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# Durability Properties of Demineralized and Torrefied Empty Fruit Bunch (EFB) Pellets

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**Abstract**. The pelletization of raw and demineralized and torrefied empty fruit bunch (TDEFB) were conducted upon mixing with bio-oil that was obtained from EFB pyrolysis. The ratio of bio-oil addition to raw and TDEFB was varied from 0-20 wt%. Then it was pressed using the single pellet press machine. The effects of the ratio of bio-oil addition to raw and TDEFB and the thickness of pellets were evaluated towards its durability. It was identified that the ratio of bio-oil addition to raw and TDEFB pellet of 5 wt% had produced the optimum durability which was 69.27 and 74.46 % respectively. In terms of the thickness of the raw and torrefied pellets, it was determined that at thickness of 3 cm, the durability was the highest which were 70.12 and 71.12 % for both raw and TDEFB pellets respectively. The raw and TDEFB pellets were analysed using Fourier Transform Infra-Red (FTIR) spectroscopy in order to evaluate the presence of functional groups within the materials. The presence of O-H hydroxyl group, C=O from carbonyl or ester and C-H alkane groups are detected in both raw and torrefied pellets.

## 1. Introduction

Crude palm oil (CPO) production has contributed highly towards Malaysian economy over these years. It was estimated that one tonne of fresh fruit bunch co-generated approximately 0.20 – 0.30 tonnes of empty fruit bunches (EFB) [1]. EFB biomass waste can be converted into suitable solid fuel via thermochemical processing such as torrefaction and pyrolysis [2-8]. However, like any other untreated biomass, EFB has high moisture contents and low calorific value. Therefore, EFB requires suitable pretreatment techniques in order to reduce the moisture content and improve the calorific value. Torrefaction process is defined as biomass thermochemical treatment that occurs from 200-300 $^{o}$C under inert environment at atmospheric pressure [9].The combination of torrefaction and demineralization on EFB has been reported as a prominent technique to improve the fuel property of EFB [10]. During the process of biomass torrefaction, the cell walls are disrupted and the product exists in loose solid form with increased hydrophobicity which is called torrefied biomass [11].

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During the process of biomass torrefaction, the cell walls are disrupted and the product exists in loose solid form which is called torrefied biomass [11]. Torrefied biomass has its limitation such as being loose that hinders its utilization as solid fuel eventhough its energy density has been improved. Therefore, pelletization is a technique that can overcome this limitation that allows the torrefied biomass to behave as solid fuel.

Biomass in the pellet form is commercially used as a commodity product for energy application. Several researchers have reported about the utilization of different types of binder such as fatty acid and resin [12], protein and water [13] and glycerol [14] during the formation of mainly raw biomass pellet. It is identified that there is scarce information that reports the effects of bio-oil from EFB pyrolysis as binder towards the durability of the pellets from raw and torrefied and demineralized EFB (TDEFB) as well as the morphological properties of raw and torrefied EFB pellets.

## 2. Materials and methods

### *2.1 Sample preparation*

The EFB was acquired from North Star Palm Oil Mills which was located in Kuala Ketil, Kedah, Malaysia. EFB was washed and rinsed to remove some impurities. Then, EFB was placed in the oven at 80 ºC for 24 hours. EFB was ground using grinder and sieved to select the particle size using Retsch sieve shaker. EFB particles size was in the range of 710-1000 µm that was used in this experiment [5].

### *2.2 Demineralization of EFB*

EFB demineralization process was performed using acid leaching to reduce the amount of alkaline and earth metal (AAEMS) contents. Approximately 100 g of EFB was located in a flask containing 500 mL of 1% nitric acid. EFB underwent leaching for about 60 and 120 minutes at room temperature. Then, it was placed in an ultrasonic bath at room temperature for sonication process. After completion of acid leaching, sample was filtered and dried at 105 ºC for 24 hours in order to constant the weight [15].

