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# Activated Carbon of Oil Palm Empty Fruit Bunch (EFB); Core and Shaggy

Osman NB[a,b,*], Shamsuddin N[a], Uemura Y[a,b]

[a]*Chemical Engineering Department Universiti Teknologi PETRONAS, Bandar Seri Iskandar, Perak, 32610 MALAYSIA,*
[b]*Center for Biofuel and Biochemical Research,Universiti Teknologi PETRONAS, Bandar Seri Iskandar, Perak, 32610 MALAYSIA*

**Abstract**

Oil palm empty fruit bunches activated carbon of different parts (shaggy and core) were characterized as to convert waste of oil palm-based into value added products. Conventional step processes of physical activation were performed where activation was undertaken after the pyrolysis process (carbonization-activation process). The pyrolysis temperatures applied were 400, 450, and 500°C in inert condition under nitrogen flow. For the activation process, 600, 700, and 800°C with the presence of 15%, 60%, and 100% $CO_2$ and holding time of 30, 60, and 120 minutes were applied. Results showed that activation temperature, $CO_2$ percentage and holding time did dictate the changes of activated carbon yield, carbon content, and textural properties of the activated carbon core and shaggy EFB produces. The highest yield of activated carbon was obtained from 700°C (middle temperature), 60% $CO_2$ (middle percentage), and 30 mins holding time (shortest). However, the highest carbon content was detected from 600°C, 60% CO2, and 60 mins of activated carbon core EFB. The size of pore determined prove that the three parameter do not correlate with each other as the bigger pore diameter was derived from 700°C, 60% of $CO_2$, and 60 mins holding time. Hence, the results revealed that there are differences of activated carbon properties when we separate EFB part into its core and shaggy.


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*Keywords:* Activated carbon; core EFB; oil palm oil empty fruit bunch; physical activation; pyrolysis; shaggy EFB

## 1. Introduction

Commercially available activated carbons were prepared from non-renewable and expensive which also contributed to uncontrol air pollution. Although, the fact it can be produced from any carbonaceous materials but in the early day, most commercialized activated carbon were coming from petroleum residues, wood, coal, lignite, and peat [1]. Nonetheless, these materials have been employed in a wide number of applications on an industrial scale including, purification technologies, removal of pollutants, and electrochemical devices. As activated carbon can be produced by both naturally occurring and synthetic of carbonaceous solid precusor it has been classed based on its starting material [1]. Hence, the production of activated carbons from waste agriculture products in particular oil palm biomass has been explored since 1996 [3,4].

Biochar is the product of thermal decomposition processes commonly by pyrolysis process with temperature below 700°C [5]. Lower pyrolysis temperature and lower heating rate process produce more char than liquid or gas product. The bio-char is used to be an underrated material until recently when their application has been expanded from soil enrichment to activated carbon. EFB has been used as a feedstock for bio-char production which will be applied later in different products [6-9].

* Corresponding author. Tel.: +6-05-368 7636; fax: +6-05-365 6176.
*E-mail address:* noridah.osman@petronas.com.my


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The use of oil palm based waste for activated carbon products specifically empty fruit bunches has been discussed by researchers [10-16]. Apart of the high volume of waste, its cost, properties, and environmental friendly factors, the EFB material different parts in specific core and shaggy have never been study as far as author knowledge. Therefore, the goal of this study was to analyze the activated carbon produced from oil palm empty fruit bunches (EFB) of two parts specifically core and shaggy by single step activation process after pyrolysis (carbonization process). In addition, the activated carbon properties will be discussed for it individual part (core and shaggy) and mixture.

## 2. Materials and Methods

All material was obtained from Oil Palm plantation nearby university area. Two parts of empty fruit bunches (EFB) of shaggy and core were prepared for the activated carbon feedstock (Figure 1). The pretreatment process, the material were processed by cutting to small size (original size to chopper size) and then dried at 105°C for 24 hours. The dried EFB was grinded to and sieved to 0.5 – 1.0 m and then stored in desiccators to preserve the moisture content (MC) which less than 10% and the later experiment. For EFB raw material, two approximate analysis of moisture content (ASTM E871) and ash content were conducted (ASTM D2866). Scanning electron microscope (SEM) (Model TM3030 HITACHI) was carried out for textural characteristic of outer part of EFB.

