



Comparative Evaluation of Biofuel Products from EFB (Empty Fruit Bunch) and PKS (Palm Kernel Shell) Based on GC-MS

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Abstract. Palm Kernel Shell (PKS) and Empty Fruit Bunch (EFB) are potential bioenergy sources because they contain lignocellulosic (cellulose, hemicellulose and lignin) so that they can be converted into biofuel through the thermal cracking process. This research was conducted in a lab scale experiment using a fixed bed reactor for 90 min with temperatures ranging from 300 °C–450 °C and using natural zeolite adsorbent as much as 7 g. The purpose of this study was to determine the hydrocarbon chain of biofuel products from PKS and EFB. The biofuel product that produces the highest yield is obtained from a temperature condition of 450 °C, using a natural zeolite adsorbent of 7 g. The result of thermal cracking in the form of liquid is separated into a clear product (liquid smoke) and a viscous product (biofuel). The biofuel was then tested using GC-MS to determine its chemical components. PKS and EFB have the same kind of most dominant fraction (C5-C14) with kerosene content where the compositions obtained are 80.93% (PKS) and 45.07% (EFB).

Keywords: Palm Kernel Shell · Empty Fruit Bunch · Thermal Cracking · GC-MS

1 Introduction

The limitations of fossil fuels, environmental pollution and the issue of global warming have become topics that attract international attention today. Fossil fuels are expected to run out in 2060. Biomass is a sustainable energy source that may be used to replace fossil fuels and mitigate environmental impact [1]. In Indonesia, palm oil waste is a plentiful source of biomass. According to the Central Statistics Agency, Indonesia's palm oil output in 2020 will exceed 44.8 million tons of crude palm oil (CPO) or around 139.71 million tons of fresh fruit bunches. Processing of fresh fruit bunches produces waste in the form of Palm Kernel Shell (PKS) as much as 6.7% and Empty Fruit Bunch (EFB) as much as 22.5% [2].

The specific characteristics of biomass affect its role as a fuel. PKS and EFB are potential sources of bioenergy because they contain lignocellulose (lignin, hemicellulose and cellulose). Thus are energy source that can be transformed into liquid, charcoal, or combustible gas [3]. In this research, the conversion process is carried out by thermal cracking to produce bio-oil. Thermal cracking of biomass is expected to increase the energy density contained in the product, in order to produce better work compared to direct combustion of the biomass. Thermal cracking is the decomposition of the chemical content of biomass by utilizing heat in the absence of a mixture of oxygen at a temperature of around 200 °C–600 °C [4, 5]. Numerous advantages and possibilities for usage as a replacement for fossil fuels. According to Klass, the gas produced generally consists of H₂, CO₂, CO, CH, C₂H, organic gas and water vapor [6]. The thermal cracking of lignocellulosic biomass consists of four stages: the emission of water vapor, the decomposition of hemicellulose, the decomposition of cellulose, and the decomposition of lignin. Hemicellulose breakdown typically occurs between 220 °C and 315 °C. Cellulose decomposes at a temperature of 315 °C–400 °C and lignin usually decomposes at a temperature of 150 °C to 900 °C [7]. The most important aspect in this research is to obtain the highest amount (quantity) of bio-oil through the thermal cracking process. From the experimental results, the physical characteristics of bio-oil that are close to optimal are obtained at a temperature of 450 °C. This study aims to determine the specifications of the bio-oil produced in terms of the carbon chain using GC-MS.

Thermal studies in large thermal cracking reactors and biofuel product analysis can be time-consuming, especially when examining pretreatment technique, adsorbent loading, and thermal cracking temperature [8, 9]. One solution is to use thermal cracking-gas chromatography/mass spectrometry. Gas chromatography (GC), is a type of chromatography used in organic chemistry for separation and analysis [10]. GC can be used to test the purity of a particular material, or to separate various components from a mixture. In some situations, GC can assist in identifying a complex. Even if there are many studies on thermal cracking of biomass in the literature [11–13]. Temperature and feedstock have been studied extensively on thermal cracking yield. This study found that the yield of the liquid product grows until a specific temperature, then decreases due to the secondary reaction's increase in gas yield. Few studies have examined the impact of thermal cracking temperature and feedstock on product analysis. This study will highlight how raw material differences affect GC-MS-analyzed products.

