

Fast pyrolysis of oil palm empty fruit bunch in an auger reactor: bio-oil composition and characteristics

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Abstract. Fast pyrolysis of oil palm empty fruit bunch (EFB) has been conducted in an auger reactor to produce bio-oil. The reactor was a continuous feeding auger reactor heated by gas-fired external heater. The experiments were conducted under different operating temperatures ranging from 400 °C to 650 °C, different nitrogen flow rates ranging from 1 to 4 L/min, and various feeding rates ranging from 2 to 8 kg/h. The maximum yield of bio-oil from EFB was 58.67 % obtained at temperature of 500 °C with feeding rate of 8 kg/h under 1 L/min of nitrogen gas flow rate. The average moisture content of bio-oil was varied between 30 and 40 % and the bio-oil produced at 500 °C provided the maximum HHV of 27.38 MJ/kg, respectively. The Fourier-transform infrared spectroscopy (FT-IR) and gas chromatography–mass spectrometry (GC-MS) spectra shows bio-oil contains high phenolic compounds. This paper explains the fast pyrolysis of empty fruit bunch using auger reactor to enhance the bio-oil yield and characteristics.

1. Introduction

Nowadays, the demands for energy has been increasing since it become the essence of modern civilization and economic advancement. Hence, the development of renewable energy has become vital in order to cater the demands and also mitigate the dependency of fossil fuel as it is not renewable and sustainable. Besides, the utilization of this source of energy increases the greenhouse gas (GHG) emission, which ultimately causes the climate change in the world. Hence, a comprehensive research has been continuously conducted on renewable energy sources, especially on biomass energy to develop an advanced technology, which can convert biomass to useful form of energy either gas or liquid [1, 2]. The fast pyrolysis process is such a technology since it can produce flammable liquid which is known as bio-oil that has the potential to reduce the dependency of petroleum fuels [3-5].

Biomass is one of the renewable energy sources that has huge potential to be utilize since it is abundantly available and cheap while being sustainable and environmentally friendly. In Malaysia, around 420 oil palm mills process the fresh fruit bunch (FFB) to produce crude palm oil, which generates huge amount of oil palm waste including empty fruit bunch (EFB), fibers, and shells [6]. Among the wastes, the EFB has been underutilized and been used as mulching



material and is sometimes fed into a boiler for low energy-efficiency combustion fuel [7]. Unfortunately, most of the EFB is not utilized and is left decomposing near the mill due to its high moisture content [8]. In addition, due to a paucity of studies on the fast pyrolysis of EFB, the commercial uptake of it for bio-oil production is still uncertain. Development of a suitable technology for EFB conversion to bio-oil with adequate quality can provide a solution for the burden of storing and disposing of waste EFB for mill owners as well as creating a new business opportunity. Due to the availability and high carbon content of EFB, their conversion into liquid fuel is promising [2]. After the appropriate upgrading, the liquid fuel can be utilized for transportation and industrial heating.

Fast pyrolysis is a thermal degradation process of biomass in the absence of air which involves very short residence times. The process occurs at temperatures around 400 °C to 700 °C and produces a burnable liquid known as bio-oil as the main products, alongside with solid char and gas as their by-products. This bio-oil has variety of applications since it contains high content of phenolic compounds including phenols, benzene, naphthalene, hydroxyl ketones, sugars, carboxylic acids and esters [9]. Since bio-oil is rich in many organic compounds, it can be utilized as combustion fuels and as a feedstock for chemical separation.

The fast pyrolysis of EFB has been investigated by many researchers, particularly with the fixed-bed reactors and fluidized bed [10-13]. Sulaiman et al. had investigated the fast pyrolysis of EFB in a bench scale fluidized bed reactor at temperature of 450 °C and yielded around 55 % bio-oil [12]. On the other hand, Sukiran et al. studied the fast pyrolysis of EFB in quartz fluidized bed reactor and yielded the maximum bio-oil of 42.28 % using fine particle size of 91-106 µm at temperature of 500 °C with 100 °C/min heating rate [14]. Most of the current bio-oil production systems utilize the fluidized bed reactor which is complex and expensive to build. In addition, fluidized bed reactor needs very fine particles of biomass which further adds the production cost. Moreover, high flow of fluidizing gas is needed which creates high speed of product stream, reducing the condensability of bio-oil vapor and in turns reducing the bio-oil yield. The auger reactor was selected for this process since it is can produced high yield of bio-oil (up to 75wt%) [15], easy to operate and scale up and can be used for larger particle size.

