Co – pyrolysis of Empty Palm Fruit Bunch (EPFB) and Low Quality Coal Under CO$_2$ Atmosphere

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Abstract. The objective of this research was to study blending ratio effect on co-pyrolysis of Empty Palm Fruit Bunch (EPFB) and low quality coal using CO$_2$ gas purge on the characteristics of gas, solid and liquid products. Characteristics of gas products consisting of H$_2$ and CO$_2$ gas concentration. Solid product characteristics consist of pore diameter, surface area, total pore volume, solid structure. Meanwhile, the characteristics of liquid products consist of liquid composition. EPFB and low quality coal were reduced to a maximum particle size of about 16 meshes at pre treatment process. The blending ratio of low quality coal and EPFB were 100/0, 80/20, 60/40, 40/60, 20/80 and 0/100, temperature at 500 °C. The result obtained that H$_2$ gas increased between 0 % - 0.423 % due to the higher blending ratio. The concentration of CO$_2$ gas was fluctuated. Meanwhile, pore diameter and total pore volume decreased due to the higher blending ratio. The pores diameter were 61.7316 nm and 18.8361 nm obtained at 0 % and 80 % EPFB, respectively. Total pore volumes were 0.0111 cm$^3$/g and 0.0063 cm$^3$/g obtained at 0 % and 80 % EPFB, respectively. Surfaces area were increased, they were 0.72 m$^2$/g and 1.3480 m$^2$/g at 0 % and 80 % EPFB, respectively. Physical analysis was obtained that the higher of blending ratio (EPFB 100%), the more concentrated of reddish brown color. Liquid products identified contain 41 compounds with the dominant compound of the alkene group. The largest compound were hexadecane (23.69%) and heptacosane (22.61%).

1. Introduction

Energy demands is increasing along with growth in the industrial and transportation sectors. During this time, most of the energy are derived from fossil fuels and minerals that are non-renewable resources. In fact, the amount of fossil fuels and minerals is limited. So it takes effort to reduce dependence on the use of fossil fuels and minerals as well as efforts to optimize the use of new alternative energy sources. Biomass is a source of renewable energy that is environmentally friendly when compared to coal. EPFB is one of the most common biomass in Indonesia. Indonesia is the largest producer of oil palm in the world with an area of 6.17 million Ha in 2013 (BPS). EPFB is a part of palm oil that is classified as untargeted lignocellulosic waste. EPFB is the largest solid waste generated by oil palm plantations. The availability of EPFB is quite abundant reaching 26,697,464 tons per year[1]. During this time, the utilization of EPFB only as fuel boiler, compost and also as hardener in oil palm plantation.
Pyrolysis is a technology to produce alternative renewable energy using biomass materials. It is a high temperature process without present of O2. In general, the material for the pyrolysis process is only biomass. It will cause some disadvantages, such as biomass having high content moisture content, low calorific value, low density and so on[5]. To overcome this problem, biomass and coal co-pyrolysis methods were used. The combination of biomass and coal in the co-pyrolysis process can complement each other's deficiencies. The addition of coal to biomass pyrolysis may affect product composition and distribution. While the high content of H/C in biomass can be used as a H2 donor in the pyrolysis process. The hydrogen can stabilize the large radical structures produced by coal pyrolysis.

Co-pyrolysis process produces three kinds of products: solid, liquid and gas. These three components can be further utilized.

In this study used CO2 as purge gas due to CO2 gas contributed to global warming. The use of CO2 gas as a purge gas in co-pyrolysis will help reduce the emissions of CO2 gas in the atmosphere. In co-pyrolysis using CO2, tar is likely to crack and become gas, otherwise tar will also be polymerized and secondary char formation occurs[2]. The use of CO2 gas as a purge gas is also capable of enlarging the specific surface area of the char and increasing the yield of the gas produced[3]. The solid product formed can be used for fuel or used as activated carbon. Liquid product as bio-oil can be used as an additive or mixture in fuel. Meanwhile, the gas formed can be burned directly or as a source of syngas[7].