2 Research Method

2.1 Thermal Cracking Process

The process and concept of biofuel production starting from raw materials to producing liquid fuel in this study are based on the author's previous experiments. Both biofuel product from EFB and TKS were treated with the same equipment and condition [14, 15].

When conducting the thermal cracking process, 3 kg of each PKS and EFB that's has been prepared before were loaded into a fixed bed reactor. The temperature set-up for thermal cracking were 300 °C, 350 °C, 400 °C, and 450 °C. After the whole processes of thermal cracking was completed, PKS and EFB biofuel obtained from temperature

of 450 °C resulting into the highest amount of yield. Both samples then analyzed using GC-MS.

2.2 Composition Analysis by GC-MS

The GC-MS analysis was conducted in South Sumatra Police Forensic Laboratory. Thermo Scientific™ ISQ™ 7000 Single Quadrupole GC-MS system were used to identify the components of the liquid products. The injector was set to 260 °C, and 1 ml of sample was injected. The oven temperature was set at 70oC for five minutes, then increased at a rate of 10oC per minute until it reached 270oC, where it remained for five minutes. Helium with a flow rate of 1 ml per minute is the carrier gas. The detector is a mass spectrometer (MS).When conducting the thermal cracking process, 3 kg of each PKS and EFB that's has been prepared before were loaded into a fixed bed reactor. The temperature set-up for thermal cracking were 300 °C, 350 °C, 400 °C, and 450 °C. After the whole processes of thermal cracking was completed, PKS and EFB biofuel obtained from temperature of 450 °C resulting into the highest amount of yield. Both samples then analyzed using GC-MS.

3 Result and Discussion

The obtained biofuels were analyzed using gas chromatography mass spectrometry (GC-MS). In this study, GC-MS analysis is used because it can identify any components contained in biofuels. The compounds separated from the GC analysis will come out of the column and flow into the MS, then these compounds are identified based on their molecular weights [16]. This is to determine the composition of the compounds in the resulting biofuel. Although the yield of biofuels produced is quite low, this study aims to determine the composition of biofuels produced from two different sources of raw materials, namely EFB and PKS. The composition of biofuels varies widely due to the many variables in the thermal cracking of biomass. A wide variety of compounds are reported for the composition of each organic component of the biofuel. The literature on the composition of biofuels is summarized in various literatures [17–19].

It can be seen from Fig. 1 that the results of the GC-MS from PKS biofuel identify thermal cracking oil which has 81 chromatographic peaks that indicate the possibility of

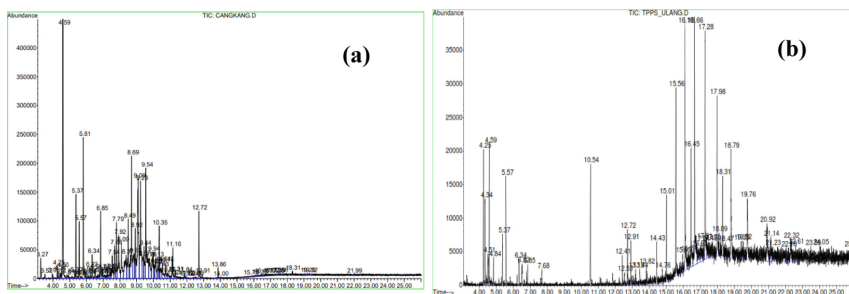


Fig. 1. GCMS Graph of (a) PKS Biofuel and (b) EFB Biofuel.

certain compounds contained in the biofuel after the thermal cracking process, with an overall retention time of 22 min, while Fig. 2 is the result of GC-MS from EFB biofuel with 51 peaks with a retention time of 26 min. Retention time is the time it takes for the solute (component) to leave the column and reach the detector. The retention time at each peak represents the compounds present in the liquid fuel product. From the retention time of each peak, the % area which is the composition of hydrocarbon compounds in liquid fuel products is obtained which is presented in Table 1 and Table 2. Based on the % area in the two tables, the product liquid fuel fractions are tabulated in Table 3.

