GENERATION AND CHARACTERIZATION OF BIO-OIL OBTAINED FROM THE SLOW PYROLYSIS OF OIL PALM EMPTY FRUIT BUNCHES AT VARIOUS TEMPERATURES

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Abstract: In the last decade, biomass pyrolysis has received more attention in the renewable energy sector. This technology converts biomass to obtain valuable products: bio-oil, biochar, and gas. Bio-oil is a liquid product from pyrolysis that can be used to fuel boilers and furnaces, or further processed to produce fuel oil and chemical products. In this study, bio-oil was generated from slow pyrolysis of oil palm empty fruit bunches (OPEFB) at various temperatures. The objective of this research is to investigate the effect of temperature on the properties of products generated from the pyrolysis of OPEFB. Six different pyrolysis temperatures ranging from 300 to 700℃ were used to produce bio-oil. It was found that operating temperature affected the product yield and its properties significantly. The higher the operating temperature of slow pyrolysis, the amount of bio-oil produced was also increased with a decrease in biochar yield. The highest yield of bio-oil was found to be 55.53% at a pyrolysis temperature of 700℃ with a yield of biochar and syngas was 24.22% and 20.25%, respectively. The GC-MS analysis was used as a quantitative means to characterize the liquid pyrolysis product. The findings of GC-MS showed that bio-oil generated in this study was dominated by phenols and ketones. In conclusion, pyrolysis of OPEFB demonstrates significant potential for industrial applications to generate valuable products especially bio-oil, providing a renewable alternative to fossil fuels.

Keywords: biomass; bio-oil; GC-MS; oil palm empty fruit bunch; pyrolysis

Abstrak: Dalam satu dekade terakhir, pirolisis biomassa semakin mendapat perhatian di sektor energi terbarukan. Teknologi ini mengubah biomassa menjadi produk-produk yang bernilai seperti: *bio-oil*, *biochar*, dan gas. Bio-oil adalah produk cair dari pirolisis yang dapat digunakan sebagai bahan bakar *boiler* dan *furnace*, atau diproses lebih lanjut untuk menghasilkan bahan bakar minyak dan produk kimia. Dalam penelitian ini, *bio-oil* dihasilkan dari proses pirolisis lambat tandan kosong kelapa sawit (TKKS) pada berbagai kondisi suhu. Tujuan dari penelitian ini adalah untuk mengetahui pengaruh suhu terhadap karakteristik produk yang dihasilkan dari pirolisis TKKS. Berbagai variasi suhu pirolisis dari rentang 300 hingga 700℃ digunakan untuk menghasilkan *bio-oil*. Hasil penelitian menunjukkan bahwa suhu pirolisis mempengaruhi *yield* produk dan karakteristiknya secara signifikan. Semakin tinggi suhu operasi pirolisis, jumlah *bio-oil* yang dihasilkan juga semakin meningkat, namun diikuti dengan penurunan *yield biochar*. *Yield* tertinggi *bio-oil* yaitu sebesar 55,53% diperoleh pada suhu 700℃ diikuti dengan *yield biochar* dan *syngas* masing-masing sebesar 24,22% and 20,25%. Analisis GC-MS

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digunakan sebagai metode kuantitatif untuk mengkarakterisasi produk cair dari pirolisis. Hasil GC-MS menunjukkan bahwa komposisi *bio-oil* yang dihasilkan dalam penelitian ini didominasi oleh senyawa fenol dan keton. Penelitian ini menjanjikan potensi dari pirolisis TKKS untuk aplikasi industri guna menghasilkan produk bernilai tinggi berupa *bio-oil* sebagai penyedia sumber energi terbarukan pengganti bahan bakar fosil.

Kata Kunci: biomassa; bio-oil; GC-MS; pirolisis; tandan kosong kelapa sawit

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Introduction

Oil palm empty fruit bunches (OPEFB) are solid waste produced by palm oil processing industries. In palm oil mills, most OPEFB is only burned or dumped in the mill area due to its high production rate (Shariff, 2014). In Indonesia, palm oil is popular as one of Indonesia's largest agricultural export commodities. During 2021, its palm oil production amounted to around 46.2 million metric tons (Statista, 2023). However, OPEFB causes environmental pollution and problems because it can reduce the ability of the soil to absorb water. In addition, OPEFB that rots in place will attract the arrival of certain types of beetles, which can potentially damage rejuvenated oil palm trees in the land around the disposal site (Puasa et al., 2022; Supriatna et al., 2022). Thus, using OPEFB as biomass feedstock can be one of the solutions to reduce this solid waste and convert OPEFB into useful and value-added products.