Many researchers have done improvement for the fast pyrolysis technology using different types of reactors and biomass including EFB, but the properties of the liquid bio-oil are yet not up to commercial standards. The study regarding fast pyrolysis of EFB in auger reactor has not been reported, hence, this work was carried out in a pilot-scale auger reactor utilizing EFB pellet as the raw material. The experiment was conducted at different operating temperature, nitrogen gas purging flow rate, and biomass feeding rate. The main objective of this work was to determine the optimum condition for fast pyrolysis of EFB in an auger reactor which maximizes the liquid bio-oil yield and quality.

2. Experimental

2.1. Feedstock

The EFB was collected in pellet form from local palm oil mill at Klang, Selangor and dried in oven for 2 hours at temperature of 110 °C before packed using storage container to prevent microorganism growth.

2.1.1. Proximate and ultimate analyses. The EFB pellet was ground and sieved into 1 – 2 mm particle size using a grinding machine in order to determine the proximate and ultimate analyses. The carbon, hydrogen, oxygen and nitrogen content of EFB was determine by an elemental analyzer (model Flash EA 1112 Series). The volatile and fixed carbon content was

determined by thermogravimetric analyzer (TGA) (Mettler Toledo) at rate of 10 °C/min from ambient temperature until 900 °C under nitrogen (N₂) flow rate of 200 mL/min using sample of 20 mg. The ash content of EFB was determine by adding 1 g of EFB into ceramic crucible and burnt isothermally overnight at a set temperature of 600 °C in the Muffle furnace (Thermolyne). The moisture content was determined by the mass different of EFB before and after dried in the oven at 110 °C overnight.

2.2. Pyrolysis procedure

The fast pyrolysis of EFB was done using an auger reactor under fast pyrolysis operating parameter. The fast pyrolysis system consists of a screw feeder, auger reactor, furnace, char collector and condenser as shown in figure 1. The biomass was continuously feed at controlled feed rate by regulating the rotation speed of the screw feeder using an inverter controller with a DC gear motor. The nitrogen flow was manipulated in the range of 1L/min to 4 L/min to ensure the pyrolysis product was flowing to the condenser to prevent the bridging of feedstock. A 2 kg of EFB was fed into the reactor hopper in the speed range of 2 kg/h to 8 kg/h and was gradually dropped at desired pyrolysis temperature of 450 °C to 650 °C. The temperature of the reactor was controlled by regulating the flow rate of liquified petroleum gas (LPG) which was supplied in the fire tubes heater outside of the auger reactor and monitor by using two K-type thermocouples. The heavy particle was separated using cyclone and the lighter vapor products was condensed in two condensers filled with dry ice and ice to ensure complete condensation of bio-oil. The bio-oil yield was obtained by mixing the liquid products in the containers. The remaining gas was burned in the fume cupboard. The reaction process was continuing for 1.5 h to ensure all biomass has been fully pyrolyze and no gases product detected in fume hood.

