Penerbit Akademia Baru

Journal of Advanced Research in Fluid Mechanics and Thermal Sciences

Journal homepage: www.akademiabaru.com/arfmts.html
ISSN: 2289-7879

Experimental Investigation on Water Washing and Decomposition Behaviour for Empty Fruit Bunch

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ARTICLE INFO

Article history:
Received 13 February 2019
Received in revised form 21 May 2019
Accepted 2 June 2019
Available online 19 July 2019

ABSTRACT

A series of experiment was performed at various operating conditions (stirring speed, biomass-to-water ratio and duration) to obtain the optimum condition for washing empty fruit bunch (EFB). The optimum condition for water washing pre-treatment was determined based on the level of ash content reduction. The washed EFB was also compared with unwashed EFB, Huangling coal (Chinese type coal) and international benchmark. The results show that stirring speed of 180rpm, biomass-to-water ratio of 1:20, and duration of 1 hour can be considered as the optimum condition with about 41% reduction if compared to the ash content of unwashed EFB. In addition, it was found that the nitrogen (N) and sulphur (S) contents decrease and the high heating value (HHV) slightly increases due to the removal of some alkali and alkaline earth materials (AAEMs) compound after washing pre-treatment. However, washed EFB was found to be less reactive after the removal of AAEMs compound. On top of that, the properties such as HHV, carbon (C), hydrogen (H), and oxygen (O) contents of washed EFB were considered slightly better than unwashed EFB.

Keywords:
Empty fruit bunch; water washing; optimum condition; ash content reduction

1. Introduction

Oil palm biomass waste in Malaysia is regarded as abundance. Based on the reported data in 2016, a considerable amount of oil palm biomass wastes has been generated about 35.19 million tonnes [1]. Empty fruit bunch (EFB) is the dominant waste among other types of palm biomass such as palm kernel shell (PKS) and mesocarp fibre (MSF) [2,3]. Interestingly, EFB can be possibly upgraded into essential energy sources for energy production. However, raw EFB has inferior properties (such as high ash content and low heating value) that cause a problematic to the energy production [4]. To address the issues, water washing pre-treatment is one of the positive efforts to upgrade fuel properties in the preliminary stage.

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Potassium (K), sodium (Na), magnesium (Mg) and others are component of alkali and alkaline earth materials compound (AAEMs) [5]. The AAEMs are the catalyst for ash formation during biomass combustion [6]. Interestingly, some of major AAEMs are reportedly to have higher solubility in water [4,7]. As previously reported, water washing can reduce about 20 to 80% of ash content from biomass [4,8–10]. For the case of water washing, duration, stirring, water medium and biomass-to-water ratio as well as temperature are the influential parameters that have significant changes to the ash reduction. For the case of duration, Abdullah, Sulaiman and Gerhausser [11] who firstly demonstrated lowest ash content for EFB of around 1% for 24 hours of water washing pre-treatment. In other study, Shariff, Aziz and Abdullah [9] who introduced the water washing in hot water (water temperature of about 90°C) have revealed that only 2 hours is required to achieve 1% of ash content for washed EFB. For the case of water medium, Rahman, Sulaiman and Abdullah [7] studied the ash reduction for PKS in various water medium such as tap, distilled and deionized water. They found that the washing pre-treatment in distilled water produces higher ash reduction of about 43% (decrease in ash content is from 5.97% to 3.4%) [7]. However, for the case of tap and deionized water, only 9 and 12% ash reduction were recorded, respectively [7]. Interestingly, if compared to the unwashed PKS, the high heating value for washed PKS increases about 24% (for the case of distilled water), 6% (for the case of deionized water) and 0.5% (for the case of tap water) due to reduction of ash content [7]. In other study, Nurdiawati *et al.,* [12] have concluded that the effect of higher ratio of biomass-to-water is dominant due to the decrease of K concentration (decreases from 32 to 1.6g/kg) for the case of washed hydrotreated EFB when the ratio is increased from 1:5 to 1:50. For the case of stirring, Pattiya, Chaow-u-thai and Rittidech [13] have revealed that the stirring effect has insignificant effect on ash reduction due to the relatively small changes in ash content after washing process of 24 hours. In the combustion perspective, water washing has showed positive reflection due to the reduction of nitrogen (N) and sulphur (S) content [14].

