Preliminary Study on The Bio-Oil Production from Multi Feed-Stock Biomass Waste via Fast Pyrolysis Process

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ARTICLE INFO	ABSTRACT
	Biomass is a good resource for renewable energy. Biomass can be converted into bio-fuel (bio-oil) through a catalytic fast pyrolysis process. Previous studies only used single feedstock biomass as raw material for bio-oil production. In this study, bio-oil production is based on a multi-feedstock biomass waste consisting of rice husk, sugar cane bagasse, and palm oil empty fruit bunch. The mixture of biomass waste as a raw material is expected to enhance the yield and quality of the bio-oil produced. This study aimed to investigate the bio-oil products obtained from catalytic pyrolysis of the biomass waste mixture. Mixture Design from Design Expert was used to study the effect of biomass composition on the bio-oil products. Each biomass, i.e., rice husk, sugar cane bagasse, and palm oil empty fruit bunch, was previously chopped and sieved into a uniform 60 mesh. The pyrolysis process was conducted at 500°C with an N_2 flow rate of 3 L min^{-1}. The mixture of biomass waste husks contains more phenolic compounds than single-feedstock. The chemical characterization also showed that the multi-feedstock bio-oil compound was dominated by aldehydes, esters, and phenolic compounds.

1. Introduction

The Indonesian people's tendency to use fossil (non-renewable) energy is exorbitant, especially oil and coal [1]. Based on study data at the Indonesian Energy Outlook (*Outlook Energi Indonesia*/ OEI) 2019, which collects data on national energy conditions until 2050, oil production has decreased in the last ten years due to vast exploitations [2]. This decline in oil production will also impact Indonesia's vulnerability to fluctuations in the availability and prices of petroleum energy in the international market. Increased use of renewable energy is required to solve the energy dilemma. Biomass can be defined as all organic material derived from plants or animals, other living organisms, and waste, which is promising to be utilized as a renewable energy source and is carbon neutral [3,4]. The processing of agricultural waste into alternative fuels (bio-oil) through a pyrolysis technique, i.e.,

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the fast catalytic pyrolysis process, has received more attention as the bio-oil can be produced in high yields and quality as fuel or raw material for chemical industries [5].

Previous studies carried out the pyrolysis of biomass into bio-oil but only used single feedstock biomass as raw material for bio-oil production. It is possible to employ biomass-based bio-oil multi-feedstock to increase the adaptability of biodiesel manufacturing processes [6]. Previous study by Fardhyanti *et al.,* on the bio-oil production based on single feedstock biomass using a non-catalytic pyrolysis process at a high temperature (500°C-700°C) with a pressure of 1 atm produced a small bio-oil yield of 50%, which was not fulfilling the Indonesian National Standard (SNI) and not close to the quality of biodiesel/diesel [7,8]. The yield and characterization of bio-oil from different types of biomass are essential to identify, both in terms of chemical components and quality. For this reason, it is required to carry out several yield test analyses, such as the determination of pH and phenol levels, the analysis of specific gravity, and identification using Gas Chromatography-Mass Spectrometry (GC-MS).

The thermal decomposition of organic components without oxygen in producing liquids, gases, charcoal, or pyrolysis can be described as the thermal degradation of materials without oxygen [9]. The process is carried out at a relatively high temperature, around 300-550°C, within 4-7 hours, depending on the method of manufacture and the raw materials used with limited air or even in the absence of air [10,11]. The main products resulting from pyrolysis are gas (fuel/gas), liquid (bio-oil) and solid (charcoal/charcoal) [12–15]. The pyrolysis process has several advantages, including the energy content of the product and the resulting high conversion ratio. The resulting products can be upgraded into basic materials for other purposes and easier process control [16]. The fast pyrolysis method is a pyrolysis method that operates at high temperatures around 400-600°C with a heating rate of 100°K/sec in the absence of oxygen [17]. However, less bio-oil will be produced if the temperature exceeds 600 °C. The cause of the low production of bio-oil is due to the decomposition and evaporation of organic compounds in the form of hemicellulose, cellulose and lignin in the gas phase. The biomass will be converted into organic steam, pyrolysis gas and bio-charcoal products during the process. In this process, about 70% of the biomass weight will be converted into bio-oil [18]. According to Noronha *et al.,* fast pyrolysis is the best method used in the production of bio-oil because, in addition to the fast process, the product produced is also more abundant than the bio-oil product in the slow pyrolysis method [19].