In a palm oil processing factory, OPEFB is a collection of thick brown fibers left behind after the fruit is boiled. OPEFB fibers have a hard and robust characteristics. The pores on the surface of the OPEFB fibers have an average diameter of 0.07 m (Hanan et al., 2018). This pore surface morphology is very useful for increasing the mechanical bond with the matrix resin when used to manufacture composites. Prior studies reported that microfibrils, cellulose, lignin, hemicellulose, and holocellulose are the main and most abundant compounds in OPEFB (Chang, 2014; Omar et al., 2011; Kerdsuwan and Laohalidano, 2011). Holocellulose and hemicellulose have the same chemical structure as cellulose, but their properties are similar to lignin. The composition of OPEFB is shown in Table 1.

Table 1. Composition of oil palm empty fruit bunches (Chang, 2014; Omar et al., 2011; Kerdsuwan and Laohalidano, 2011)

Components	Units	Values
Cellulose	%	23.70 – 65.00
Lignin	%	14.10 – 30.45
Hemicellulose	%	20.58 – 33.52

Components	Units	Values
Holocellulose	%	68.30 – 86.30
Ash content	%	1.30 – 13.65
Moisture content	%	2.40 – 14.28
Volatile matter	%	70.03 – 83.86
Carbon	%	43.80 – 54.76
Hydrogen	%	4.37 – 7.42
Oxygen	%	38.29 – 47.76
Nitrogen	%	0.25 – 1.21
Sulfur	%	0.035 – 1.10

The energy crisis in the last decade demands the search for alternative energy sources to fulfill world energy needs. Biomass has been identified and developed as an eco-friendly and ecologically friendly energy source (Ighalo and Adeniyi, 2019; Wang et al., 2017). Eighty-six percent of Indonesia's energy comes from fossil fuels (Larasati, 2023). To alter this structure, Indonesia's rising energy demand must also be taken into account. Indonesia must then make an effort to use renewable energy to meet this growing energy demand. By looking at these opportunities, OPEFB can be developed into fuel through thermo-chemical conversion. These mainly include biomass gasification, torrefaction, and pyrolysis (Sarkar and Wang, 2020). Compared to two other methods, pyrolysis stands out in several key aspects, such as its simplicity and versatility in handling various types of feedstock. Additionally, the high heating value and mass energy density of the products obtained from pyrolysis makes it a promising technology for energy production. The efficient conversion of biomass into energy-rich products like bio-oil and syngas highlights the potential of pyrolysis as a sustainable energy conversion technology (Synder, 2019; Yang and Chen, 2021).

Pyrolysis is a thermal decomposition process of biomass at high temperatures without oxygen. The decomposition process in pyrolysis is also often referred to as devolatilization. There are two broad categories of pyrolysis techniques: slow and fast pyrolysis. Slow pyrolysis consists of slow heating rates of 0.1–1°C/s and a temperature ranging of 400–600°C. In comparison, fast pyrolysis is achieved through rapid heating rates of 10 to >1000°C/s with temperatures ranging from 400–650°C and rapid quenching of the vapors during the process (Dickerson and Soria, 2013; Balat et al., 2009; Handoko et al., 2021). The main products from pyrolysis of biomass are solid (charcoal), liquid (bio-oil), and gas. Charcoal, also known as biochar, can be utilized as an adsorbent (activated carbon) or fuel because it has a high heating value equivalent to coal. The bio-oil produced from the pyrolysis process can be used as an alternative fuel or an additive in fuel after upgrading. Meanwhile, the gas formed can be burned directly (Park et al., 2008; Qu et al., 2011; Tripathi et al., 2016).

Bio-oil is a pyrolysis product that can be used in several different ways. Bio-oil can be processed into fuel oil and chemical products or used directly as fuel for boilers and furnaces (Bridgwater, 2003). Organic compounds like aromatic hydrocarbons, phenol, ketones, esters, esters, sugars, amines, alcohols, furan, and water have been identified as components in bio-oil (Kumagai et al., 2015). The water content in bio-oil is moderately high, generally in the range of 20-25% (Gai et al., 2013). The objective of this study is to perform slow pyrolysis of OPEFB and study the potential of converting OPEFB to biofuels. The effect of temperature on the yields and composition of bio-oil produced by this process is also investigated and identified.