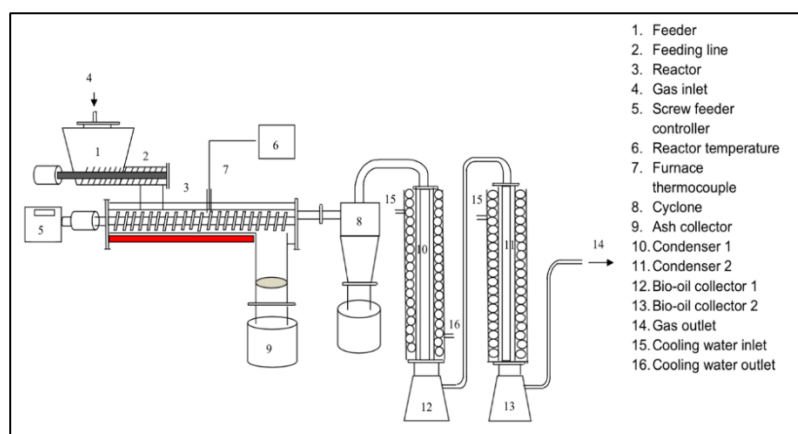


Figure 1. Schematic diagram of the pilot scale auger reactor.

2.3. Characterization of bio-oil

2.3.1. Physical characteristics. The HHV of bio-oil was determined by using an oxygen bomb calorimeter (Parr Oxygen Bomb Calorimeter, Model 6400), while the acidity and density was determined by using a digital pH meter (Model – HI 8424), and a pycnometer, respectively. The Karl Fischer titrimetric method (ASTM D-1744) was used to determine the moisture content in the bio-oil samples. The phase separation method was employed to determine the lignin content of the bio-oil as described in the literature [16].

2.3.2. *Chemical characterization of bio-oil.* The Perkin-Elmer Spectrum GX FT-IR spectrometer was used to identify the functional group of compounds present in the bio-oil and the spectra was recorded in the range of 4000 and 400 cm^{-1} . The identification of chemical compounds exist in the bio-oil was investigated using GC-MS-QP2010 by comparing the mass spectra with NIST08 library.

3. Results and discussion

3.1 Proximate and ultimate analyses of EFB

In bio-oil production from biomass, the chemical composition and physical properties of feedstock play a vital role in the yield and characteristics of bio-oil, and thus they were evaluated. The chemical composition and physical properties of EFB were summarized in Table 1. The EFB used was in pellet form ranging from 10 mm to 30 mm in size consists of shredded fiber material from oil palm mill. The bulk density of EFB pellet was 669 kg/m^3 , while the moisture content obtained was around 12 %. The proximate analysis of EFB reported a composition of 77 % volatile fraction, 18 % ash content and 5 % fixed carbon. The ultimate analysis provided the carbon, oxygen, and hydrogen content of 49.4 %, 5.1 %, and 45.5 %.

Table 1: Proximate and ultimate analysis of EFB feedstock*

Properties	EFB
Moisture content (%)	12
Pellet size (mm)	10-30
Proximate analysis (% , without moisture)	
Volatile fraction	77
Fixed carbon	18
Ash content	5
Ultimate analysis (%)	
C	49.4
H	5.1
O	45.5

* EFB was oven dried.

3.2. Fast pyrolysis of empty fruit bunch

3.2.1 *Effect of temperature.* The effect of temperature on the fast pyrolysis of EFB in the range of 400 to 650 $^{\circ}\text{C}$ is shown in figure 2. The maximum bio-oil yield obtained of 58.67 % was achieved at 500 $^{\circ}\text{C}$. The decomposition rate of solid is higher at higher temperature causing reduced char yield. However, the volatile component consisting of liquid and gas formation is competitive with each other as a function of temperature. The secondary decomposition of organic vapor is low at lower temperature, hence decreasing the yield of gas at temperatures below than 500 $^{\circ}\text{C}$ [13]. The gas yield increased with increasing temperature because temperatures higher than 500 $^{\circ}\text{C}$ favor the secondary decomposition of vapor.

