid product in Table 2, if you look at the results of the component analysis, it shows that the liquid product resulting from thermal cracking is dominated by C_5-C_{15} compounds, reaching 45.07%. This is not in line with the components of diesel/diesel, which are dominated by C_{15}-C_{18} compounds. Thermal cracking liquid products based on their constituent components are more specific to the kerosene component. However, this liquid product still does not have the high combustion power needed to be used as fuel.

4. Conclusion

Based on the research that has been done, it can be concluded that the characteristics of the raw material in the form of EFB in terms of the delignification process with HCL; 15.34% lignin obtained in EFB treated with 5% HCL; 14.49% for 10% HCL; 18.71% obtained in using aquadest; and 24.87% lignin obtained if without any delignification treatment. The characteristics of the raw material obtained from EFB have the potential to be converted into fuel.

The physical properties of biofuel are as follow; brownish black color and rancid odor after being adsorbed, the color changes to brown, brownish yellow and yellow. The resulting volume after distillation is 100-123 ml. The resulting liquid product is close to the optimum condition at a temperature of 450^{o}C with a density of 927 kg/m^3, viscosity of 1.67 mm^2/s, a flash point of 66oC, and water content of 6880 ppm. Based on the results of biofuel analysis using GC-MS, the C atomic chain is dominated by C_5-C_{15} compounds of 45.07%.

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