This study was expected to know the effect of blending ratio on co-pyrolysis between EPFB and low quality coal using CO2 purge gas in terms of gas, solid and liquid product characteristics. It was analyzed in terms of quantitative (product yield) and qualitative (product composition analysis). In addition, this study was also expected to optimize the use of resources in the form of low quality coal and EPFB in order to produce alternative energy products.

2. Experimental
2.1 Material
EPFB were obtained from Malang, East Java, coal was obtained from Semen Gresik Tuban Factory and CO2 purge gas was obtained from Genta Prima Gas Surabaya.

2.2 Co-Pyrolysis of EPFB and Low Quality Coal Using CO2 Purge Gas
Co-pyrolysis was used free fall reactor that operated at atmospheric pressure. The equipment scheme can be seen in Figure 1. In the preparation stage, there were smoothing and sieving process and also drying process. Coal and EPFB were smoothed and sieved with a maximum size of 16 mesh or 1.19 mm. For the drying process, the finely fed feeds were fed into the oven at 110 °C for 3 hours or until constant weight was achieved. The drying process was needed due to the pyrolysis process was based on the standard drybasis material. Then, the low quality coal and EPFB were mixed according to the variable blending ratio determined by 100 gram. Before the sample was put into the reactor, the reactor was heated with a constant temperature of 500 °C while the CO2 gas was flowed. After the temperature was reached and constant, sample was fed into the reactor through the hopper using gas flow conditions discharged so that no sample is pushed out due to pressure from the CO2 gas. Feed for pyrolysis was dropped slowly into the reactor by adjusting the hopper valve. During the pyrolysis process, the temperature was also kept constant and the CO2 gas carrier fed into the reactor at a constant rate of 1 L/min. This co-pyrolysis process lasts for 1 hour. From the results of co-pyrolysis, produced three types of products were gas, liquid and solid. After the pyrolysis was complete, the reactor was cooled till reached the room temperature. Char was a solid product that collected using a beaker glass and placed at the bottom of the reactor. Liquid or primary gas product was a condensed gas product that turns phase into liquid and collected into erlenmeyer. While, the gas product or secondary gas was an uncondensable gas that collected in a gas holder. The hot solid products, cooled at room temperature and then weighed to obtain a solid mass. Liquid product that was collected in erlenmeyer, then filtered to got solid liquid solid-free product, then afterwards weighed to obtained the
mass of liquid. The mass of gas obtained by reducing the mass of initial feed by the total solid and liquid mass (assuming the input material was equal to the output).

\[ \text{Yield of solid} = \frac{\text{Mass of solid}}{\text{Mass of raw material}} \times 100\% \]  (1)

\[ \text{Yield of liquid} = \frac{\text{mass of liquid}}{\text{Mass of raw material}} \times 100\% \]  (2)

\[ \text{Yield of Gas} = \frac{\text{Mass of gas}}{\text{Mass of raw material}} \times 100\% \]  (3)

2.3 Analysis Methods
In this study was conducted several analysis methods for each product. GC (Gas Chromatography) was used to analyze the composition of CO\(_2\) and H\(_2\) gases in gas product, the equipment was available in Biochemistry Laboratory, Chemical Engineering Department, ITS. The solid product was analyzed using BET (Brunauer-Emmett-Teller) method to determine the total surface area, pore diameter and pore volume performed in the Electrochemistry laboratory, Chemical Engineering Department, ITS. In addition to using BET, solid product was also analyzed using SEM (Scanning Electron Microscopy) method to find out the solid morphology structure of the product conducted in Integrated Physics Laboratory, State University of Malang. While liquid product was analyzed using GC-MS (Gas Chromatography-Mass Spectrometry) to found out the liquid product composition conducted in Gelora Djaya Laboratory, Surabaya.