### *2.3 Torrefaction of demineralized EFB*

Torrefaction process of demineralized EFB was performed in a fixed-bed reactor and the temperature was set at 240 ºC. About 10 g of EFB was fed into a fixed-bed reactor. The reactor was purged with nitrogen at a flowrate of 150ml/min for 20 minutes. The temperature was increased from room temperature to 240 ºC and hold for 30 minutes. After torrefaction process was completed, the torrefied biomass was allowed to cool under a stream of nitrogen. The mass of torrefied biomass was taken out from the reactor and weighed. This process was repeated for several times to obtain a certain amount of torrefied EFB [15]. The sample of torrefied and demineralized EFB is referred as TDEFB.

### *2.4 Pelletization of raw and TDEFB*

Bio-oil was mixed with raw and TDEFB prior to pelletization. Approximately 0.25 g of bio-oil was measured and heated to 75-80 ºC before mixing with 4.75 g of raw and TDEFB. This indicated ratio of bio-oil addition to raw and TDEFB of 5 wt%. The ratio was varied from 0 – 20 %. Pelletization was performed using the single pellet machine. The pelletizing process was conducted using a mould which was able to accommodate approximately 5.0 g of material that originated from a mixture of 4.75 g of raw or TDEFB with approximately 5 wt% of bio-oil addition that were properly inserted into the mold until full. The bio-oil to raw or TDEFB ratio was varied from 0 – 20 wt%. Then, the mould was placed into the press machine and the pressure was applied to the mould in order to form the pellet. The pressure applied was 4 bar. After applying the pressure, the mould was left for 2 minutes to allow pellet formation. Finally, the pellet was taken out from the mold and ready for durability testing.

*2.5 Pellet Durability Testing*

The pellet durability properties of biomass was tested using a sieve shaker. This method was known as the tumbling process where the sample being tumbled for 10 minutes. The samples were placed at the top of stack sieve shaker for the durability test. The pellets were weighed for initial and final mass before and after tumbling process. The sieving duration was 10 minutes. The mass changes were recorded in order to calculate the durability percentage of the biomass pellet according to Equation 1. The samples used in this process were the raw and TDEFB. The durability experiments were conducted in duplicate.

$$\text{Durability} = 100 - \frac{initial\ mass\ (g) - final\ mass\ (g)}{initial\ mass\ (g)} \times 100\% \qquad \text{(Eq 1)}$$

*2.6 Proximate analysis*

The proximate analysis of raw and TDEFB pellets were conducted according to American Standard of Testing Material (ASTM) method. The moisture content was determined following the ASTM E871-82, the volatile matter and ash content were evaluated based on ASTM E872-82 and ASTM D1102-84 respectively. The fixed carbon content was calculated by difference [11].

*2.7 Predicted Ultimate Analysis*

For elemental analysis, the component of Carbon (C), Hydrogen (H) and Oxygen (O) were determined by using correlation based on the Equation 2, 3 and 4 [16].

$$C = -35.9972 + 0.7698VM + 1.3269FC + 0.3250ASH \qquad \text{(Eq.2)}$$
$$H = 55.3678 - 0.4830VM - 0.5319FC - 0.5600ASH \qquad \text{(Eq.3)}$$
$$O = 223.6805 - 1.7226VM - 2.2296FC - 2.2463ASH \qquad \text{(Eq.4)}$$

The value of volatile matter (VM), fixed carbon (FC) and ash (ASH) were taken from the proximate analysis.

*2.8 Predicted High Heating Value*

The heating value also known as calorific value and it was calculated based on the correlation in Equation 5 [17].

$$HHV = 0.3536FC + 0.1559VM - 0.0078ASH \qquad \text{(Eq.5)}$$

The value of fixed carbon (FC), volatile matter (VM) and ash (ASH) were taken from the proximate analysis.

*2.9 Fourier Transform Infrared (FTIR) Spectroscopy*

The raw and TDEFB pellets were analysed with FTIR spectrometer at a wave numbers in the range of 4000 to 600 $cm^{-1}$. The samples were prepared using potassium bromide (KBr) at 3:1 ratio between KBr to sample.