Literature reviews indicate that water washing can be practically employed as a technique to reduce the ash-related problem and upgrading biomass fuel properties at the preliminary stage. However, comprehensive studies on the optimum condition for water washing pre-treatment of pulverized oil palm biomass is still lacking for simultaneous consideration of continuous stirring, biomass-to-water ratio, duration. Besides, it can be said that the existing studies on water washing pre-treatment of oil palm biomass used longer duration (i.e 2 to 24 hours) and bigger biomass-to-water ratio for washing, ranging 1:20 to 1:70. On top of that, none of the previous researchers has considered number of important parameters simultaneously in their study such as continuous stirring, duration and biomass-to-water ratio with hot water (90fig) washing to investigate the optimum condition for water washing pre-treatment. In the previous studies, the purpose of stirring speed such as 250 rpm, 360 rpm and 650 rpm during washing pre-treatment was for the purpose of homogeneity [4,5,14], therefore has not been comprehensively studied. For the case of hot water, temperature of 90°C was only employed to the large particle size of EFB ranged from 5 to 8cm [9] while EFB with particle size ranged from 0.149mm to 4mm were water washed at 25 to 55°C [4]. In other cases, sufficiently low biomass-to-water ratio of 1:5 was employed to the hydrothermal treatment of EFB [12]. Meanwhile, the aim of the present study is to investigate the optimum condition for water washing of pulverized EFB with particle size ranging from 500 to 700µm at various stirring speeds (180, 360 and 540rpm), washing duration (30, 60 and 120minutes) and biomass-to-water ratio (1:10 and 1:20). Here, the optimum condition was chosen based on the maximum ash content reduction. Further, the performance of washed pulverized EFB was compared with that of previous studies, coal and international benchmark. The results of proximate, ultimate and other fuel properties are presented and discussed comprehensively in the following sections.

2. Material and Methods

2.1 Material

Empty fruit bunch (EFB) was collected from a local oil palm mill and sun-dried for three days. The EFB was pulverized by using a cutting mill (RETSCH type SM 100, Germany) and sieved by using 500 to 700µm screen. The raw EFB properties such as moisture, volatile matter, fixed carbon, ash content, carbon, hydrogen, sulphur, nitrogen and oxygen are listed in Table 1.

Table 1
The properties for raw unwashed EFB (UWEFB)

Analysis	UWEFB
Proximate analysis	
Moisture content	1.41±0.01
Volatile matter (wt, db%)	81.62±0.01
Ash content (wt, db%)	3.98±0.06
Fixed carbon*(wt, db%)	14.43±0.02
Ultimate analysis	
C (wt, db%)	46.35±0.14
H (wt, db%)	6.26±0.27
N (wt, db%)	0.81±0.01
S (wt, db%)	0.15 ±0.01
O* (wt, db%)	42.48±0.21
H:C	0.14±0.01
O:C	0.92±0.00
High heating value, HHV (MJ/kg)	15.16±0.33

2.2 Experimental Procedure

A series of tests were experimentally conducted with various experimental conditions as listed in Table 2. For the case of water washing pre-treatment, the method introduced by Lam *et al.,* [4] was implemented in this study. The experimental setup for water washing is shown in Figure 1. In this study, the biomass-to-water ratio of 1:10 was firstly performed. About 0.2L of distilled water was poured into 1L of beaker and placed inside water bath. Then the distilled water was stirred by using magnetic stir bar and heated on a digital hot plate magnetic stirrer (Thermo Scientific, SP88857107, Hot Plate Stirrer Cimarec+ Series, Canada) until stable at designated operating conditions. About 20g of pulverized EFB was then loaded in the beaker and beaker was covered by metal sheath lid. The temperature of heated water was continuously monitored throughout the process. The speed and temperature were set to zero after reaching the desired operating time and allowed to cool at ambient condition. The insoluble sample (sediment) was vacuum filtered and rinse about three times during the filtration process. Finally, the filtered sample (washed EFB) was dried in an oven for 24 hours at 105°C. The similar processes were repeated for the case of biomass-to-water ratio at 1:20. The washed EFB was characterized based on proximate analysis and the optimum conditions were selected based on the ash content reduction.