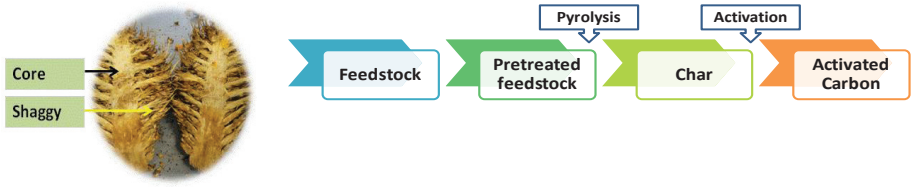


Fig. 1. Empty fruit bunch, core and shaggy (left) and schematic diagram of activated carbon production (right).

Figure 1 shows the schematic of activated carbon production where the activated carbon started from charring of EFB. And, the experimental design is shown in Table 1. The bio-char was derived from pyrolysis process of EFB in a drop type pyrolyzer at 450°C. The ultimate analysis was conducted for the bio-char by Series 11 CHNS/O Analyzer 42400 Perkim Elmer in particular determination of carbon content. The textural properties of char were also determined with the same SEM instrument.

Table 1. An experimental design for the activated carbon of oil palm EFB bio-char (Pyrolysis- 450°C).

| Type / Activation | Temperature (°C) | $CO_2$ % | Activation time (mins) |
|---|---|---|---|
| Core & Shaggy | 600 | 60 | 60 |
| | 700 | 60 | 60 |
| | 800 | 60 | 60 |
| | 700 | 15 | 60 |
| | 700 | 60 | 60 |
| | 700 | 100 | 60 |
| | 700 | 60 | 30 |
| | 700 | 60 | 60 |
| | 700 | 60 | 120 |

Activation process was performed by a tube furnace for EFB pyrolized bio-char as Figure 2. The boat was loaded with 1.5g of bio-char then activated with the desired temperature (600, 700, and 800°C). Heating rate was set to a rate of 20°C/min and nitrogen gas ($N_2$) flow. A carbon dioxide gas ($CO_2$) was introduced to activate the sample at 15%, 60%, and 100% with different activation time, 30, 60, and 120 mins. After the activation step, solid samples were left in the reactor for cooling step to room temperature under $N_2$ gas flow. Further analysis were conducted for these samples for it physical properties, their yield and morphology characteristic (SEM image). In term of activated carbon yield (Y %), it is calculated based on mass (dry weight, g) of the final activated carbon product ($W_c$) over the initial mass of char precursor (dry weight, g) ($W_o$) times 100 ($Y = W_c / W_o \times 100$). In addition, Fourier Transform Infrared (FT-IR) spectroscopy analysis to determine its surface functional groups of raw and activated carbon EFB (potassium bromide-KBr accessory, spectrum range 500-4000 $cm^{-1}$).

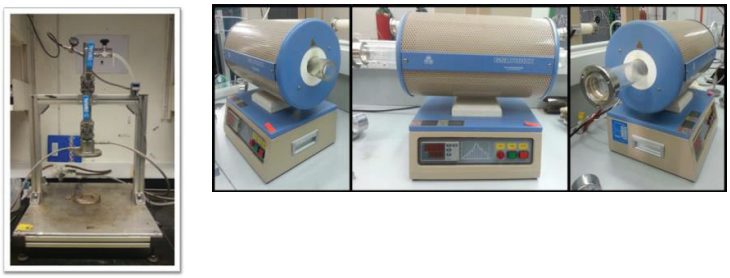

Fig. 2. Drop type pyrolyzer (left) and tube furnace (right).