3. Results And Discussion
3.1 Characteristics of Raw Material
EPFB were obtained from Malang-East Java, while coal obtained from Semen Gresik Tuban Factory. Before use, the feedstock was dried and chopped to a maximum particle size of 16 mesh or 1.19 mm. The characteristics of each raw materials were analyzed by proximate analysis including total moisture, volatile matter, ash content and fixed carbon. ASTM method was used for proximate analysis. The results of the proximate analysis of coal and EPFB were presented below.
Based on the results of the proximate analysis in Table 1, the coal used in this research was categorized as low rank coal (subbituminous). The fixed carbon range for subbituminous coal is about 43.6%. EPFB that used in this study had a lower total moisture and higher volatile matter than coal, obtained about 58.17% and 13.02%, respectively.

3.2 Pyrolysis Result

3.2.1 Blending ratio on EPFB and Low Quality Coal Co-pyrolysis at 500°C

This research was focused on the effect of blending ratio of coal and EPFB on co-pyrolysis at 500°C. Time of pyrolysis was 1 hour, the pyrolysis process was purified using 1L/min of CO₂ gas. The products from pyrolysis process were liquid, solid and gas products. The yield of data obtained from the acquisition of solid, liquid, and gas products is presented in Table 2.

Table 1. Result of coal and EPFB proximate analysis

| Parameter   | Analysis Result (%) | Method |
|-------------|---------------------|--------|
|             | Coal                | EPFB   |       |
| Total Moisture | 27.01              | 13.02  | ASTM  |
| Volatile Matter      | 17.70              | 58.17  | ASTM  |
| Ash content          | 11.73              | 10.43  | D3174 |
| Fixed Carbon        | 43.56              | 18.38  | -      |

Table 2. Result of pyrolysis product of EPFB and low quality coal at Low Temperature 500°C

| Coal       | Liquid (%) | Solid (%) | Gas (%)    |
|------------|------------|-----------|------------|
| 100 %      | 2.7053±2.5 | 70.9358±0.5 | 26.3589±1.5 |
| 80 %       | 2.7200±1.03 | 69.5830±1.04 | 27.6970±2.4 |
| 60 %       | 3.2113±1.08 | 67.7850±0.1 | 29.0037±0.13 |
| 40 %       | 3.7186±1.7 | 65.1030±0.2 | 31.1784±0.61 |
| 20 %       | 4.4427±1.03 | 48.1288±9.2 | 47.4285±10.74 |
| 0 % (EPFB 100 %) | 5.1744±2.13 | 30.7161±3.15 | 64.1095±1.74 |

Figure 2. Effect of Blending Ratio on Yield of Products

It can be concluded from Figure 2 that the greater blending ratio of the coal, the more yield of liquid and gas obtained, meanwhile the less yield of solid product obtained. It was happened due to the
highest blending ratio of coal (100 %), the components in coal were more difficult to evaporate and decompose than the components in EPFB. It can be proved by looking at the volume of volatile matter of coal (17.70 %) less than the volatile matter of EPFB (58.17 %). The yield of solid product was larger compared to liquid and gas due to during the pyrolysis process it was possible to formed a second char. In addition, CO\textsubscript{2} gas reacted with liquid products cause the decreasing of liquid products while gas products increased. The increasing of micropore caused the rich of solid product in carbon elements. Other factors that influence product yield were particle size, temperature, heating rate, residence time, steam/biomass ratio, catalyst and others[2]. However, this study more focused on the influence of blending ratio.

### 3.3 Analysis Result

#### 3.3.1 Effect of Blending Ration on BET Analysis Result

Based on Table 3, it was found that blending ratio had an effect on char pore diameter, char surface area and char total pore volume. With high blending ratio condition, pore diameter and total pore volume were decreased while surface area was increased. Based on the theory, the effect of CO\textsubscript{2} gas in pyrolysis was to increase the surface area of char[2]. The most important factor affecting particle size is temperature, the higher of temperature, the smaller of particle size obtained[8]. It was also obtained the decreasing result of pore diameter and total pore volume. Based on the theory, the decreasing of particle char size had an effect on the increasing of gas yield and decreasing of liquid and solid yield. Particle size not only affecting heat and mass transfer but also the possibility of a second reaction within a char particle. The volatile component in char can trigger a second reaction (eg cracking, condensation and polymerization) inside the char particles. The polymerization of some volatile components due to the accumulation of large molecules on the pore wall, resulting in increasing of yield char and decreasing of volatile components, is occurring more frequently if the particle size of char is larger[4].