According to previous studies, bio-oils production focuses on only one biomass waste source. In the present research, the utilization of multi-feedstock biomass waste was studied. Rice husk ash, sugarcane bagasse, and oil palm empty fruit bunch were the sources of the biomass.

2. Materials and Methods
2.1 Materials

Biomass wastes used in this study were rice husk (RA), sugarcane bagasse (SB), and oil palm empty fruit bunch (OPEFB). The rice husk and sugarcane bagasse were obtained from local farmers around Semarang, Central Java, Indonesia, while the palm oil empty fruit bunch was acquired from Pangkalan Bun, Central Kalimantan, Indonesia.

2.2 Methods

2.2.1 Raw material preparation

Each biomass waste was first washed with deionized water and cut into small pieces. The biomass wastes were then dried in an oven at 105°C overnight and subsequently stored at room temperature. Crushing and sieving were performed to obtain a uniform size of the biomass wastes (60 mesh).

2.2.2 Multi-feedstock composition

The composition of the biomass feedstock was varied by using Mixture Model with a linear model with one replication. The mixture design is shown in Table 1.

Table 1
Mixture design of multi-feedstock biomass waste pyrolysis

Run	Component 1 Rice Husk	Component 2 Oil Palm Empty Fruit Bunch	Component 3 Sugarcane Bagasse	Response 1 Yield Bio-Oil	Response 2 Yield Biochar
1	100	0	0		
2	0	100	0		
3	0	0	100		
4	33.3333	33.3333	33.3333		
5	66.6667	16.6667	16.6667		
6	100	0	0		
7	16.6667	66.6667	16.6667		
8	16.6667	16.6667	66.6667		

The effect of component composition on the yield of bio-oil and bio char can be expressed by a quadratic equation, as shown in Eq. (1).

$$y = \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \tag{1}$$

where y is the response variable which is yield of bio-oil and bio char, x_1, x_2 and x_2 are the biomass composition, and β_i is the quadratic linear constants.

Biomass Waste Pyrolysis. A lab-scale fixed bed reactor was used to perform the pyrolysis of biomass wastes. The reactor had a diameter of 50 cm and a height of 70 cm. With nitrogen flowing at a rate of 3 L min-1, the operational parameter for this process was at a temperature of 500°C. 1 kg of biomass waste mixture with a composition as shown in Table 1 was fed into the reactor. The nitrogen was blown from the bottom of the reactor to remove the excess air inside the reactor. Once the gas burner starts, the vapour mixture will be produced as a function of time due to the pyrolysis process. The vapour was then flown through the double condensing system with a water cooler temperature of 5°C to obtain the bio-oil. In a gas bag, the non-condensable gas was gathered. Figure 1 shows the design of the pyrolysis reactor.
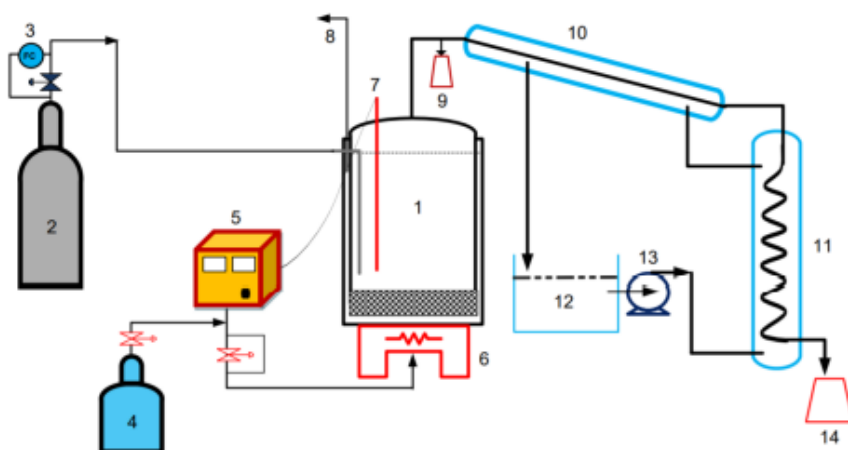
Fig. 1. Schematic diagram of fast pyrolysis reactor, i.e.: (1) fast pyrolysis reactor; (2) N_2 gas storage tank; (3) flow meter; (4) Liquefied Petroleum Gas (LPG) storage tank; (5) temperature control; (6) burner; (7) thermocouple; (8) reactor closure (9); tar collector; (10) condenser I; (11) condenser II; (12) cooler bath; (13) pump; and (14) bio-oil tank

2.2.4 FTIR (Fourier Transform Infrared) analysis

The functional group of biomass waste material was examined by using FTIR-UATR Perkin Elmer Spectrum Two. Spectral scanning range from 4000-400 cm^{-1}, with a scanning number of 16, 4 cm^{-1} resolutions, and data interval of 1 cm^{-1}.