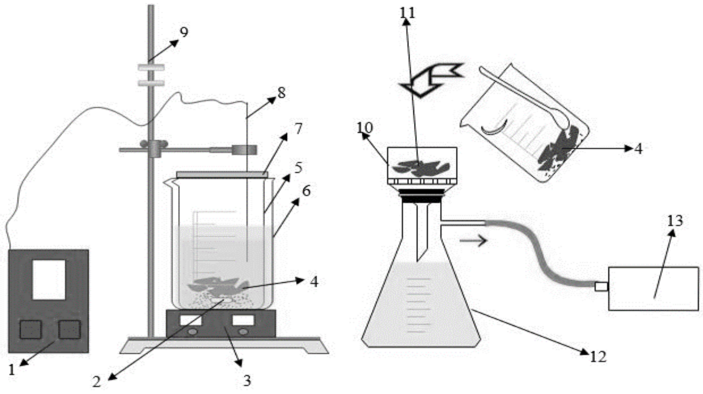


Fig. 1. (a) Water washing pre-treatment experimental set-up and (b) filtration process: (1) PID temperature controller; (2) magnetic stirrer bar; (3) magnetic hot plate; (4) sediment; (5) beaker; (6) water bath; (7) metal sheath lid; (8) K-type thermocouple; (9) retort stand; (10) Büchner funnel; (11) residue; (12) conical flask; (13) vacuum pump

Table 2
Various conditions during water washing pre-treatment of EFB

Biomass	Weight B:W (g)	Stirring speed (rpm)	Time (minute)	Temp (°C)	B: W ratio
EFB	B=20g; W=200g	180	120	90	1:10
			60		
			30		
		360	120		
			60		
			30		
		540	120		
			60		
			30		
EFB	B=20g; W=400g	180	120	90	1:20
			60		
			30		
		360	120		
			60		
			30		
		540	120		
			60		
			30		

B: biomass; W: water

2.3 Product Analysis

The proximate analysis such as moisture content, volatile matter content, and ash content for unwashed and washed EFB were determined in accordance to the EN ISO 18134-2:2015 [15], EN ISO 18123:2015 [16], and EN ISO 18122:2015 [17], respectively. Elemental analysis was performed by using CHNS analyser (ELEMENTAR, Germany) for the case of unwashed and washed EFB. Meanwhile, the heating value of unwashed and washed EFB were determined by using IKA calorimeter system, model C2000. The decomposition study of unwashed and washed EFB were separately performed based on thermogravimetric/derivative thermogravimetric analysis by using PerkinElmer Thermogravimetric Analyzer with DSC (8500). 5mg of sample were placed inside an alumina crucible and heated up to 900°C with slow heating rate of about 5°C/min in a 20 mL/min of nitrogen. In this study, the TG/DTG was repeated in triplicate.

3. Results and Discussion

3.1 Effect of Duration

Figure 2 shows the results of ash content for unwashed and washed EFB at different conditions. The summary of labels for unwashed and washed EFB is shown in Table 3. As shown by Figure 2, the ash content for unwashed EFB was recorded 3.98±0.06%. Interestingly, improvement in terms of ash reduction was observed for the case of washed EFB at when the washing duration is increased. For the case of WEFB-3 to WEB-1, the ash content was recorded of about 2.83±0.06 to 2.19±0.00% (ash reduction of about 29 to 45% with respect to ash content of unwashed EFB), followed by WEFB-6 to WEFB-4 with 2.71±0.08 to 2.33±0.04% (ash reduction of about 32 to 41%), and 2.99±0.04 to 2.46±0.00% (ash reduction of about 25 to 38%) for the case of WEFB-9 to WEFB-7. Similar inclinations were also observed for WEFB-12 to WEFB-10 with 2.89±0.02 to 2.69±0.03% (ash reduction of about 27 to 32%), WEFB-15 to WEFB-13 of about 3.57±0.03 to 3.30±0.07% (ash content reduction of about 10 to 17%), and 3.68±0.06 to 3.10±0.08% (ash content reduction of about 8 to 22%) for the WEFB-18 to WEFB-16. Based on these findings, it can be said that the longer duration contributes to aggressive diffusion of AAEMs compound in water [4,9]. Based on this result, it can be considered that 60 minutes duration is sufficient for ash content reduction of about 41%. This condition was taken after considering the energy consumption effect as well as the insignificant difference in terms of ash content reduction at 120 minutes with about 45%.