| Parameter          | Coal : EPFB |
|--------------------|-------------|
|                    | 100:0       | 80:20     | 40:60     | 20:80     |
| Pore diameter (nm) | 61.7316     | 30.3056   | 46.5352   | 18.8361   |
| Surface area (m\textsuperscript{2}/g) | 0.7230      | 2.7190    | 0.7090    | 1.3480    |
| Total pore volume (cm\textsuperscript{3}/g) | 0.0111      | 0.0206    | 0.0082    | 0.0063    |

#### Table 4. BET analysis result of feedstock

| Parameter          | Feedstock |
|--------------------|-----------|
|                    | Coal      | EPFB      |
| Pore diameter (nm) | 74.4808   | 51.9846   |
| Surface area (m\textsuperscript{2}/g) | 0.2150 | 0.1480     |
| Total pore volume (cc/g) | 0.0040 | 0.0019   |

Table 4 is the result of BET analysis on feedstock. It can be seen that the difference of pore diameter on coal material before and after pyrolysis process, which was approximately 12.7492 nm. The pore diameter of the coal feedstock before pyrolysis was greater than after pyrolysis. Meanwhile, the surface area of coal feedstock was larger after pyrolysis. It indicates that pyrolysis was capable of enlarging the material surface area. Similarly, the total pore volume becomes larger after pyrolysis.
3.3.2 Effect of Blending Ratio on SEM Analysis Result

Figure 3 shows the SEM analysis of co-pyrolysis solid product at 500 °C. Images were taken with 1500 times of magnification. The three images were almost identical, having irregular, heterogeneous and irregular structures. However, if observed by naked eye, EPFB had more pores (more porous) than coal.

![SEM Analysis Images](image)

**Figure 3.** SEM Analysis (a) Coal: EPFB = 40:60 (b) Coal : EPFB = 100:0 (c) Coal: EFFB = 0: 100

3.3.3 Effect of Blending Ratio on GC Analysis of Gas Product

Pyrolysis gas process was a product that cannot be condensed into liquid products. During the pyrolysis process, the resulting gas is accommodated in a gas trap (balloon). GC analysis was used to know the level of H$_2$ and CO$_2$ on gas. Char was converted into gas by thermal cracking process in pyrolysis process. Char was very reactive with CO$_2$ gas.

3.3.4 Effect of Blending Ratio on Physical and Chemical Condition of Gas Product

Liquid product that obtained from this research was called as bio-oil. Bio oil is the main product desired from any pyrolysis process because it has many advantages. One of them is as a fuel mixture. Bio oil in the liquid phase was obtained by condensing the output gas from the reactor. Not all amounts of gas phase changed into a liquid. The condensed gas was called the primary gas, while the non-condensed gas was called secondary gas. The physical differences of liquid products in each blending ratio are shown in Table 6.

Shortly after the liquid product was obtained, the comparison of color was done by naked eye. In general, bio oil was reddish brown color. Based on Table 6, it is represent the difference between each blending ratio. In the variable coal/EPFB = 100/0 obtained a bright reddish brown liquid product. As the blending ratio increased, the density of color and texture of liquid products was sharper till the dark brown color obtained from 100% EPFB. EPFB gave a sharper and thicker brown color due to cellulose, hemicellulose and lignin content contained in EPFB. Based on the GC results in Table 5 can be concluded that the higher of blending ratio (EPFB 100%), the higher of H$_2$ gas concentration obtained. It was happened due to the fact that biomass is rich in H$_2$. So as to be possible in this pyrolysis process, EPFB functions as a H$_2$ donor and allows for synergistic effects[8]. However, the highest concentration of H$_2$ gas was obtained in blending ratio of coal: EPFB = 20:80, it was equal to 0.8258 %. This condition were optimal conditions to produce H$_2$ gas during pyrolysis process.