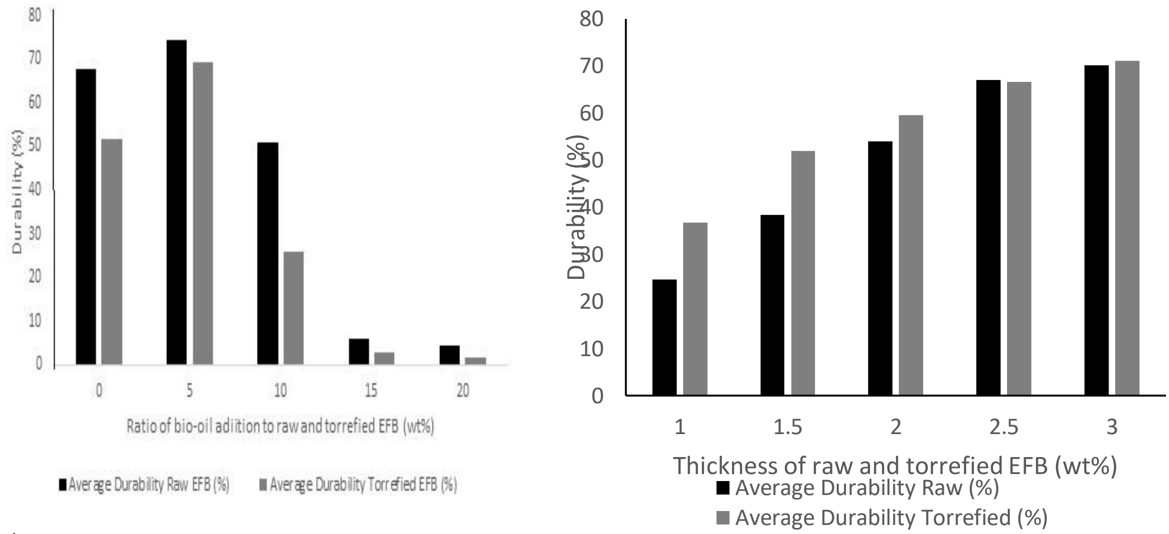
*2.10 Scanning Electron Microscope (SEM)*

The surface morphological analysis of raw and TDEFB pellets were performed using scanning electron microscope (SEM) (Hitachi TM 3000). The powder was mounted onto SEM stubs layer which was the layer with sticky tape. The stub was placed in sputter coater which was Auto line cutter, JEOL-1600 for five minutes for coating with platinum to give high quality of being reflective during scanning process.

## 3. Results and discussion

### *3.1 The Effect of Bio-Oil Addition and Thickness to Durabillity of Raw and TDEFB Pellets*

Figure 1(a) shows the durability (%) against the ratio of bio-oil addition to raw and TDEFB (wt%). When the ratio of bio-oil addition to raw and TDEFB was 0 wt%, the values of durability were approximately 69 % and 50 % for raw and TDEFB respectively. This indicated that in the absence of binder, the value of durability of TDEFB was lower compared to raw EFB. This could be due to the fact that TDEFB difficult to form strong pellets without the presence of binder compared to raw EFB. When the ratio of bio-oil addition to raw and TDEFB was increased to 5 wt%, the durability increased to approximately 74.5 % and 70.1 % respectively. When the ratio of bio-oil addition to raw and TDEFB was increased to further, the durability decreased. This indicated that the higher ratio of bio-oil addition to raw and TDEFB had resulted in the reduction of durability of the pellets. Thus it may affect the strength of the pellets negatively. Hence, it is determined that the highest durability was achieved at 5 wt % ratio of bio-oil addition to raw and TDEFB. Therefore, for the subsequent studies, this particular 5 wt% ratio of bio-oil addition to raw and TDEFB was selected to be considered for the next analysis which was the thickness of the raw and TDEFB pellets. From Figure 1(b), it is evident that when the thickness of pellet increased, the durability percentage increased as well. Therefore, there is a linear correlation between the thickness of raw and torrefied EFB pellets and its durability. It is determined that when the thickness of raw and torrefied EFB pellets was 3 cm, its durability was at its maximum which were 70.1 % and 71.1 % respectively.