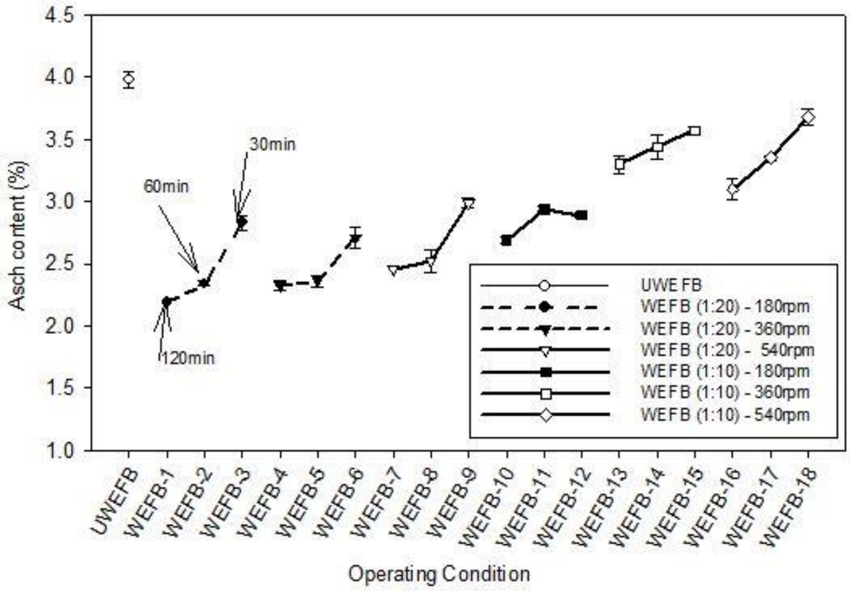


Fig. 2. Ash content at different operating conditions

Table 3
Summary of labels for unwashed and washed EFB at designated operating conditions

Conditions		**Label**
	Unwashed EFB	UWEFB
Washed	1:20, 180 rpm, 2 hr, 90°C	WEFB-1
	1:20, 180 rpm, 1 hr, 90°C	WEFB-2
	1:20, 180 rpm, 30 min, 90°C	WEFB-3
	1:20, 360 rpm, 2hr, 90°C	WEFB-4
	1:20, 360 rpm, 1 hr, 90°C	WEFB-5
	1:20, 360 rpm, 30 min, 90°C	WEFB-6
	1:20, 540 rpm, 2 hr, 90°C	WEFB-7
	1:20, 540 rpm, 1 hr, 90°C	WEFB-8
	1:20, 540 rpm, 30 min, 90°C	WEFB-9
	1:10, 180 rpm, 2 hr, 90°C	WEFB-10
	1:10, 180 rpm, 1 hr, 90°C	WEFB-11
	1:10, 180 rpm, 30 min, 90°C	WEFB-12
	1:10, 360 rpm, 2hr, 90°C	WEFB-13
	1:10, 360 rpm, 1 hr, 90°C	WEFB-14
	1:10, 360 rpm, 30 min, 90°C	WEFB-15
	1:10, 540 rpm, 2 hr, 90°C	WEFB-16
	1:10, 540 rpm, 1 hr, 90°C	WEFB-17
	1:10, 540 rpm, 30 min, 90°C	WEFB-18

U: unwashed; W: washed

3.2 Effect of Biomass-to-Water Ratios

Biomass-to-water ratio can be one of the influential parameters in water washing pre-treatment. The ash content at ratios of 1:20 and 1:10 are also shown in Figure 2. Based on the figure, it is noticeable that sufficiently low ash content was obtained at ratio of 1:20, that was within the range of 2.19±0.00 to 2.99±0.04% if compared to that at ratio of 1:10, that was within the range of 2.69±0.03 to 3.68±0.06%. These findings reveal that ratio of 1:20 has bigger capacity and hence, allow more dispersion of AAEMs compound [4,12]. For the case of 1:10, the ash content reduction is lower due to lower capacity to absorb large volume of finest AAEMs. In this case, the existence of sufficiently large volume of finest AAEMs is possibly due to the grinding effect that has been performed during preparation stage [18].

3.3 Effect of Stirring

In the present study, the effect of stirring was investigated for various speeds (180 to 540rpm). Based on the results as shown by Figure 2, it can be clearly observed that the ash content increases when the stirring speed is increased from 180 to 540rpm. This is supposed mainly due to finest AAEMs remained intact with particles of washed sample at higher speed. The authors believe that this finding has not been reported in the previous studies. In contrast to the present study, the stirring effect only has been reported for the use of homogenous mixing [4] and they have found that stirring effect increases removal rate of AAEMs compound [4,14]. Based on these contradict findings, it seems that stirring speed and biomass-to-water ratio are interdependence parameters.