## 3. Results and Discussion

The results of the proximate analysis of raw EFB were comparable for both core and shaggy with 3.8% moisture content and ash content were 1.4% and 1.3% respectively [7,8]. EFB-char ultimate properties are shown in Table 2 with C, H, and N composition [9]. The samples were obtained randomly from different spot of core and shaggy materials as cited by numbering (e.g. C1 and S1). Shaggy char showed lower carbon content compare to core parts underwent 450°C. These values were higher compare to Abu Sari et al. (54.08% C, pyrolysis 500°C) and comparable to Sukiran et al. (65% C, 400 and 500°C) however slightly lower than Shariff et al. (72.23% C, pyrolysis 550°C) [7,8]. This could be due to the relationship between ash and carbon contents but the differences of carbon content is not significance when the ash content is lower than 10%. Although the percentage of carbon content did showed an increment with reduction of ash content of the char EFB precursor as reported by Shariff et al. [8].

Table 2. Properties of EFB char, core-c and shaggy-s (Pyrolysis- 450°C).

| Char | Percentage (%) | | |
|---|---|---|---|
| Sample | Carbon, C | Hydrogen, H | Nitrogen, N |
| C1 | 71.43 | 3.50 | 0.72 |
| C2 | 69.47 | 3.21 | 0.61 |
| C3 | 71.81 | 3.17 | 0.70 |
| S1 | 62.13 | 4.49 | 0.90 |
| S2 | 65.21 | 3.54 | 0.89 |
| S3 | 62.71 | 3.96 | 0.80 |

### *3.1 Activated carbon*

With temperature different for pyrolysis, the results in Table 3 show that C, H, and N % also changed for both shaggy and core for same activation process parameter (700°C, $CO_2$ 60%, 60 mins activation time). For shaggy samples, the carbon content (C%) were 41.16, 41.11, and 41.56% for 400, 450, and 500°C, respectively. Meanwhile for core sample, the C% was slightly higher 48.74, 51.52, and 53.29% with the same ascending pyrolysis temperature. Wirasnita et al. recorded relatively high carbon content 71.23% in comparison to our finding at substantially lower activation temperature 500°C and Hidayu et al. reported 68.32% carbon content of 765°C [12,16]. Based on these two studies it could be concluded that the lower carbon content may be a result of pyrolysis process acting as double process of heating the material (carbonization-activation process). In which, the core part showed even lower than shaggy as proving process contribute to the changes of carbon content in activated carbon material.

Table 3. Effect of different pyrolysis temperature with similar activation condition on carbon content (%).

| Char | Pyrolysis | Percentage (%) | | | Carbon (C%) |
|---|---|---|---|---|---|
| Sample | Temp (° C) | Activation time (min) | $CO_2$ (%) | Temp (° C) | |
| Core | 400 | 60 | 60 | 700 | 41.16 |
| | 450 | 60 | 60 | 700 | 41.11 |
| | 500 | 60 | 60 | 700 | 41.56 |
| Shaggy | 400 | 60 | 60 | 700 | 48.74 |
| | 450 | 60 | 60 | 700 | 51.52 |
| | 500 | 60 | 60 | 700 | 53.29 |

Activated carbon yield showed that as temperature increase (600, 700, 800°C) the yield decrease by less than 3% for both shaggy and core (29.16%, 28.10%, 26.80%) as in Table 4. Although it shows that the reduction in gradually manner and may be not even significant different at large and distinct temperature different. Interestingly, the trends were similar for shaggy and core of EFB activated carbon samples on both AC yield and carbon content. However this activated carbon yield may not correlate with carbon content, 70.38, 41.11, and 63.6% of shaggy sample and 60.56, 51.52, and 55.56% of core EFB samples upon changes of activation temperature. The higher yield at low temperature may be due to slow rate of carbon and carbon dioxide reaction with the weight loss resulted from the released of volatile matters [17]. Our results also indicated higher AC yield values than studies by Alam et al. (25-27% AC yield) and Hameed et al. (17-21% AC yield) [10,11]. Alam et al. found that activation temperature have the greatest effects to the activated carbon yield which opposite than our finding [11]. Nonetheless, activation did change the activated carbon yield content in the core and shaggy samples and also altered their carbon content.