2.2.5 XRD (X-ray Diffraction) analysis

The crystallinity of the biomass waste was characterized by the XRD patterns obtained using Cu radiation at 40 kV and 30 mA, in the scanning angle θ - 2θ ranging between 10 and 79.987° with a scan rate of 2° min^{-1} and step size of 0.0189°.

2.2.6 Py-GCMS analysis

Using Py-GCMS, the distribution of organic products inside the biomass was studied. A sample weighing between 500 and 600 g was placed into an eco-cup made of SF PY1-EC50F and sealed with glass wool. The eco-cup was then pyrolyzed using a multi-shot pyrolyzer (EGA/PY-3030D) linked to a GC/MS QP-2020 NX (Shimadzu, Japan) and outfitted with an SH-Rxi-5Sil Column MS for one minute at 500 degrees Celsius (with a film thickness of 30 mm x 0.25 mm; id 0.25 mm). It also has Helium as a gas carrier and 70 eV electrons. The procedure was run at 20.0 kPa (15.9 mL min-1 and column flow of 0.61 mL min-1). A minute of stability followed by a 5°C heating rate increase to 280°C marked the GC temperature profile, which began at 50°C. The temperature was held constant at 280°C for 13 minutes. By matching the retention period with the mass spectral data in NIST LIBRARY 2017, the pyrolysis product was later determined. [20].

2.2.7 GC-MS (Gas Cromatography Mass Spectrometry) analysis

The bio-oil product from the pyrolysis process was then analysed by using GC-MS Shimadzu GC 2010 Plus with a column of Rtx-5MS. Column specification was 30 m in length with 0.22 mm internal diameter. Helium was used as the gas carrier. Column pressure was operated at 13.7 kPa, with a flow

rate of 25.9 cm s^{-1}. Column temperature ranged between 70 – 300°C with a split ratio of 73.0. Injection and interface temperatures were operated at the same temperature, which was 250°C. The column flow rate was 0.5 mL min^{-1} with a total flow rate of 40 mL min^{-1}.

3. Results
3.1 FTIR

FTIR analysis is typically used to identify the absorption spectrum of a compound by collecting high-resolution information with a spectral range between 4000 and 400 cm^{-1}. FTIR can easily determine the detailed characteristics of the surface of the compound to obtain information on the functional groups present in the biomass. The FTIR spectra were analysed by setting the peak ratio and then compared with the calibration spectra measured under the same conditions to exhibit different chemical bonds in the tested material.

Figure 2 displays typical peaks in the FTIR spectrum, showing that there were C-H group strains in rice husk biomass (RA), sugarcane bagasse (SB), and OPEFB with a wave number of 2919.01 cm^{-1}, 2917.87 cm^{-1}, and 2917.01 cm^{-1}, respectively. C-H groups were found at wave numbers 2840 and 2937 cm^{-1}, identifying the presence of lignin compounds in the biomass [21]. Meanwhile, C=C group strains occurred in rice husk biomass, sugarcane bagasse, and OPEFB with a wave number of 1643.40 cm^{-1}, 1603.89 cm^{-1}, and 1625.70 cm^{-1}, respectively. The stretch of the group followed the standard strain group C=C in the alkene type wave range of 1600-1680 cm^{-1} [22]. The phenolic O-H functional group was also found, with a wave number of 3300.55 cm^{-1}, 3335.17 cm^{-1}, and 3285.09 cm^{-1} in rice husks, sugarcane bagasse, and OPEFB, respectively. These results were in line with Lazzari *et al.,* who found that the standard strain of the O-H functional group occurs in the wave number range of 3000-3600 cm^{-1}, which indicates the presence of cellulose [23].
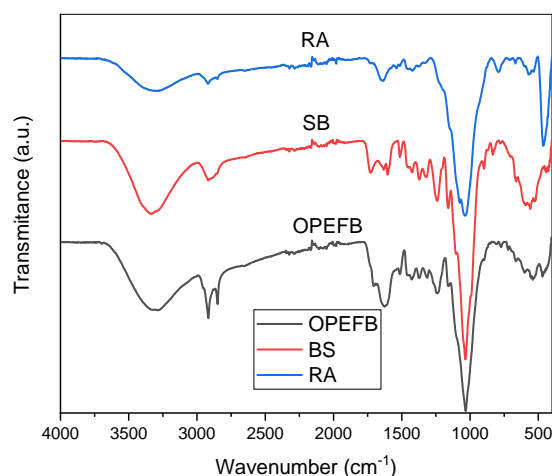