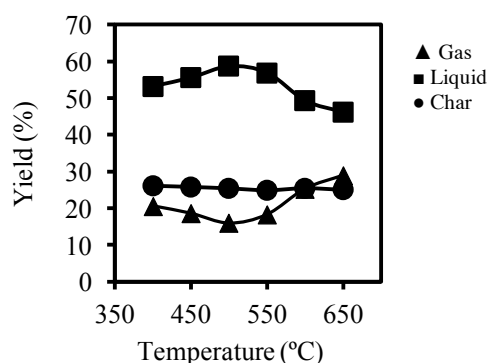


Figure 2. Effect of temperature over product yields

The data obtained in the fast pyrolysis of EFB using the auger reactor is comparable with our previous study using a bubbling fluidized bed reactor [5]. In this study, the repolymerization of devolatilized organic molecules and vapor-solid reaction occurred at lower temperature resulting higher solid yield [17]. The repolymerization of devolatilized organic molecules is referred to as the product obtained from breaking of the linkage between two aromatic rings at low temperature [18]. The continuous rotation of the screw in the auger reactor caused faster heat transfer between biomass particle and reactor hot surface. The heat input and horizontal arrangement of the auger reactor causes the temperature difference at the upper and lower part of the reactor. In addition, the gap between top and bottom surface of the screw in the auger reactor design channeled the vapors through the gap, preventing further contact with char. Hence, the secondary reaction between vapor and solid were reduced, leading to the high yield and low moisture content of the bio-oil.

3.2.2. Effect of nitrogen gas flow rate. The nitrogen gas acts as sweeping gas, pushing the pyrolysis products produced in the auger reactor to the condensation system. The effect of different nitrogen gas flow rate indicates the effect of residence time on the product yield. The nitrogen gas flow rate was varied from 1 L/min to 4 L/min to investigate the effect of nitrogen flow rate in fast pyrolysis of EFB as shown in Figure 3. Higher char and liquid yield are obtained with lower nitrogen flow rate since the longer vapor residence time in the condenser allows a more complete condensation process.

Higher nitrogen flow rate lead to higher gas yield and lower liquid yield and char yield. Liquid and char yield were lower due to slower polymerization reaction of the organic vapor since the vapor are diluted with the nitrogen gas. The gas yield was higher due to increased heat transfer between phases at higher gas flow rate, which enhanced intra-molecular decomposition of gas. In addition, the vapor residence time between molecules are shorter at higher nitrogen flow rate causing the vapor not fully condense in the condenser; thus, decreased the liquid yield [19]. Therefore, shorter vapor residence time is not favorable for condensation process, although the secondary decomposition of organic vapor can be reduced at higher nitrogen flow rate.

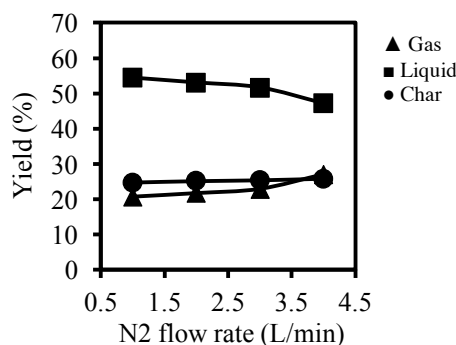


Figure 3. Effect of nitrogen flow rate over product yields

3.2.3. Effect of biomass feeding rate. The fast pyrolysis process of EFB in the auger reactor was investigated from the standpoint of the effect of biomass feeding rate from 2 kg/h to 8 kg/h at 500 °C with 1 L/min nitrogen flow rate. The result is shown in Figure 4 indicating that increasing biomass feeding rate lead to increasing bio-oil yield. The gas and char yield are higher while the liquid yield is lower at lower feeding rate, whereas the liquid yield is higher at higher feeding rate. The longer residence time of vapor in the reactor enhanced the volatile-char interaction under lower feeding rate, and thus leads to the coke/char formation reactions [12]. In addition, the longer vapor residence time also increases the gas yield at lower feeding rate because of the vapor products cracking to gas while the secondary reaction took place. On the contrary, at higher feeding rate, the increased amount of vapor produced leads to a shorter vapor residence time. The shorter vapor residence time leads to a higher bio-oil yield since there was less opportunity for the undesirable secondary cracking and repolymerization reactions. Unfortunately, higher biomass feeding rate caused incomplete pyrolysis due to inadequate heat transfer within the reaction, leading to increased char production.