(a) (b)

**Figure 1.** The durability (%) against ratio of bio-oil addition to raw and TDEFB (wt%)(a) and thickness of raw and TDEFB pellets (b)

### *3.2 Proximate Analysis*

The results for the proximate analysis of raw and TDEFB pellets are as shown in Table 1.

**Table 1**.Proximate analysis of raw and torrefied EFB pellets.

| Ratio of bio-oil addition to REFB and TDEFB pellets (wt %) | Proximate Analysis Moisture Content (%) | | Volatile Matter | | Ash Content (%) | | Fixed Carbon | |
|---|---|---|---|---|---|---|---|---|
| | REFB | TEFB | REFB | TEFB | REFB | TEFB | REFB | TEFB |
| 0 | 8.476 | 5.366 | 74.832 | 81.762 | 0.805 | 1.288 | 15.887 | 11.584 |
| 5 | 9.201 | 6.394 | 75.932 | 82.355 | 0.802 | 1.592 | 14.074 | 9.659 |
| 10 | 10.714 | 6.682 | 77.288 | 82.833 | 0.939 | 1.935 | 11.059 | 8.347 |
| 15 | 11.268 | 7.501 | 77.703 | 83.455 | 0.977 | 2.138 | 10.052 | 6.906 |
| 20 | 11.680 | 8.021 | 78.571 | 84.082 | 1.038 | 2.597 | 8.711 | 5.300 |

From Table 1, raw EFB pellet has higher moisture content compared to TDEFB pellet. This is in good agreement with Sirajuddin et al [18]. It is suggested that torrefaction has successfully removed physisorbed water. In this case, the moisture content increased with increasing ratio of bio-oil addition to raw and TDEFB since bio-oil itself contained water. Therefore, it contributed to the slight increment of moisture content. Generally, the moisture, volatile matter and ash content for both raw and TDEFB pellets increased but the fixed carbon decreased when the ratio of bio-oil to biomass was increased from 0 to 20 wt%. The volatile matter for both raw and torrefied EFB pellets showed an increasing trend upon increasing the bio-oil to biomass ratio because the bio-oil itself originated from EFB. Therefore, it contained certain complex organic molecules that directly contributed to the volatile matter. In terms of values, the TD EFB pellet had higher volatile matter content. Therefore, it is expected that TDEFB pellet has a better ignition property during combustion process compared to raw EFB pellets.

### *3.3 Predicted Ultimate Analysis and HHV content*

The results for predicted ultimate analysis of raw and TDEFB pellets are shown in Table 2. Generally, the carbon content decreased at the expense of increasing hydrogen and oxygen contents when the ratio of bio-oil addition was increased from 0-20 wt%. This was due to the contribution of bio-oil which has highly oxygenated compounds. From Table 2, the values of HHV for raw and TDEFB pellets decreased from 16.83 to 14.96 MJ/kg and from 17.28 to 15.32 MJ/kg when the ratio of bio-oil addition was increased from 0-20 wt%. The bio-oil itself has a lower calorific value, thus the use of bio-oil in a mixture had resulted in a decreasing HHV.

### *3.4 Morphological Analysis of Raw and TDEFB Pellet*

Figure 2 shows SEM pictures of raw and torrefied TDEFB pellets. At 30 and 200X magnification, the raw EFB pellet is fibrous with the absence of pores (Figure 2 (a) and (b)). However, in Figure 2(d) the presence of pores are observed in 200X magnification of the torrefied EFB pellets [11].