Methods

Materials

This study used oil palm empty fruit bunches (OPEFB) as raw biomass materials for pyrolysis. The empty fruit bunches were obtained from PT Perkebunan Nusantara V, Riau, Indonesia. OPEFB was first washed to remove the remaining dirt and then chopped into smaller sizes (1-2 mm). The water content of OPEFB was removed by drying in the oven at 105℃ for 2 hours (Aprianti et al., 2020).

Methods

The experiment was conducted using a cylindrical fixed-bed reactor made from stainless steel, with dimensions of 400 mm inner diameter, 44 mm outer diameter, and 600 mm height. The reactor is equipped with an external coil from nickel for heating and connected to a thermocouple to detect temperature and heating speed during the reaction (Jamilatun et al., 2022). The schematic diagram of the pyrolysis equipment is illustrated in Figure 1.

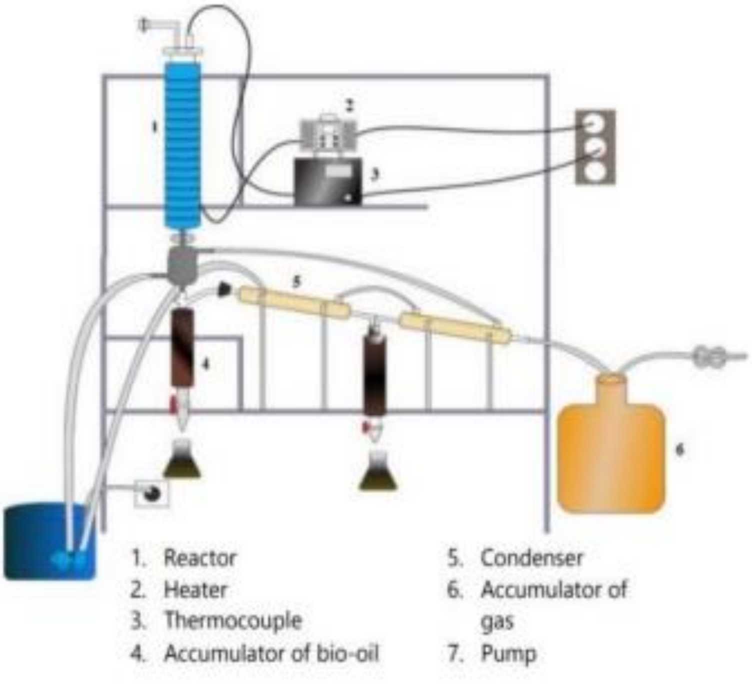

Figure 1. Schematic diagram of pyrolysis equipment (Jamilatun et al., 2022)

Pyrolysis was started by adding 15 grams of OPEFB into the reactor. Heating was carried out with an electric current through a nickel wire wrapped around the outside of the reactor with an average speed of between 5-35°C/minute. Pyrolysis was carried out at six variations of temperatures: 300, 400, 500, 500, 550, 600, and 700°C and monitored with a thermocouple. The condensed gas was collected in a bio-oil accumulator and weighed. The gas that did not condense was flowed into the water tank to be absorbed, while the biochar formed in the reactor was taken after the pyrolysis was finished and then weighed. Pyrolysis was stopped after reaching the desired temperature and allowed to stand at a constant temperature for 30 minutes. The characterization of the liquid product obtained from the slow pyrolysis of OPEFB (bio-oil) was quantitatively and qualitatively analyzed by Gas Chromatography-Mass Spectrometry (GC-MS) Shimadzu QP2010.

Results and Discussion

Effect of Temperature on Product Yield

Slow pyrolysis of OPEFB generated three products: bio-oil, biochar, and syngas. Figure 2 shows the effect of temperature on the yield of each product from OPEFB.