The concentration of CO$_2$ gas was fluctuated due to the pretreatment of different raw materials. So, H$_2$O content in the raw materials also varies as well. The amount of H$_2$O caused more CO$_2$ formation, because H$_2$O was also an oxygen supply (O$_2$) which reacted with carbon (C) and produced CO$_2$. In addition, CO$_2$ used as a purge gas was also possible to react with hydrogen supplied by biomass and form H$_2$O and CO.[2]

**Table 5.** GC Result of Gas Product

| Coal/EPFB | Surface area | Gas volume in syringe (mL) | Concentration (v/v) |
|-----------|--------------|----------------------------|---------------------|
| 40:60     |              |                            |                     |
| 100:0     |              |                            |                     |
| 0: 100    |              |                            |                     |
The volume of liquid products obtained was varying due to less optimal condensation process during pyrolysis. The efficiency of the condenser in the course of the pyrolysis process was less than the maximum due to several things, the gas contact with the cooling medium was minimum and the blockage of the condenser by feedstock.

In addition to the effect of equipment unefficiency, the volume of liquid products was also affected by the type of reactor used. Since the reactor type was a free fall reactor, with a height reactor design of about 90 cm enabling when the pyrolysis process occurs, the reaction between feedstock and CO\textsubscript{2} purge gas had some problems. High reactors and a small volume of feedstock caused the volume of gas spaces was high. It cause temperature instability in the reactor, the temperature panels often showed a temperature drop about 20 °C. In addition, the distance between the reactor heights with a condenser length prevented the condensing of gas; it was affected to the formed liquid product.

In addition to known physical conditions of liquid products were formed, chemical conditions also needed to analyze using GC-MS. The obtained liquid was an unhomogeneous oil phase, tar and water was not homogen. The content of water in tar was not separated due to many compounds contained in tar dissolved in water. The following Figure 4 is a liquid chromatogram of liquid product and Table 7 is a component contained in liquid products.

### Table 6. Physical differences of liquid product on each blending ratio

|  EPFB  | H\textsubscript{2} | CO\textsubscript{2} | H\textsubscript{2} | CO\textsubscript{2} | Total (H\textsubscript{2}+CO\textsubscript{2}) | H\textsubscript{2} | CO\textsubscript{2} |
|--------|-----------------|-----------------|-----------------|-----------------|---------------------------------|-----------------|-----------------|
| 100/0  | -               | 66.320          | -               | 0.237783        | 0.237783                        | -               | 118.891399%    |
| 20/80  | 4955            | 58378.0         | 0.01652         | 0.209308        | 0.210959                        | 0.825833%       | 104.653831%    |
| 40/60  | 892             | 45.588          | 0.000297        | 0.163451        | 0.163748                        | 0.148667%       | 81.725288%     |
| 60/40  | 1.134           | 61.609          | 0.000378        | 0.220892        | 0.221270                        | 0.189000%       | 110.446022%    |
| 80/20  | -               | 52.645          | -               | 0.188753        | 0.188753                        | -               | 94.376322%     |
| 0/100  | 2.538           | 86.304          | 0.000846        | 0.309433        | 0.310279                        | 0.423000%       | 154.716575%    |

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In addition to the effect of equipment unefficiency, the volume of liquid products was also affected by the type of reactor used. Since the reactor type was a free fall reactor, with a height reactor design of about 90 cm enabling when the pyrolysis process occurs, the reaction between feedstock and CO\textsubscript{2} purge gas had some problems. High reactors and a small volume of feedstock caused the volume of gas spaces was high. It cause temperature instability in the reactor, the temperature panels often showed a temperature drop about 20 °C. In addition, the distance between the reactor heights with a condenser length prevented the condensing of gas; it was affected to the formed liquid product. In addition to known physical conditions of liquid products were formed, chemical conditions also needed to analyze using GC-MS. The obtained liquid was an unhomogeneous oil phase, tar and water was not homogen. The content of water in tar was not separated due to many compounds contained in tar dissolved in water. The following Figure 4 is a liquid chromatogram of liquid product and Table 7 is a component contained in liquid products.