Fig. 2. FTIR spectra of biomass samples

3.2 XRD (X-Ray Diffraction)

Crystalline materials having a specific level of crystallinity are frequently characterized via XRD analysis. The degree of cellulose's crystallinity must be taken into account when converting biomass [24].

In the biomass conversion process into bioenergy, the crystalline nature of cellulose inhibits the performance of enzymes to convert cellulose into sugar which will be fermented into ethanol. Therefore, the lower the crystallinity of biomass, the higher the ethanol produced [25]. The results

of the XRD analysis, as shown in Figure 3, indicate the level of crystallinity and various typical peaks with different angles on various types of biomass. The level of crystallinity in OPEFB, rice husk, and sugarcane bagasse biomass yielded a value of 9.1127%, 8.0786%, and 25.7813%, respectively. The highest crystallinity level was indicated by sugarcane bagasse biomass, while the lowest was shown by rice husk biomass. Next, in the type of OPEFB, rice husk, and sugarcane biomass, the peak that appears at 2θ was at an angle of 21.718°, 22.2094°, and 44.1985°, respectively.
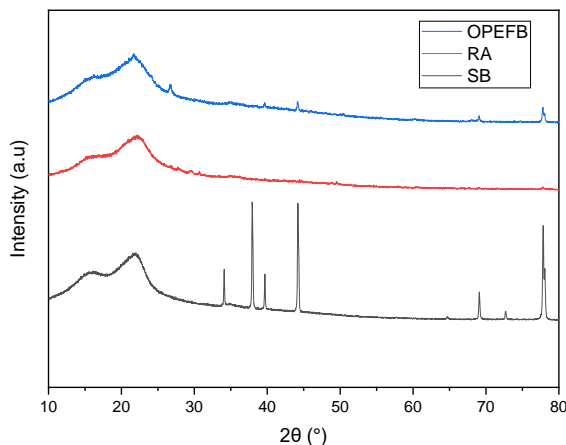

Fig. 3. XRD spectra of biomass sample

3.3 Py-GCMS

Biomass is an attractive material for pyrolysis due to the complex compounds in the bio-oil produced, especially phenols, ethers, esters, hydrocarbons, acids, furans, alkanes, ketones, etc. The results of the biomass analysis using Py-GCMS showed the compounds contained in the biomass, which were further categorized according to functional groups, as shown in Figure 4.
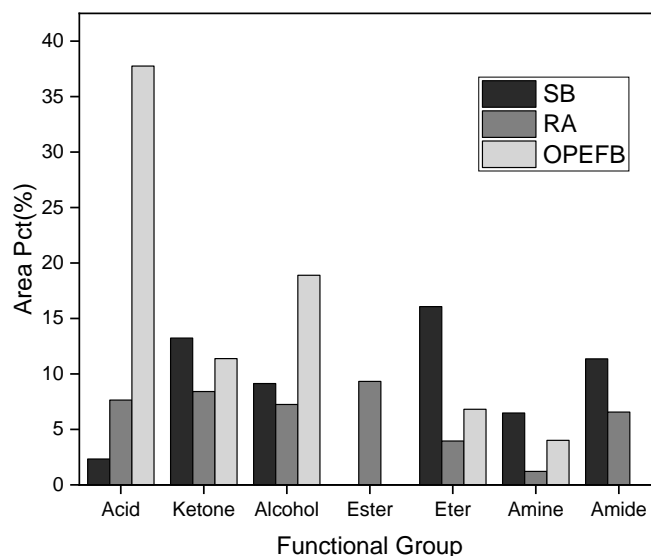