Table 4. Effect of activation temperature on activated carbon (AC) yield and carbon content (%).

| AC | Percentage (%) | | | AC Yield (%) | Carbon (C%) |
|---|---|---|---|---|---|
| Sample | Activation time (min) | $CO_2$ (%) | Temp (° C) | | |
| Core | 60 | 60 | 600 | 29.16 | 70.38 |
| | 60 | 60 | 700 | 28.10 | 41.11 |
| | 60 | 60 | 800 | 26.80 | 63.6 |
| Shaggy | 60 | 60 | 600 | 33.55 | 60.56 |
| | 60 | 60 | 700 | 29.22 | 51.52 |
| | 60 | 60 | 800 | 28.57 | 55.56 |

Table 5 shows the effect of $CO_2$ percentage on yield of activated carbon is significant at temperature 700°C with holding time 60 mins (Table 5). $CO_2$ percentage has a control on the rate of reaction of carbon and $CO_2$ can be seen from the reduction of activated carbon yield as discussed by Yang et al. [17]. So far author could not retrieve research on physical activation with different $CO_2$ condition, the closes one was Alam et al. reported on several $CO_2$ flow rate which displayed very little effect on activated carbon yield, 27.65% [11].

Table 5. Effect of $CO_2$ percentage % on activated carbon (AC) yield and carbon content (%).

| AC | Percentage (%) | | | AC Yield (C%) | Carbon (C%) |
|---|---|---|---|---|---|
| Sample | Activation time (min) | $CO_2$ (%) | Temp (° C) | | |
| Core | 60 | 15 | 700 | 32.70 | 54.02 |
| | 60 | 60 | 700 | 29.22 | 51.52 |
| | 60 | 100 | 700 | 27.52 | 54.03 |
| Shaggy | 60 | 15 | 700 | 31.19 | 67.82 |
| | 60 | 60 | 700 | 28.10 | 41.11 |
| | 60 | 100 | 700 | 25.66 | 58.51 |

Meanwhile, the effect of holding time on the yield of activated carbon is shown in Table 6. As the holding time increase the activated carbon yield decreased versus carbon content increased for both core and shaggy samples. This could be expected as the longer holding time more release of volatiles matter will occurs. In addition, the extent of carbon-carbon dioxide reaction will increase leading to higher carbon burn-off. Our activation time is longer three times than the Alam et al. highest activation time (45 mins) [11]. Noticeably, their activated carbon yield lower content than our study which indicates that activation time plays some role in the activation process. In addition, the carbon content showed higher value shaggy than core sample which improved carbon properties while compromising the AC yield.

Table 6. Effect of holding time on the yield of activated carbon (AC) and carbon content (%).

| AC | Percentage (%) | | | AC Yield (wt%) | Carbon (C%) |
|---|---|---|---|---|---|
| sample | Activation time (min) | $CO_2$ (%) | Temp (° C) | | |
| Core | 30 | 60 | 700 | 36.18 | 55.04 |
| | 60 | 60 | 700 | 29.22 | 51.52 |
| | 120 | 60 | 700 | 28.29 | 57.16 |
| Shaggy | 30 | 60 | 700 | 30.57 | 63.04 |
| | 60 | 60 | 700 | 28.10 | 41.11 |
| | 120 | 60 | 700 | 27.27 | 69.1 |

*3.2 Functional Groups of activated carbon core and shaggy EFB*

Figure 3 shows FTIR spectra for the EFB core and shaggy activated carbon with 60 mins holding time, 600°C, and 60% $CO_2$ activation condition. In the FTIR spectrum of raw oil palm EFB sample, a broad and strong band at 3400 $cm^{-1}$ for hydroxyl group (-OH) stretching vibration was reported by Wirasnita et al. [16] however observed that the band shrunk for activated carbon samples. Vaporization of moisture content would be the significant caused for the reduction during the activation process [13].The disappearance of this group was also severe when compare to char sample as recorded by Abu Sari et al. (2014) [9]. Meanwhile, the absorption peaks at 2900-2850 $cm^{-1}$ for C-H stretching vibration of the $–CH_3$ group was totally removed from activated carbon samples. Carbonyl groups (C=O) were observed present in the native EFB at 1740-1700 $cm^{-1}$ which expected derived from lignin network [18]. However, this peak almost absence after pyrolysis and the activation process due to vaporization of volatile matters. The peaks range in between 1200-1000 $cm^{-1}$ were assigned to C-O stretching as well as 830 $cm^{-1}$ Si-O as a yield of silica containing minerals [9,19,20]. Hidayu et al. stated that IR spectra have proved that their prepared activated carbon was successfully converted into carbon [12]. Indeed, this is congruent with our finding although core and shaggy samples did showed much different in term of their functional group where core sample is not much affected with activation condition as compare to shaggy sample.