### Table 6. Physical differences of liquid product on each blending ratio

| Coal/EPFB | Color            | Odor  | Volume (mL) | Density (g/mL) |
|------------|------------------|-------|-------------|----------------|
| 100/0      | Bright reddish brown | Oil scent | 3           | 0.9            |
| 80/20      | Reddish brown slightly dark | Oil scent | 2.9         | 0.94           |
| 60/40      | Darker reddish brown | Oil scent | 4.2         | 0.76           |
| 40/60      | Darker reddish brown | Oil scent | 3.9         | 0.95           |
| 20/80      | Almost solid reddish brown | Oil scent | 5.8         | 0.77           |
| 0/100      | Solid brown      | Oil scent | 4.7         | 1.1            |
Figure 4. Chromatogram result of GC-MS analysis
on blending ratio Coal/EPFB = 80/20

Based on the results of analysis using GC-MS obtained 41 compounds that were identified. With the largest area were hexadecane (23.69%) and heptacosane (22.61%).

3.3.5 Synergis Effect
Coal and EPFB have complex structures, which may indicate differences in thermal decomposition. It was enable the synergistic effect between the two raw materials both in quantity and quality. Synergistic effects were defined as an increase or decrease in the results of co-processing experiments with the results of calculation[6]. Based on the approach taken by [6],[8]:

$$Y_{cal} = Y_c \times BR_c + Y_b \times (1 - BR_c)$$

(4)

Where: $Y_{cal}$ was the yield of the calculated result, $Y_c$ and $Y_b$ were yields of individual experimental measurements of coal and EPFB, and $BR_c$ was the blending ratio of coal. If $Y_{exp} > Y_{cal}$ there was a synergistic effect with the addition of EPFB. The following were the $Y_{exp}$ and $Y_{cal}$ calculations of each blending ratio at 500 °C.

| Coal:EPFB | Solid | Liquid | Gas |
|-----------|-------|--------|-----|
|           | Exp   | Cal    | Exp | Cal   | Exp | Cal |
| 100/0     | 70.935| 70.9358| 2.705| 2.7053| 26.358| 26.358|
| 80/20     | 69.583| 62.8918| 2.72 | 3.1991| 27.697| 33.909|
| 60/40     | 67.785| 54.8479| 3.211| 3.6929| 29.003| 41.459|
| 40/60     | 65.103| 46.8039| 3.718| 4.1867| 31.178| 49.009|
| 20/80     | 48.128| 38.7600| 4.442| 4.6805| 47.428| 56.559|
| 0/100     | 30.716| 30.7161| 5.174| 5.1744| 64.109| 64.109|

Based on Table 7 it can be concluded that there was a synergistic effect on solid product where $Y_{exp} > Y_{cal}$. Synergistic effects occurred when there was a chemical interaction between two materials. Synergistic effect occurred when the process was in a condition above the heating temperature, ie 500 °C. According to Pan YG, under low heating conditions, two processes will be separated and no synerggetic effects are found. Factors that can increase the synergistic effect not only temperature and blending ratio but also rate of heating and reaction time[8]. High heating rates and short reaction residence time prevented the formation of secondary reactions. So, the more primary gas was produced, which was condensable gas becomes tar. However, short residence time made reaction contact between two raw materials was not guaranteed.

4. Conclusion
It can be concluded that the higher blending ratio (EPFB 100 %) caused the concentration of H$_2$ gas increased, the CO$_2$ gas concentration fluctuated and gas yields increased. The influence of blending ratio on solid characteristic was higher blending ratio (EPFB 100 %), char pore diameter and total pore volume tend to decreased, surface area tend to increased and yield of solid decreased. While, the effect of blending ratio on the liquid characteristic was the higher blending ratio (EPFB 100 %), the darker of reddish-brown color, the density increased and the liquid yield increased.

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