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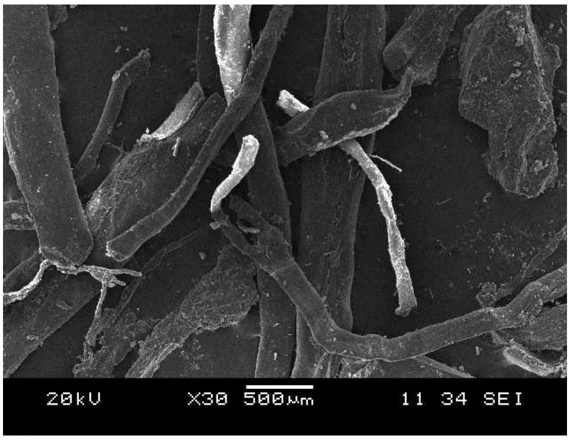
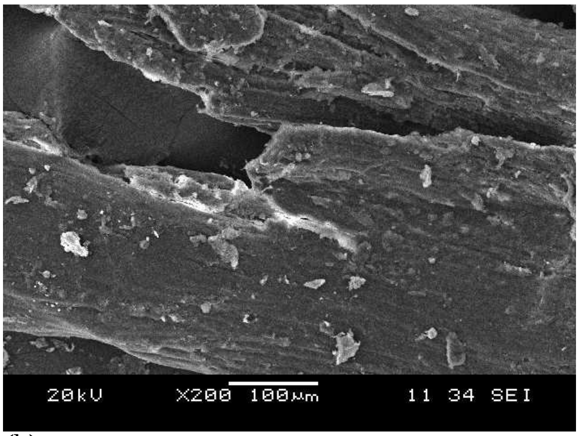
**Table 2**.Predicted Carbon, Hydrogen and Oxygen and predicted HHV contents for Raw and Torrefied EFB.

| Ratio of bio-oil addition to R and T pellets (wt %) | Carbon (%) | | Hydrogen (%) | | Oxygen (%) | | HHV | |
|---|---|---|---|---|---|---|---|---|
| | R | T | R | T | R | T | R | T |
| 0 | 42.95 | 42.73 | 10.32 | 8.99 | 57.54 | 54.12 | 16.83 | 17.28 |
| 5 | 41.39 | 40.73 | 10.76 | 9.56 | 59.70 | 56.70 | 16.24 | 16.81 |
| 10 | 38.48 | 39.47 | 11.63 | 9.84 | 63.78 | 58.04 | 15.85 | 15.95 |
| 15 | 37.47 | 38.10 | 11.94 | 10.19 | 65.22 | 59.72 | 15.44 | 15.66 |
| 20 | 36.38 | 36.61 | 12.20 | 10.48 | 61.038 | 61.19 | 14.96 | 15.32 |

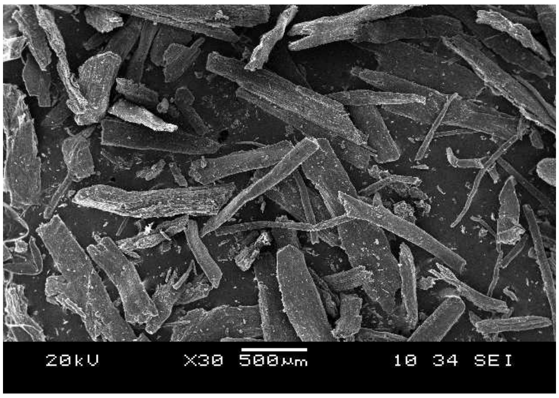
R= raw EFB. T= TDEFB



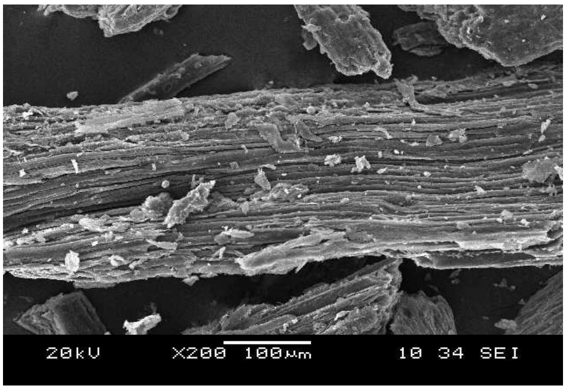
(a)



(b)



(c)



(d)

**Figure 2.** SEM image of raw and TDEFB (a) magnification of raw EFB at 30X, (b) magnification of raw EFB at 200X, (c) magnification of TDEFB at 30X, (d) magnification of