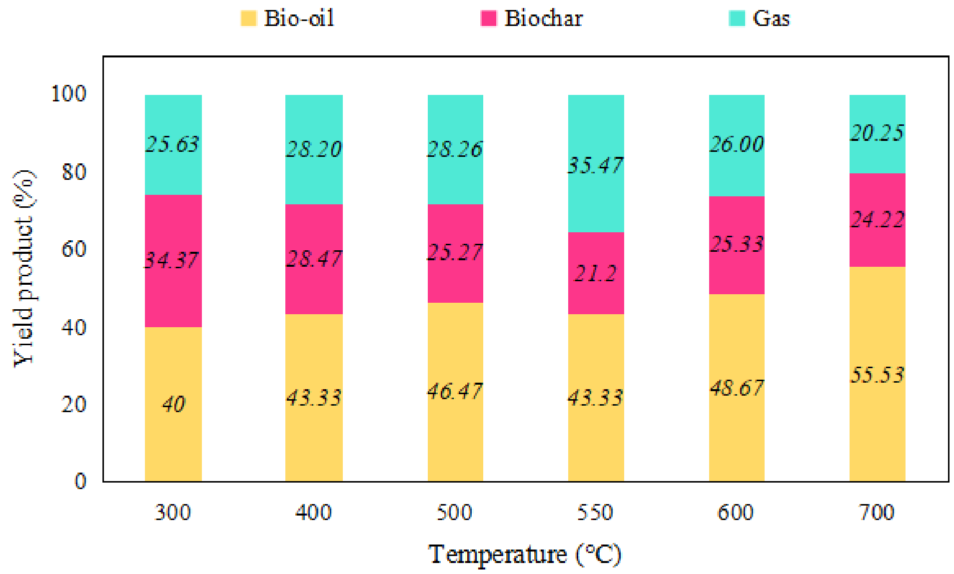

Figure 2. Effect of temperature on the yield of pyrolysis product derived from OPEFB

It can be seen from Figure 2 that generally, the yield of bio-oil increases with an increase in temperature from 300 to 700°C, while the maximum yield of bio-oil occurred at 700°C, which was 55.53 wt.%. This experiment found that bio-oil yield increased from 40 wt.% to 46.67 wt.% when the temperature ranged from 300°C to 500°C. However, at 550°C, the bio-oil yield slightly decreased to 43.33 wt.% and then increased again at 600°C and 700°C. Based on this result, 700°C was the optimum temperature for producing bio-oil from OPEFB by slow pyrolysis without a catalyst.

The decrease in bio-oil yield is affected by secondary cracking in tar (both bio-oil and water phases). Secondary cracking in pyrolysis plays a crucial role in the formation of various products. As the primary pyrolytic products are generated, secondary cracking reactions occur (mostly at above 600°C), leading to the production of lighter compounds. In the pyrolysis process, a cracking reaction occurs, namely breaking the C—C bonds from long carbon chains (polymers) and massive molecular weights to short carbon chains (monomers) with small molecular weights (Zhang et al., 2016). The cracking reaction can affect the increase in pyrolysis temperature; the more bonds (hydrocarbon chains) are broken, the more yield increases. High temperature also affects the reduction of liquid product and is consistent with its top gas product. A secondary cracking occurs, breaking long chains of organic compounds and hydrocarbons into shorter chains so they are difficult to condense again (Jamilatun et al., 2020; Jamilatun et al., 2019).

Figure 2 also shows the effect of temperature on the yield of biochar (solid product or charcoal) from pyrolysis of OPEFB. The results indicate that the yield of charcoal fluctuated. The highest yield of biochar was obtained from the lowest temperature (300ºC), generating 34.37 wt.% solid product. Meanwhile, pyrolysis at 550ºC yielded the lowest biochar (21.2 wt.%). Pyrolysis at low temperatures of less than 400°C or relatively low heating has generated relatively high charcoal products (Dickerson and Soria, 2013). The majority of the biochar is made up of carbon and partially pyrolyzed materials like high-molecular-weight hydrocarbons. Lower heating rates and longer residence times cause secondary cracking reactions, affecting bio-oil properties (Hasan et al., 2020). The higher the pyrolysis temperature, the lower the charcoal content because the constituents of the OPEFB will decompose, and the volatile matter content will decrease as the pyrolysis temperature increases.

Characterization of Bio-Oil

The chemical composition of bio-oil produced at two different temperatures (300°C and 600°C) was studied using the GC-MS study. This identification was carried out to determine the distribution of compound components in the liquid pyrolysis product. The spectrum results are shown in Figure 3 (300°C) and Figure 4 (600°C), respectively.