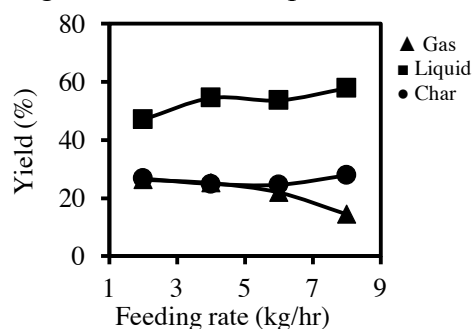


Figure 4. Effect of feeding rate over product yields

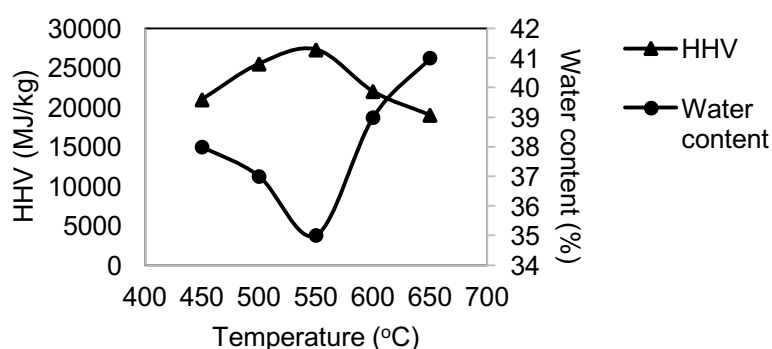
3.3. Bio-oil characterization

3.3.1. Physical characteristics. The physical properties of bio-oil at optimum condition obtained are summarized in Table 2. The density and viscosity of bio-oil are 1040 kg/m³ and 2.28 cP, respectively. The pH value obtained is 3.5, which indicates that the bio-oil is highly acidic due to the presence of heavy oxygenated organic compounds such as benzoic acids and phenol.

Table 2: Physical properties of bio-oil.

Properties	EFB
Density (g/mL)	1.04
Viscosity (cP)	2.28
pH	3.5
Lignin content (wt%)	7.09
Solid content (wt%)	3.20
Ash content (wt%)	1.17

The lignin content of this bio-oil is 7.09 %, which is higher than that of bio-oil produced from palm kernel shell (PKS) (2.25 %) [5] indicating higher water insoluble fraction in EFB bio-oil [5]. On the other hand, the solid and ash content of this bio-oil are 3.19 % and 1.16 % which are higher than in the bio-oil derived from PKS [5]. This may be due to the fact that EFB contains more ash (around 5 %) which is mostly made up of potassium which vaporizes at a comparatively lower temperature and would condense into the condenser together with the bio-oil. The average water content varied between 30 to 40 % depending on temperature. The higher water content is observed in bio-oil at lower temperature possibly due to water molecules released by condensation polymerization of bio-oil vapors. However, at higher temperature of 550 °C, the higher water content was observed may be due to the dehydration of bio-oil which released water molecules and other aromatic compounds. The highest HHV was found at 27.3 MJ/kg for bio-oil produced at 550 °C. The HHV obtained varies from 18.9 MJ/kg to 27.3 MJ/kg at different temperatures due to high moisture content and high oxygen content molecular compound produced at higher temperature as shown in Figure 5. The lower water content of bio-oil contributes to the higher value of HHV, which are 27 MJ/kg for auger reactor and 21-22 MJ/kg for fluidized bed reactor [20].

**Figure 5.** The effect of temperature on the HHV and water content of bio-oil

3.3.2. Chemical characteristics. The chemical characteristics of EFB bio-oil produced under optimum conditions were evaluated using FT-IR and GC-MS as shown in Figure 6 (a) and Figure 6 (b). Figure 6 shows that the bio-oil produced from EFB are rich in oxygenated