3.4 Proximate Analysis

The proximate analysis results for the WEFB-1 to -9 and WEFB-10 to -18 are shown by Figure 3. Based on the figure, it can be clearly observed that the volatile matter becomes lower when ash content is higher. For the case of ratio of 1:10 and unwashed EFB, the volatile matter is recorded lower owing to higher ash content of about 2.69 ±0.03 to 3.68 ±0.06% and 3.98±0.06%, respectively. In contrast, ratio of 1:20 gives higher volatile matter due to lower ash content that are only 2.19±0.00 to 2.99±0.04%. These findings are also agreed well with findings by Yu *et al.,* [14]. Based on these findings, WEFB-1 and WEFB-2 demonstrate promising volatile matter among other conditions with 84.65±0.10 and 84.29±0.04%, respectively. For the case of moisture content obtained in the present study, WEFB-1 to WEFB-9 gives the range of 1.2 to 1.5% while 1.5 to 1.6% for the case of WEFB-10 to WEFB-18. Based on the figure, in terms of fixed carbon content, it was found that WEFB-1 to -18 gives the range of 13.16±0.10 to 15.19±0.12% (for the case of ratio of 1:20) and 14.87±0.07 to 16.12±0.09% (for the case of ratio of 1:10). These findings have revealed that the washed EFB has a suitable properties for higher formation of char during torrefaction due to a considerable amount of fixed carbon [3].

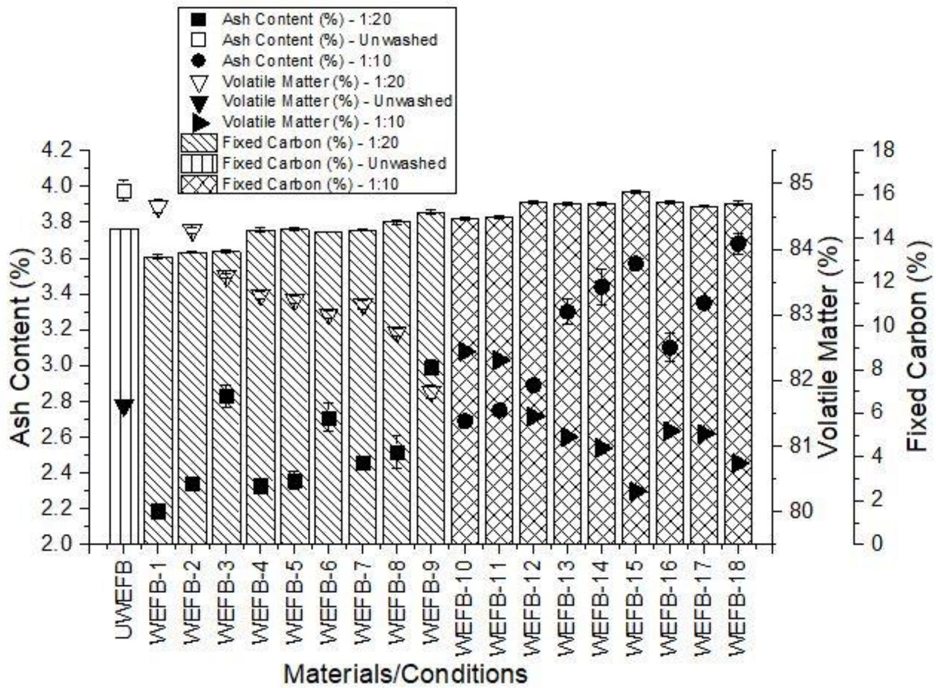


Fig. 3. Proximate analysis for unwashed and washed EFB

3.5 Fuel Properties of Unwashed and Washed EFB

The properties of EFB washed at an optimum condition (ratio of 1:20, duration of 1 hour, temperature of 90°C and speed of 180rpm) were compared with those of unwashed EFB, Huangling coal as well as international benchmark.

Based on Table 4, it can be observed that the ash content for washed EFB are significantly improved with only 2.34±0.03% if compared to that for unwashed EFB (3.98±0.06%), Huangling coal (18.65%) and international benchmark (≤5%). Meanwhile, in terms of volatile matter, washed EFB has almost same volatile matter content (82.31±0.05%) with that of unwashed EFB (81.62±0.01%) and higher than that of Huangling coal (30.55%). These findings reveal that the water washing offers positive outcomes in removing AAEMs compound and reducing the catalytic effect from EFB in the ash formation.