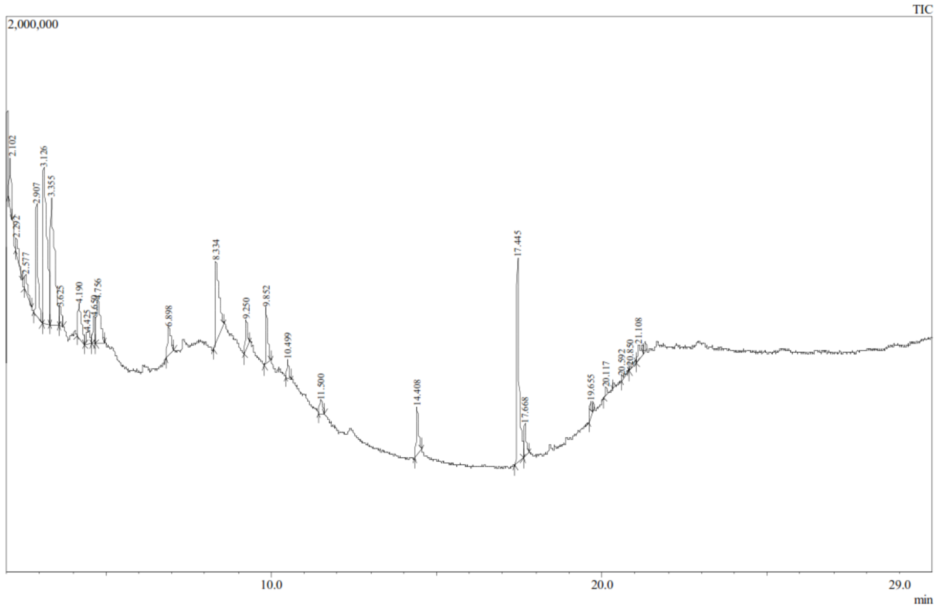

Figure 3. GC-MS result from the identification of bio-oil compounds at a temperature of 300℃

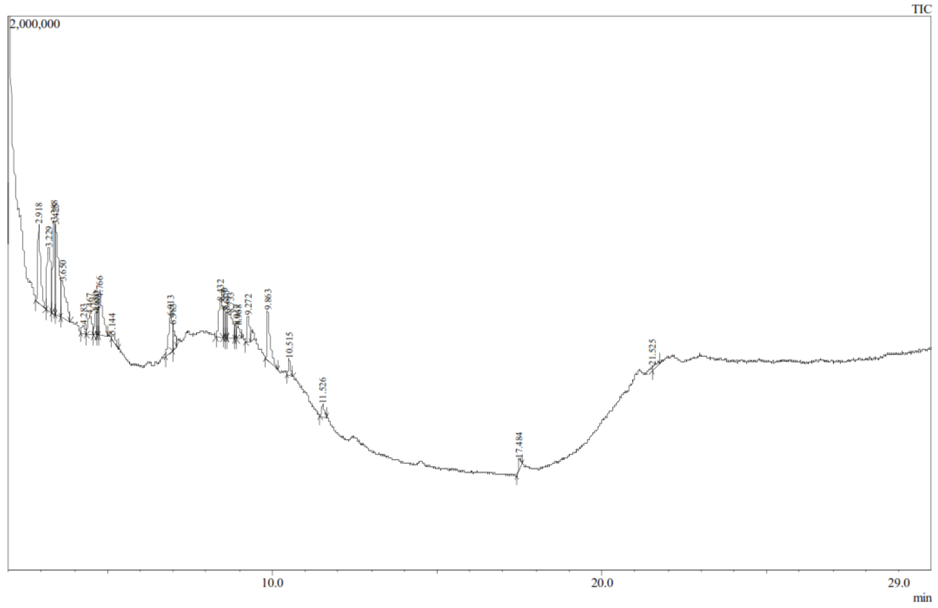

Figure 4. GC-MS result from the identification of bio-oil compounds at a temperature of 600℃

The GC-MS study identified numerous oxygenated compounds like ketones, alcohols, aromatics, organic acids, phenols, and hydrocarbons. GC-MS results also indicated that the pyrolysis temperature significantly affects the composition and amount of bio-oil obtained from OPEFB. In this study, higher bio-oil percentages were obtained by elevating the temperatures. The bio-oil yield increased from 40.00% at 300°C to 48.67% at 600°C due to the decomposition of lignin and cellulose in OPEFB. This result is by previous studies reported increasing temperature contributes to maximizing bio-oil production, as rapid cooling of volatiles and specific particles occurred at higher temperatures (Maulinda, 2023; Aboulkas et al., 2017).