Table 1 further describes the list of compounds as well as the % area in the resulting biofuels. Many compounds were found, such as naphthalene, phenol, furfural and other minor compounds such as silicic acid etc. Although phenolic compounds held the highest percentage of all compounds found, the selectivity of the resulting compounds was not high.

The list of compounds with relatively high content obtained from biofuel with EFB as raw material under optimum conditions is shown in Table 2. and the graph of GC-MS biofuel with EFB as raw material is shown in Fig. 1(b).

The results of GC-MS analysis from PKS biofuel showed that the liquid fuel fraction produced was classified into 6 groups, namely the gas fraction ($<C_5$) of 1.09%; gasoline (C_5-C_{14}) by 80.93%; kerosene/diesel ($C_{15}-C_{17}$) by 14.23%; lubricating oil ($C_{18}-C_{20}$) by 0.86%; residue ($>C_{20}$) of 1.77%; and other compounds by 1.12%. The most dominant fraction in biofuel products is the gasoline fraction (C_5-C_{14}) which is 80.93%. The presence of kerosene (C_5-C_{14}) in the sample is due to the continuous cracking process so that more light hydrocarbon chains are produced. The diesel fraction shows the most dominant fraction after the gasoline fraction.

The resulting product contains substances that have a fraction $<C_5$. Hydrocarbons below C_5 are gas-phase hydrocarbons and cannot be condensed, so they evaporate into the air. In this study, the condensation process has not run optimally because the cooler does not work optimally so that the product that comes out is still in the form of gas. Meanwhile, the content of $C_{18}-C_{20}$ and $>C_{20}$ in the sample was due to the palm oil shell not being fully cracked. This is presumably because the fatty acids contained in the raw materials have not reacted completely. In addition, there are also other compounds that are not included in the hydrocarbon chain by 1.12%. This is because the thermal cracking process forms side reactions such as isomerization, alkylation, and cracking, so that the thermal cracking process can produce by-products in that form.

As for the results of GC-MS from EFB biofuel, it can be seen that the main components of the thermal cracking product liquid are phenol, acid compounds, oxygenate compounds and some aromatic compounds. Phenol and its derivatives are the compounds with the largest composition. The presence of these phenolic compounds can increase the flammability of the liquid product, but 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.

Table 1. Relative proportions (% Area) of the main compounds of PKS Biofuel Obtained

Compounds	% Area
Furfural	0.57
Cyanomethyl 2-chloroethyl sulfide	0.09
2-Cyclopenten-1-one, 2-methyl	0.19
Hydroperoxide, 1-ethylbutyl	0.26
Piperazine, 2-methyl	0.16
2-Furancarboxaldehyde, 5-methyl	0.41
Phenol	24.36
Benzene, 1,3,5-trimethyl	0.37
Bicyclo[3.2.0]heptan-2-one	0.09
2-Cyclopenten-1-one, 2,3-dimethyl	0.17
Phenol, 2-methyl	1.79
Acetic acid, phenyl ester	0.05
Phenol, 4-methyl	1.48
N-Cyano-2-methylpyrrolidine	0.07
Phenol, 2-methoxy	3.17
Phenol, 2,5-dimethyl	0.25
Propanoic acid	0.13
Phenol, 2-ethyl	0.28
Phenol, 3,5-dimethyl	0.70
Phenol, 3-ethyl	0.43
Phenol, 2,5-dimethyl	0.28
Hydrazine, (2-methoxyphenyl)	0.18
Phenol, 2-methoxy-4-methyl	1.78
Undecane, 2,5-dimethyl	0.32
1-Cycloheptene, 1,4-dimethyl-3-(2-methyl-1-propene-1-yl)-4-vinyl	0.15
2-Methylbicyclo[4.3.0]non-1(6)-ene	0.21
1,4-Benzenediol, 2,3,5-trimethyl	0.30
1-Tetradecanol, 14-chloro	0.20
Oxirane, tetradecyl	0.27
4-Methyldocosane	0.29
Octane, 3,6-dimethyl	1.07
Phenol, 4-ethyl-2-methoxy	1.18