Table 4
Comparison on fuel properties of unwashed and washed EFB with Huangling coal as well as international benchmark

Analysis	UWEFB	WEFB	Huangling coal[a]	International Benchmark
Proximate analysis				
Moisture content	1.41±0.01	1.31±0.10	3.45	≤10[b]
Volatile matter (wt, db%)	81.62±0.01	82.31±0.05	30.55	-
Ash content (wt, db%)	3.98±0.06	2.34±0.03	18.65	≤5[c]
Fixed carbon* (wt, db%)	14.43±0.02	14.94±0.06	47.35	-
Ultimate analysis				
C (wt, db%)	46.35±0.14	47.15±0.04	77.82	-
H (wt, db%)	6.26±0.27	6.52 ±0.02	4.85	-
N (wt, db%)	0.81±0.01	0.65±0.01	3.48	≤0.5[d]
S (wt, db%)	0.15 ±0.01	0.07±0.02	-	≤0.03[e]
O* (wt, db%)	42.48±0.21	44.04±1.20	13.53	-
H:C	1.61	1.65	0.74	-
O:C	0.69	0.70	0.13	-
High heating value, HHV (MJ/kg)	15.16±0.33	15.38±0.30	27.40	≥20[f]

C: Carbon; H: Hydrogen; N: Nitrogen; S: Sulphur; O: Oxygen
*Calculated by difference ([a] Du, Yang and Fan [19], [b]EN14774-1:2009 [20], [c]EN ISO 18122:2015 [17], [d]EN ISO 16948:2015 [21], [e]EN ISO16994:2015 [22], [f]EN ISO18125 [23])

In terms of ultimate analysis, C, H and O contents for washed EFB was observed to increase very slightly, from 46.35±0.14 to 47.15±0.04, 6.26±0.27 to 6.52 ±0.02, and 42.48±0.21 to 44.04±1.20%, respectively. Based on the Van Krevelen diagram as shown by Figure 4, the atomic H:C and O:C ratios for the case of washed EFB was found higher than that of Huangling coal.