To indicate their relative area percentages, the described compounds from GC-MS results have been grouped into each functional group. Figure 5 compares the percentage of main chemical compounds in bio-oil at temperatures 300°C and 600°C. Results show that the main components of bio-oil consist of phenol, ketone, and organic acid as they are compounds with the largest composition. This analysis indicated that slow pyrolysis of OPEFB at a lower temperature (300°C) mainly generated phenolic compounds as the key components of bio-oil produced. However, the concentration of phenol decreased with an increase in pyrolysis temperature to 600°C, followed by a higher fraction of alkanes in bio-oil produced. During the initial stages of pyrolysis, low-molecular-weight species are distilled.

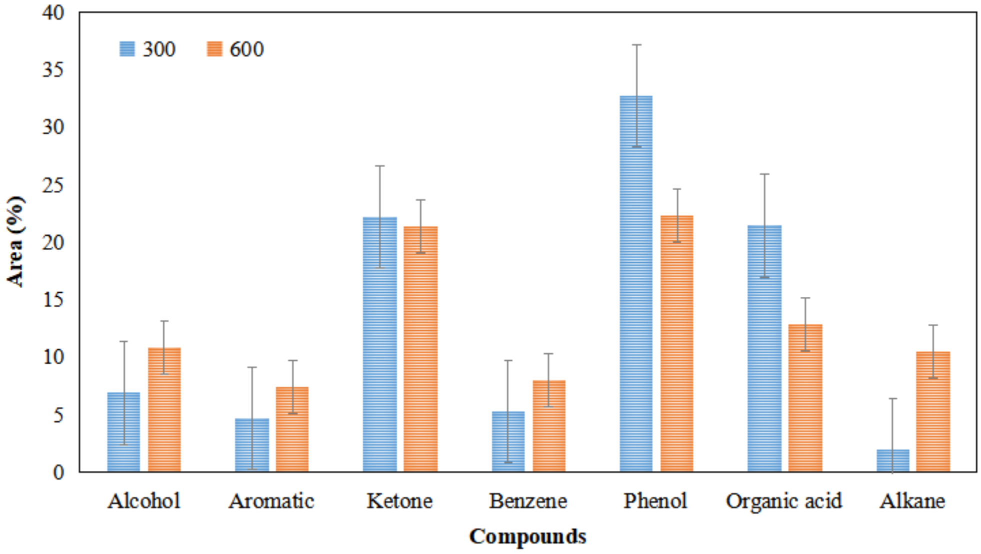

Figure 5. Major functional groups present in bio-oil from OPEFB at temperatures 300°C and 600°C

Nevertheless, in addition to the increased rate of volatilization caused by the gradual evaporation of larger molecules, the cracking of compounds may also occur as the temperature rises, producing volatile fragments (Al-Harahsheh et al., 2011). Due to an increase in temperature during the lignin and cellulose degradation process at the early stage, the degradation of other compounds decreased. This leads to a decrease in phenol concentration (Adinda et al., 2023). GC-MS results in this study present the existence of some oxygenated compounds that could influence the properties of bio-oil. In order to eliminate these undesirable compounds and make bio-oil usable as a substitute for fossil fuels, it is necessary to upgrade the bio-oil product due to the presence of oxygenated and nitrogen compounds.

Conclusions

Bio-oil from OPEFB was generated in a fixed bed reactor under slow pyrolysis conditions between 300°C to 700°C temperature range. The liquid, solid, and gas products obtained from this experiment were observed, and the liquid product (bio-oil) was characterized using GC-MS analysis. It was found that operating temperature affected the product yield and its properties. The higher the operating temperature of slow pyrolysis, the more bio-oil produced, while the yield of biochar decreased. The highest bio-oil yield was obtained at 700°C, where bio-oil yield was 55.53%, biochar yield was 24.22%, and gas yield was 20.25%. Thus, a temperature of 700°C was the optimum condition for conducting slow pyrolysis of OPEFB without a catalyst. From GC-MS analysis, phenol was the dominant compound in bio-oil.

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