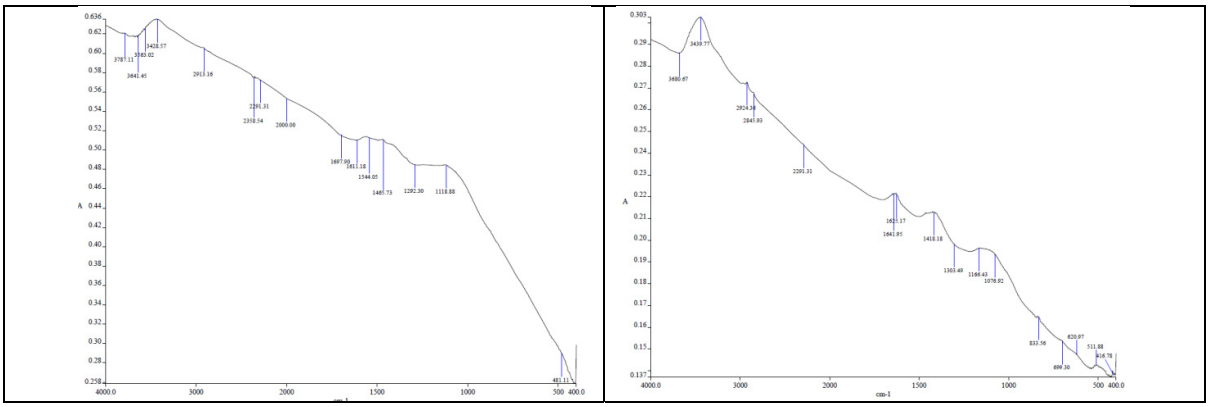

Fig. 3. FTIR spectra for EFB activated carbon of core (left) and shaggy (right) samples (600°C activation temp, 60% C.

*3.3 Textural Characteristic of dried raw, pyrolysis char, and activated carbon core and shaggy EFB*

Figure 3 shows the micrograph of raw EFB core and shaggy samples which the white circular craters of silica-bodies proof the expected future problem of EFB [11]. The presence of this silica commonly complicated the pulping and bleaching manufacture products of this material as the silica-bodies are hard. Average size of this core part from cross-section angle is about 6-7 µm pore diameter as supported by Law et al. [21]. The image of EFB char seems to be disintegrated when compare to the original structure of core EFB (outer surface). Moreover, the small cell cavity with non-developed porosity and patches of crack were observed as well as the cell wall breakdown. It was noticed that char average pore diameter size was wider comparable to Abu Sari et al. [9], with slightly bigger range 10 µm up till about 30µm. Nonetheless, the credit of producing uniform pores and smooth wall surface pyrolysis process cannot be denied showed by both parts of EFB [9,10]. Upon activation process the pore was developed further shown in the image of core EFB activated carbon. This indicates that the porosity was expanded by activation agent and process [10,11,16]. The average size determined of pore cross section diameter was about 8µm. Shaggy EFB is shown in Figure 3 (right) with white circular crater was also spotted on the raw material similar to core sample. Its pore size of cross section determined to range between 10-14 µm which is slightly wider than core sample. However, the pyrolysis process significantly impact the pore structure compare to core sample as indicated by the irregular cavity developed on the outer surface of the material. Meanwhile, after activation process the average pore diameter from cross section view was about 12.6 µm which is also larger than core sample. The images of SEM micrographs of the both core and shaggy samples activated carbon demonstrated that carbonization by pyrolysis and activation processes created porosity and large surface area for absorption thus aligned with the literature which is the goal in production of activated carbon.