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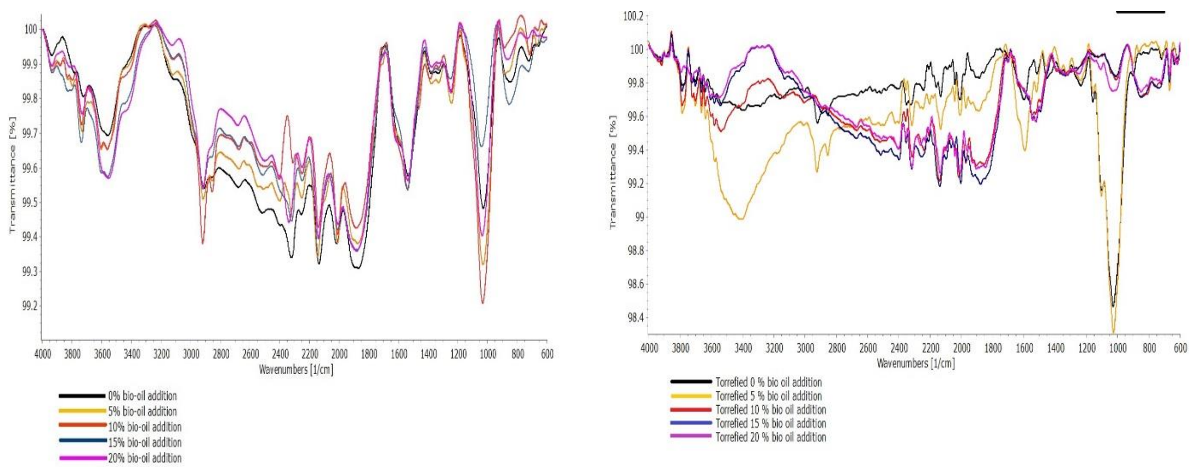
TDEFB at 200X.

*3.5 FT-IR Analysis*

Figure 3(a) and (b) show the existence of functional groups in raw and TDEFB pellets respectively at different ratio of bio-oil to raw and TDEFB pellets. The broad peak at wavenumber of 3420-3435 $cm^{-1}$ was assigned to O-H stretching that became broader when the ratio of bio-oil addition to raw and TDEFB was increased from 5-20 wt% [15, 19]. This was mainly due to the contribution of water molecules existed in bio-oil. Bio-oil is known for having high water content. When the ratio of bio-oil addition to raw and torrefied EFB was increased, the water content increased as well. This accounted for the increasing broad peak. Two sharp peaks were observed at wavenumber approximately 2930-2950 $cm^{-1}$ and 1430-1440 $cm^{-1}$ were assigned to the presence of C-H stretching frequency [19-21]. This showed that both raw and torrefied EFB pellets had alkane groups. The peaks at wavenumber of 1730-1750 $cm^{-1}$ and 1045-1055 $cm^{-1}$ were assignable to C=O stretching [19-21]. The presence of this bond recommended that the raw and EFB pellets contained carboxylic or aldehyde or ketone groups. The peak at approximately 1045-1055 $cm^{-1}$ increased in its intensity when the ratio of bio-oil addition to raw and TDEFB was increased which may be due to the existence of increasing amount of bio-oil that contained oxygenated compounds.




(a) (b)

**Figure 3.**FTIR spectra of (a) TDEFB (b) raw EFB.

## 4. Conclusion

In conclusion, pelletization of raw and torrefied EFB was conducted after mixing with bio-oil. The effects of ratio of bio-oil addition to raw and torrefied EFB pellets as well as its thickness were investigated towards its durability. It was identified that when the ratio of bio-oil addition of raw and torrefied EFB was 5 wt%, both torrefied and raw EFB pellets showed highest durability which were 69.27 and 74.46 % respectively. It was also determined that when the thickness of the pellets was 3 cm, the pellets showed the highest durability which were 70.12 and 71.12 % respectively. The proximate analysis of raw and torrefied EFB pellets were analyzed. It was indicated that the torrefied EFB pellet had higher volatile matter with higher carbon content than raw EFB pellet. The moisture content shows the highest value at torrefied EFB pellet compared to raw EFB pellet.