Fig. 4. Percentage of biomass functional cluster area

In Figure 4, it can be seen that the dominant functional groups of the three biomass are different. In sugarcane bagasse biomass, the ether functional group dominated with an area of 16.07%. In the biomass pyrolysis process, the ether functional group has a role in forming CO gas, which will then be condensed [26]. Next, there was the ester functional group that dominated the rice husk biomass

type with a percent area of 9.33%, but this functional group was not contained in other types of biomass. The ester functional group was neutral, and its content in bio-oil was useful for reducing the polarity level so that the bio-oil produced was more in line with liquid fuel standards. Meanwhile, in the OPEFB biomass type group, the most dominating functional group was acid, with a percentage of the area of 37.75%. A high acid/acid group will impact the highly corrosive nature of the bio-oil, so it is necessary to carry out a different process, i.e., esterification, to convert the acid group into an ester compound.

3.4 Biomass Multi Feedstock Ratio

When producing multi-feedstock biomass-based bio-oil, a model is needed to determine the amount of biomass type composition to produce bio-oil yield with the best ratio based on multi-feedstock biomass, which can be made in a simulation using Design Expert.

The simulation results of the multi-feedstock biomass with composition, can be seen in Figure 5.
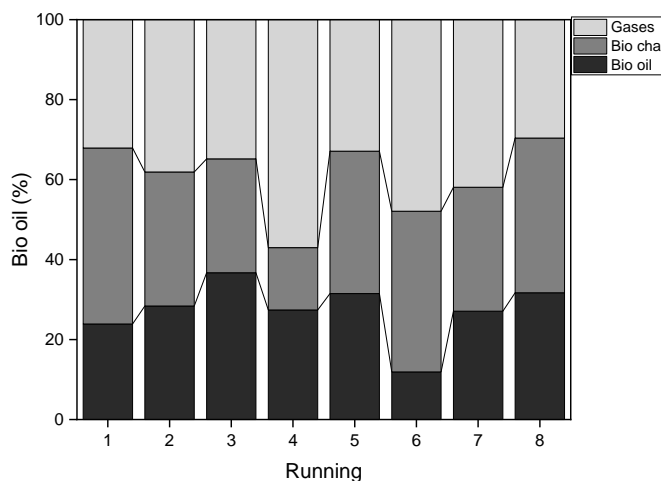

Fig. 5. Yield of multi-feed stock fast pyrolysis products

The model graph for the yield of bio-oil and biochar as a function of different biomass waste feedstock can be seen in Figure 6(a) and (b).
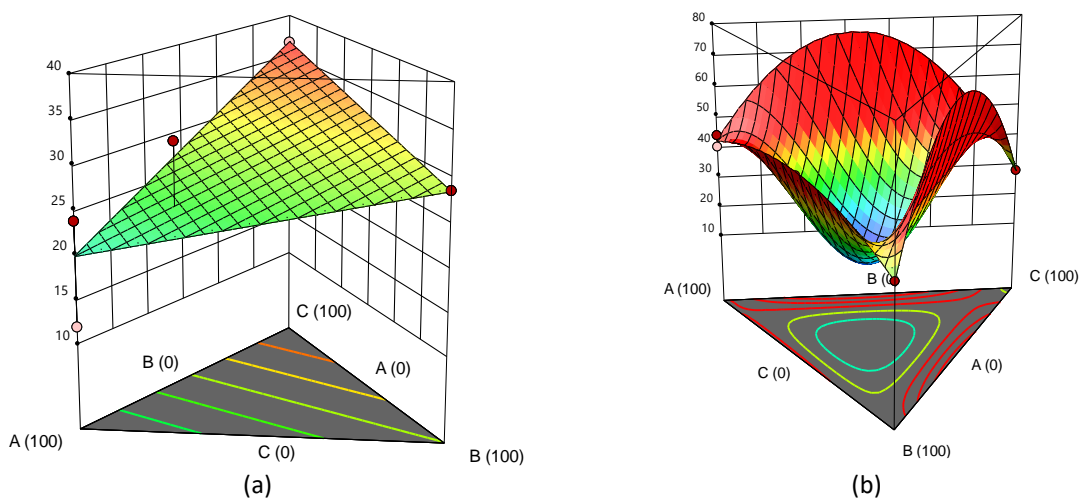