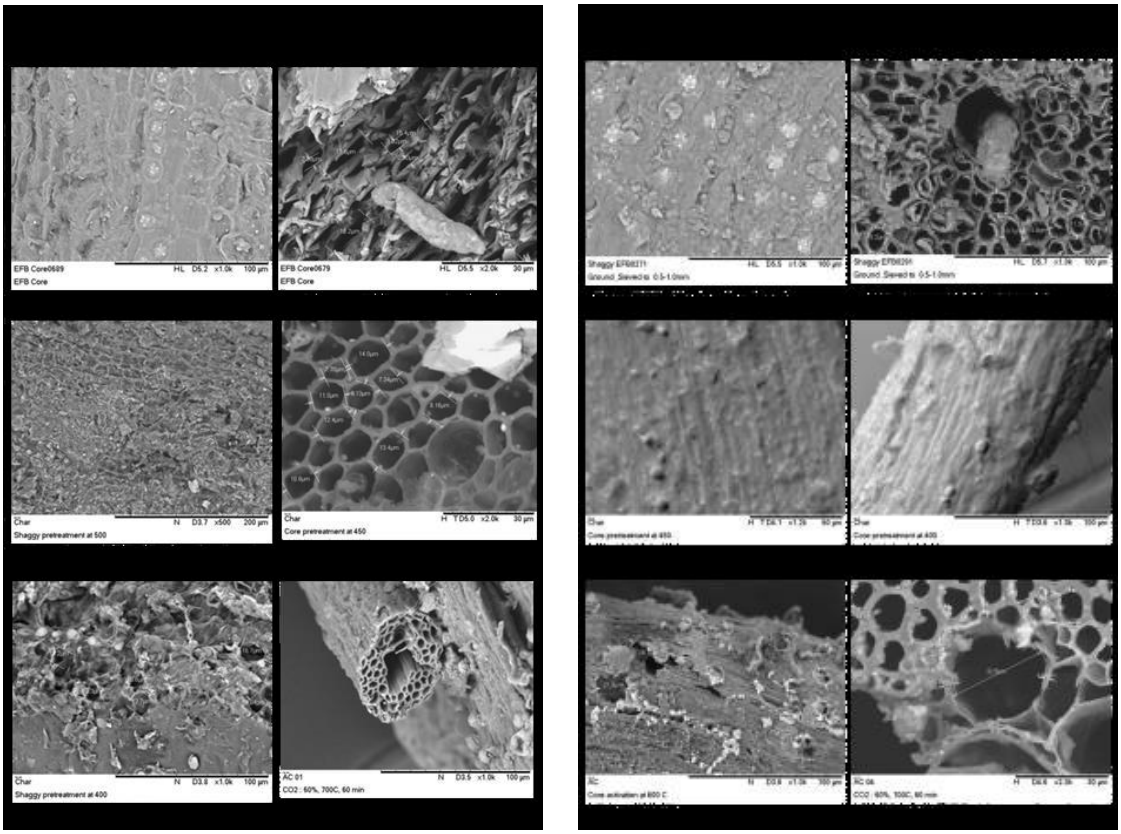

Fig. 3. (a) Micrograph of raw, char, and activated carbon of EFB core (left) and shaggy (right) samples.

## 4. Conclusion

This study has shown that both part of EFB shaggy and core char have high amount of carbon making them a good starting materials or precursor to produce activated carbon. Carbon content of char sample from both core and shaggy EFB has showed significant amount to be prepared as precursor for activated carbon. The activated carbon yield and carbon content (%) showed slight differences with each other upon carbonization-activation process for both core and shaggy materials. The highest yield of activated carbon precursor was produced at the medium activation temperature (700°C), medium $CO_2$ (60%), and shortest holding time (30 mins). There are significant different between raw or native EFB core and shaggy samples with clear results when compare to char and activated carbon. In addition, the generation of uniform pore and porosity of activated carbon samples were successful for both materials as well in which the removal of volatile matter and decomposition of chemical component occurred during the process. This proved that the materials are potential for precursor activated carbon at low price. Further, this single step physical activation process certainly minimizing the cost of production using EFB material.

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