(continued)

Table 1. (continued)

Compounds	% Area
Tridecane	1.43
Naphthalene, 1-methyl	2.39
Naphthalene, 2-methyl	1.92
Nonahexacontanoic acid	2.53
Dodecane, 2,6,10-trimethyl	2.57
Benzoic acid, 2-methoxy-, methyl ester	0.50
Tetradecane	4.87
Naphthalene, 2,3-dimethyl	2.22
Naphthalene, 2,7-dimethyl	1.82
Naphthalene, 2,6-dimethyl	7.25
Undecane	5.24
Naphthalene, decahydro-1,6-dimethyl-4-(1-methylethyl)	1.48
Decahydro-4,4,8,9,10-pentamethylnaphthalene	1.81
Pentadecane	5.35
Naphthalene, 2-(1-methylethyl)-	2.03
Naphthalene, 1,6,7-trimethyl	2.04
Dodecane, 6-cyclohexyl	0.61
Naphthalene, 2,3,6-trimethyl	1.53
Azulene, 4,6,8-trimethyl	0.81
Hexadecane	1.23
2,2'-Dimethylbiphenyl	0.90
Phenol, 4-(1-methylpropyl)	1.01
Pentacosane	0.69
1,2,3-Triazole-4-methanol, 1-(4-aminofurazan-3-yl)	0.32
Cyclopentanecarboxaldehyde, 2-methyl-3-methylene	0.13
Pentadecane, 2,6,10,14-tetramethyl	1.36
Undecanoic acid, methyl ester	0.27
N,N-Diethylhept-1,3,5-trieneamine	0.10
Nonacosane	0.13
Heptadecane	0.05
1,3,5-Ethanylylidene-2-thiacyclobuta[cd]pentalen-7-one, octahydro	0.03
2-Formylbenzeneboronic acid	0.08

(continued)

Table 1. (continued)

Compounds	% Area
Methoxyacetic acid, nonyl ester	0.07
Hexadecanoic acid, methyl ester	1.64
alpha.-D-Glucopyranoside, methyl 4,6-O-nonylidene	0.09
1,1'-Bicyclohexyl, 4-methyl-4'-propyl	0.28
Silane, [(4-ethoxycyclohexyl)oxy]trimethyl	0.03
1-Nitro-9,10-dioxo-9,10-dihydro-anthracene-2-carboxylic acid	0.21
Cyclotrisiloxane, hexamethyl	0.35
Silicic acid, diethyl bis(trimethylsilyl) ester	0.11
Silicic acid, diethyl bis(trimethylsilyl) ester	0.53
4-Dehydroxy-N-(4,5-methylenedioxy-2-nitrobenzylidene)tyramine	0.05
Silicic acid, diethyl bis(trimethylsilyl) ester	0.16
2,3,4-Trimethoxyphenylacetone nitrile	0.08
Silicic acid, diethyl bis(trimethylsilyl) ester	0.12
Silicic acid, diethyl bis(trimethylsilyl) ester	0.13
Cyclotrisiloxane, hexamethyl	0.12
Silicic acid, diethyl bis(trimethylsilyl) ester	0.05
Silane, 1,4-phenylenebis[trimethyl	0.02

After the characteristics of the fractions and chemical compounds of the liquid product are shown in Table 3. If you look at the results of the component analysis, it shows that the liquid product resulting from thermal cracking is dominated by C₅-C₁₅ compounds reaching 45.07%. This is not in line with the components of diesel/diesel which are dominated by C₁₅-C₁₈ 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.

As compared to biofuels obtained with EFB raw materials, the selectivity of biofuel compounds is not high.

Most of the compounds have not completely decomposed into the desired compounds. In contrast, with shell raw materials, the selectivity of the compound increases and the resulting product is narrower and more controlled. Hydrocarbons, in particular, are highly desirable in biofuel due to their potential fuel applications. To be more specific, aromatic hydrocarbons are used as both vital industrial chemicals and essential octane-boosters in transportation gasoline [20].