functional groups. The phenol and its derivatives were the major constituents of the bio-oil based on the FT-IR spectra of bio-oil which are comparable with results from literature [20]. The O-H stretching vibration found at the band between 3200 cm^{-1} and 3600 cm^{-1} can be attributed to phenol and alcohol compounds in the bio-oil. Besides that, the C of benzene ring exists in the bio-oil based on the bands between 2800 cm^{-1} and 3000 cm^{-1} which is attributed to C-H stretching vibration of benzene. The presence of methyl group was found in the band between 1350 cm^{-1} and 1470 cm^{-1} which indicates the C-H bending vibrations. In addition, the C=O stretching vibration indicating the presence of aldehyde, ketones and quinones compounds was found at 1705 cm^{-1} . The aromatic compounds and carboxylic acid were found at the band between 1580 cm^{-1} and 1650 cm^{-1} and strong band at 1650 cm^{-1} , respectively. The band between 1500 cm^{-1} and 1510 cm^{-1} is attributed to the alkenes compounds which represents the C=C stretching vibrations. The bands ranging from 950 cm^{-1} to 1300 cm^{-1} represent the C-O stretching and O-H deformation vibration which is attributed to the presence of phenol, esters, ethers and alcohols compounds. On the other hand, the polycyclic and mono aromatic compounds were represented by the peaks existing between 690 cm^{-1} and 900 cm^{-1} and 1350 cm^{-1} and 1450 cm^{-1} .

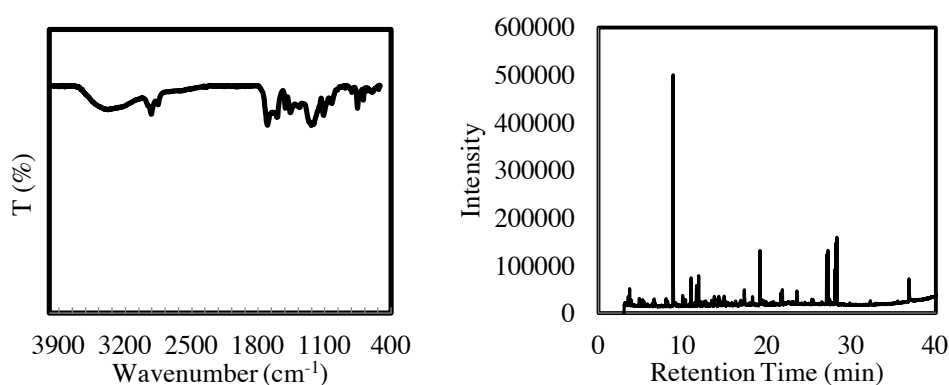


Figure 6. The FT-IR spectra (a) and GC-MS spectra (b) of EFB bio-oil.

The GC-MS was used to identify the type of potential compound existed in the bio-oil produced at $500\text{ }^{\circ}\text{C}$. The result was illustrated in Figure 2 (b). Based on the observation on the GC-MS data, various types of organic compounds are present in the bio-oil, although the phenolic compounds are the major constituents. The bio-oil comprises of heavy organic compounds in the carbon range of C_4 to C_{17} . There are numerous tiny peaks of different compounds, which are not considered in this study. The thermal degradation of EFB yielded a bio-oil containing mostly oxygenated aromatic compounds. Hence, the thermal instability of bio-oil is caused by the presence of phenol and aldehyde group in the bio-oil.

4. Conclusion

The auger reactor was used in the fast pyrolysis of EFB for the production of bio-oil. The maximum bio-oil (58.67 %) was obtained at temperature of $500\text{ }^{\circ}\text{C}$, nitrogen gas flow rate of 1 L/min and biomass feeding rate of 8 kg/h . One homogeneous phase bio-oil was produced throughout the experiments. The water content obtained ranged from 30 to 40 % and it decreased with increasing pyrolysis temperature. The GC-MS data shows that phenol is dominant in the bio-oil since it has the highest peak area ratio compared to any other detectable compound in bio-oil. The bio-oil yield and HHV obtained is consider higher compared to literature and the bio-oil contain high phenolic compounds. Thus, the bio-oil obtained has huge

potential to be used as bio-fuel after up-gradation process, feed for steam reforming for power generation and liquefaction for valuable chemical synthesis.

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