(a)

(b)

Fig. 6. Model graph for the yield of (a) bio-oil and (b) biochar, where (A) rice husk, (B) OPEFB, and (C) sugarcane bagasse

From the ANOVA analysis, the final equation in terms of actual components for the yield of bio-oil and bio char can be seen in Eq. (2) and Eq. (3).

$$Y_{bio-oil}=0.199989*SP+0.283180*TK+0.367980*BT \tag{2}$$

$$Y_{biochar} = 0.421*SP + 0.335*TK + 0.285*BT + 0.0039*SP*TK + 0.1614*SP*BT + 0.01578*TK*BT - 0.00159*SP*TK*BT \tag{3}$$

where SP is the composition of rice husk ash, BT is the composition of sugarcane bagasse, and TK is the oil palm empty fruit bunch composition. The parity plot of these equations shows high level of agreement between experimental data and the model equation [27].

Figure 5 and 6 demonstrate that the highest bio-oil yield value appears at the single composition of sugarcane bagasse with a bio-oil yield of 36.7%, so the optimization of multi-feedstock biomass on bio-oil yield cannot be carried out. Furthermore, by adding the lowest bio char yield requirements, simulations were carried out using Design Expert and showed the best bio-oil yields would be achieved with the composition of rice husks 30,5383%, OPEFB 36,6376%, and sugarcane bagasse 32.8241% as much as 15.6%. However, based on the experiments conducted, the yield of bio-yield bio char produced was as much as 20% (higher than the simulation results).

3.5 Characteristics of Bio-oil from The Multi-Feedstock Biomass Waste Pyrolysis
3.5.1 Acid number analysis

The acid number portrays the free fatty acids contained in a fat or oil [28]. This acid value can be defined as the basic requirement of KOH to neutralize free fatty acids in 1 g of oil. This study's determination of the number of bio-oil acids using the acid-base titration method was based on the analysis carried out; the number of bio-oil acids is 112.2-140.3 mg KOH g-1. This high acid number is a result of lignin and hemicellulose degradation in biomass which produces phenolic compounds or carboxylic acids such as Lauric acid, palmitic acid, and acetic acid [29]. In addition, it can also be caused by the presence of extractive acids [7]. Based on SNI7182:2015 for bio-diesel quality as a biofuel, the maximum allowable acid number is 0.5 mg KOH g-1. The acid number produced by bio-oil is still far from the quality standard of bio-diesel. As it has a relatively high acidity value, the focus of using bio-oil is as a hydrocarbon fuel. Bio-oil is not recommended for engine fuel since it is corrosive and can cause engine rust. For this reason, in industries such as boilers, combustion engines, gas turbines, and stationary diesel engines, bio-oil upgrading is needed to switch to using bio-oil as fuel.

3.5.2 Analysis of chemical components of bio-oil with GC-MS

Bio-oil has a complex component consisting of various types of chemical compounds. The distribution of chemical elements in bio-oil was identified using an analytical instrument called GC-MS. This identification determines the bio-oil characteristics from the pyrolysis process of biomass waste based on multi-feedstock. GC-MS analysis was performed according to the feedstock biomass ratio.

In the experimental results of pyrolysis of bio-oil from various biomass wastes of rice husk, sugarcane bagasse, and OPEFB, which took place at a temperature of 500°C, 20 components could be identified from the number of peaks formed, namely 20 peaks. The spectral peaks on the chromatogram indicate the types of compounds and chemical elements contained in the bio-oil

resulting from the pyrolysis experiment. The chemical compounds in bio-oil include alcohol, phenols, alkanes, ketones, aldehydes, carboxylic acids, and other aromatic compounds.