However, raw EFB pellet also has highest ash content than torrefied EFB. From the predicted ultimate analysis, it was indicated that the C content was at its maximum value of 42.95 and 42.73 % when the addition of bio-oil to biomass ratio was 0 wt%. The correlated high heating value showed that the highest HHV of 17.28 MJ/Kg was obtained when the addition of bio-oil to biomass ratio was 0 wt%. For the ultimate analysis, the carbon was highest at 0 % of bio-oil addition while the hydrogen and oxygen content was highest at 20 % of bio-oil addition to biomass for both raw and torrefied EFB pellet. Finally, the raw and torrefied EFB were analyzed by SEM and it was observed that particles binding force and the structure of lignin crack after torrefaction. From FTIR, it was showed that the presence of functional groups within the materials was identified that the high addition of bio-oil to biomass ratio had induced the presence of hydroxyl (O-H) group.

**References**

[1] Suksong W, Tukanghan W, Promnuan K , Kongjan, P, Reungsang, A, Insam H and O-Thong S 2020 *Bioresour. Technol* **296** 122304.

[2 ]Mohamed, A R, Hamzah, Z and Daud M Z M 2014 AIP Conference Proceedings 1605 1172 doi: 10.1063/1.4887756

[3] Kasim N N, Mohamed A R, Ishak M A M, Ahmad R, Nawawi W I, Ismail K and Salleh N H M 2018 *A*IP Conference Proceedings, 2013 020003

[4] Mohamed A R and Hamzah Z 2015 *React Kinet Mech Catal* **114** 529-545.

[5] Mohamed A R, Hamzah, Z, Daud, M Z M and Zakaria, Z 2013 Procedia Engineering **53** 185 – 191.

[6] Mohamed, A R, Hamzah, Z and Daud, MZM 2014 Advanced Materials Research, **925**, 115-119.

[7] Mohamed, A R, N Li1, K S A Sohaimi, N I Iberahim, N M Rohaizad and R Hamzah 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* 318 012019

[8]Kasim, N N, Ismail K, Ishak M A M, Ahmad R, Mohamed A R and Nawawi W I 2016 *IET Conf Pub* **2016 (CP688)**.

[9]Chen, D, Li, Y, Deng, M, Wang, J, Chen, M, Yan, B and Yuan, Q 2016 *Bioresour. Technol*. **214** 615–622.

[10]Kasim, N N, Mohamed A R, Ishak M A M, Ahmad R, Nawawi W I, Ali S N and Ismail K 2019 *J. of Therm. Anal. and Calorim*, **138(1)** 343-350.

[11] Awang A N, Mohamed A R, Mohd Salleh N H, Hoo P Y and Kasim N N 2019 *React. Kinet. Mech. Catal.***128** 663–680.

[12] Samuelsson R, Thyrel M, Sjöström M and Lestander T A 2009 *Fuel Process. Technol.* **90(9)** 1129–1134.

[13]Tumuluru J S 2018 *Carbon Resour. Convers.***1(1)** 44–54.

[14]Lu D, Tabil L G, Wang D, Wang G and Emami S 2014 *Biomass and Bioenergy* **69(17)** 287–296.

[15]Kasim N N, Ismail K, Mohamed A R, Ishak M A M, Ahmad R, Nawawi W I 2018 *Adv. Sci. Technol. Eng. Syst. J.* **3** 365–373

[16] Nhuchhen, Daya Ram. *Fuel* 180 (2016): 348-356.

[17] Parikh J, Channiwala S A and Ghosal G K 2005 *Fuel* 84(5) 487–494.

[18] Sirrajudin M S, Rasat M S M, Wahab R, Amini M H M, Mohamed M, Hashmi M I, Moktar J, Ibrahim M A 2016 *ARPN J. Eng. Appl. Sci.***11 (9)** 6122–6127.

[19] Ahmad R, Ishak M A M, Ismail K and Kasim N N 2018 *Adv. in Sc. Technol. and Eng. Sys. J.* **3(5)** 166-170.

[20] Ahmad R, Hamidin N, Ali U F M and Abidin C Z A 2014 *J. of Mech. Eng. and Sc.* **7** 1134-1140.

[21] Ahmad R, Ishak M A M, Ismail K and Kasim N N 2019 *Energy* 167 538-547.

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