Table 2. Relative proportions (% Area) of the main compounds of EFB Biofuel Obtained

Compounds	% Area
Oxirane, 2,2-dimethyl-3-propyl	2.76
Oxirane, 2-methyl-2-pentyl	1.92
Benzene, 1-ethyl-4-methyl	0.69
3-Methylpyridazine	3.34
Benzene, 1,2,3-trimethyl	0.55
Phenol, 2-methyl	1.16
Phenol, 4-methyl	2.36
Benzaldehyde, oxime	0.85
Benzene, 1-methoxy-4-methyl	0.76
2,2-Dimethylocta-3,4-dienal	0.35
3-Methoxy-4-methylaniline	0.26
Phenol, 2-(1-methylpropyl)	2.72
1,2-Benzenedicarboxylic acid, butyl 2-ethylhexyl ester	0.78
Butane, 1-isocyanato	0.36
Hexadecanoic acid, methyl ester	1.18
n-Hexadecanoic acid	1.09
Decane, 1,1'-oxybis	0.47
1-Hexadecanol	0.41
Tetradecane, 4-methyl	0.66
Decane	1.71
Borane, 2,3-dimethyl-2-butyl(er)	1.07
Hexadecane, 2,6,10,14-tetramethyl	2.80
Eicosane, 7-hexyl	4.36
5H-Inden-5-one, octahydro-7a-methyl-3-(2-methylpropyl)	1.23
Heneicosane	7.83
Nickel, (eta.-4-diallyl ether)-(2,4-dimethyl-3-pentylisonitrile)	0.97
1,2-Benzenedicarboxylic acid, mono(2-ethylhexyl) ester	4.23
Eicosane	10.29
1,2-Dihydroanthra[1,2-d]thiazole-2,6,11-trione	1.48
1,3-Bis(trimethylsilyl)benzene	1.55
Cyclotrisiloxane, hexamethyl	0.56
Heneicosane	8.25

(continued)

Table 2. (continued)

Compounds	% Area
Silicic acid, diethyl bis(trimethylsilyl) ester	0.94
Silicic acid, diethyl bis(trimethylsilyl) ester	0.54
Heptadecane	5.39
2-Ethylacridine	0.41
2,6,10,14,18,22-Tetracosahexaene,2,6,10,15,19,23-hexamethyl	2.89
1,1,1,3,5,5,5-Heptamethyltrisiloxane	0.83
Eicosane	4.71
Cyclotrisiloxane, hexamethyl	0.35
Silicic acid, diethyl bis(trimethylsilyl) ester	0.87
Octadecane, 3-ethyl-5-(2-ethylbutyl)	3.31
Docosane, 9-butyl	2.26
Dodecahydropyrido[1,2-b]isoquinolin-6-one	1.08
Silicic acid, diethyl bis(trimethylsilyl) ester	0.84
1,2-Bis(trimethylsilyl)benzene	0.93
1,2-Bis(trimethylsilyl)benzene	1.87
Cyclotrisiloxane, hexamethyl-	0.49
Silicic acid, diethyl bis(trimethylsilyl) ester	1.27
1H-Indole, 5-methyl-2-phenyl	0.99
Cyclotrisiloxane, hexamethyl	1.01

Table 3. Comparison of Fraction and Chemical Composition of Biofuel from PKS and EFB

Fraction	Component	Composition (%)	
		PKS	EFB
<C ₅	Gas	1,09	0,36
C ₅ -C ₁₄	Kerosene	80,93	45,07
C ₁₅ -C ₁₇	Diesel	14,23	13,42
C ₁₈ -C ₂₀	Lubricants	0,86	9,52
>C ₂₀	Residue	1,77	14,61
-	Others	1,12	17,02
Total		100,00	100,00

4 Conclusion

The biofuel produced will increase with increasing temperature. Biofuel analysis using gas chromatography can be concluded that PKS and EFB have the same dominant fraction (C₅-C₁₄) with kerosene content where the compositions obtained are 80.93% PKS and 45.07% EFB. In this case, the thermal cracking process of the PKS is more dominant than the EFB. This is because the PKS cracking process is more perfect than EFB so that more light hydrocarbon chains are produced.

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