The GC-MS results, as shown in Figure 7, presents a chromatogram of the pyrolysis of bio-oil from OPEFB, sugarcane bagasse, rice husk, and a mixture of the three biomass which are rice husk, OPEFB and sugarcane bagasse (MF). It is known that more than 20 chemical components were dominated by phenolic groups, esters, and aldehydes. The results of GC-MS chromatography showed that the most dominant compound composition was the ester compound, with 48.96% contained in rice husk bio-oil. High ester content in bio-oil is preferred because it indicates better stability in bio-oil [30]. Esters in pyrolysis bio-oil products increased compared to esters in rice husk biomass which only amounted to 9.33%. In addition, it can also be seen that the acid compounds were decreasing, which indicates that the pyrolysis process is efficient in converting acidic compounds into esters contained in bio-oil.
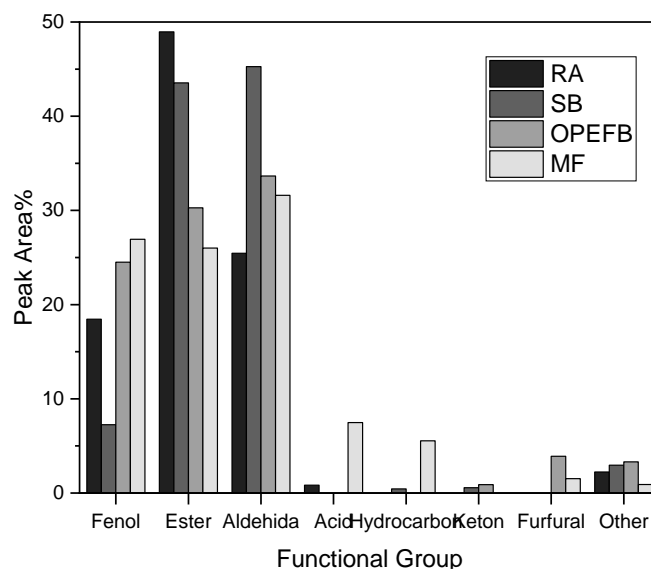

Fig. 7. GC-MS analysis of the bio-oil product

There were also aldehyde compounds that dominated the pyrolysis bio-oil. Aldehydes result from dehydrogenation of alcohol which has a characteristic odour. Figure 7 shows that all bio-oils with different types of biomass contained about 25-45% aldehydes, and the highest content of OPEFB biomass was 45.26%. However, the high aldehyde content makes it easier for bio-oil to hydrate. It indicates that the water content in bio-oil was still high, making it arduous to separate.

Phenol or phenyl alcohol (C_6H_5OH) is a colourless, crystalline solid easily soluble in water. Phenol has acidic properties, which means it can release H+ ions from its hydroxyl group. Compared to other aliphatic alcohols, phenol is more acidic. Phenolic compounds in OPEFB bio-oil and rice husk bio-oil were 7.25% and 18.46%, respectively. In contrast, phenolic compounds in sugarcane bagasse bio-oil were higher, i.e., 24.51%. The highest phenol was in bio-oil based on multi biomass feedstock (rice husk, OPEFB, sugar cane bagasse) of 26.94%. In general, the content of phenolic compounds in the pyrolysis liquid is affected by the temperature at the pyrolysis and the lignin contained in biomass. Phenolic compounds have stable properties and are difficult to separate, so they will only decompose at high temperatures, such as pyrolysis at 300-500°C. High levels of phenol in bio-oil will affect the high viscosity and acid number, which will cause corrosion, decrease the degree of acidity (pH), and reduce the calorific value of bio-oil [8].

There is also furfuryl alcohol ($C_5H_6O_2$), which is a furfural derivative that is a flammable material and is used as rocket fuel. Besides furfural, another furan class compound commonly found in bio-oil is benzo furan. In this study, benzo furan was found in sugarcane bagasse bio-oil and multi-feedstock,

with a percentage of 1.47% and 1.53%, respectively (Figure 7). Furan compounds in bio-oil will decrease when a fast pyrolysis reaction occurs at 500°C and increase phenolic compounds. At high temperatures, furan and phenol derivatives will be depolymerized to form large molecular components and solid products, increasing the yield of solid residues [31].

4. Conclusions

A mixture of OPEFB biomass waste, sugarcane bagasse, and rice husks has the potential to be utilized as raw material for bio-oil production using the fast pyrolysis process for 60 minutes at a temperature of 500°C because the lignocellulosic content in the biomass is quite large. The modeling results reveal that the bio-oil yield will reach the highest value at the composition of rice husks at 30.5383%, EFB at 36.6376%, and sugarcane bagasse at 32.8241% but still contains 20% bio char yield. In addition, the results of the bio-oil acid number obtained are 140.3 mg KOH g^{-1}. The results of chemical characterization showed that the multi-feedstock bio-oil compound was dominated by aldehydes, esters, and phenolic, where these compounds were the main components of petroleum fuels.

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