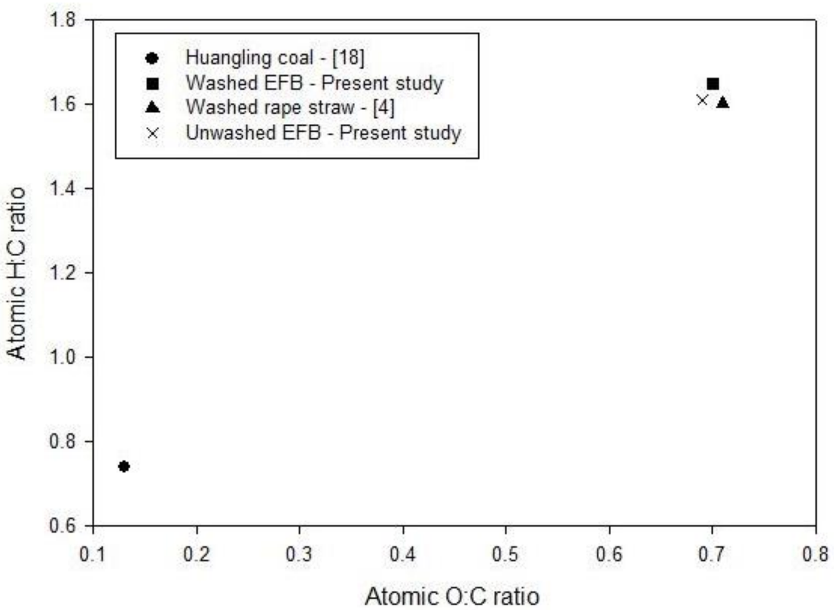


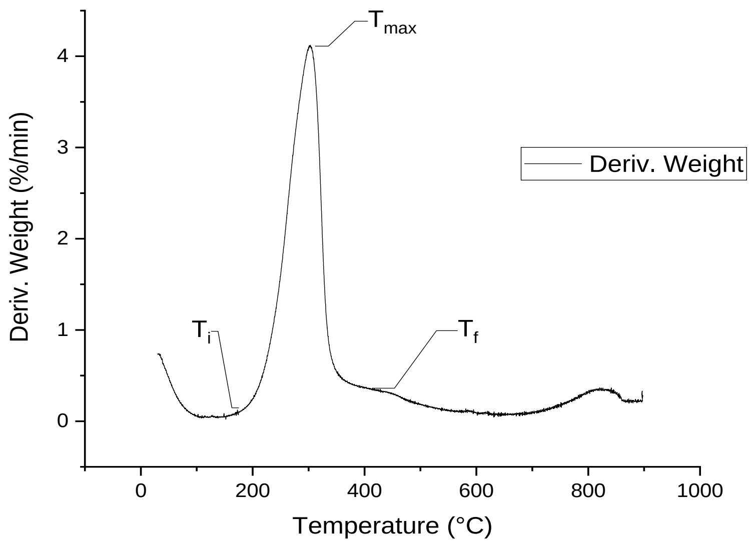
Fig. 4. The Van Krevelen diagram for unwashed EFB (present study), washed EFB (present study), Huangling coal [19] and washed rape straw [5]

The atomic H:C and O:C ratios for washed EFB can be improved with further thermochemical conversion. The N and S contents for washed EFB reduced from 0.81±0.01 to 0.65±0.01 and 0.73±0.02 to 0.52±0.02%, respectively, thus proving that the water washing pre-treatment can contribute to the production of cleaner-fuel. However, the N and S contents for washed EFB was still higher than the international benchmark.

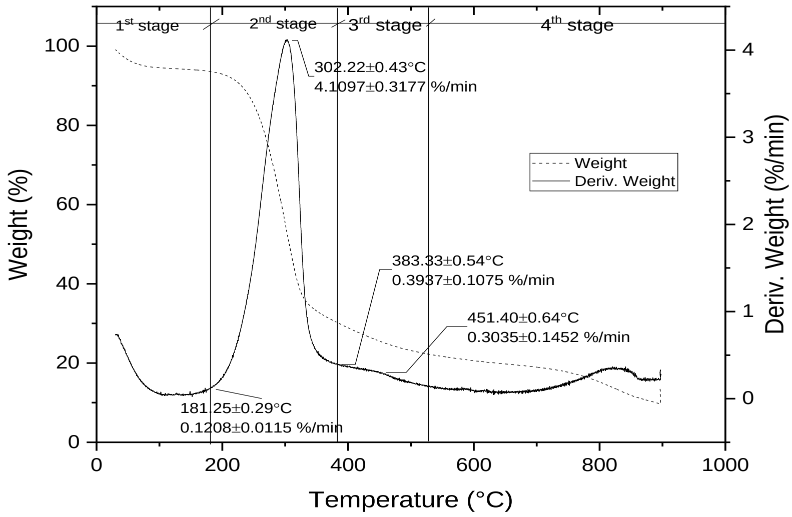
The high heating value for washed EFB was found to increase slightly from 15.16±0.33 to 15.38±0.30MJ/kg, thus can be regarded as uncompetitive if compared to the Huangling coal and international benchmark.

3.6 Thermogravimetric (TG) and Differential Thermogravimetry (DTG) Analysis

Biomass decomposition can be illustrated through TG and DTG analysis. However, the decomposition of each biomass is different due to the unique chemical structure [24]. Based on the other studies, typical oil palm biomass waste decomposed within the range of 150 to 380°C [25,26], 226.85 to 690°C for the wheat straw [27] and for *Cupressus lusitanica* (woody waste) decomposition occurred within the range of 172.1 to 448.7°C [28]. Generally, decomposition of hemicellulose, cellulose and lignin occur within the decomposition range; hemicellulose (150 to 350°C), cellulose (275 to 350°C) and lignin (250 to 550°C) [29]. From the TG and DTG curves, biomass decomposition can be grouped into three to four stages, varies upon type of biomass [24,28]. As referring to the TG and DTG curves in this study (in Figure 5(b)-(e)), biomass decomposition stages consist of release of mild volatile or mild decomposition (first stage), decomposition (second stage), mild decomposition due to excessive lignin in biomass (third stage) and biomass residues at stage four [24]. From the DTG curve as shown by Figure 5(a), specific parameters can be obtained such as the initial temperature (T_i) that also corresponds to the beginning of decomposition or at which the hemicellulose degradation occurs, T_m corresponds to the temperature that reflects the maximum decomposition and T_f is the final temperature of decomposition that represents point at which only lignin degradation still occurs [24,28].



(a)



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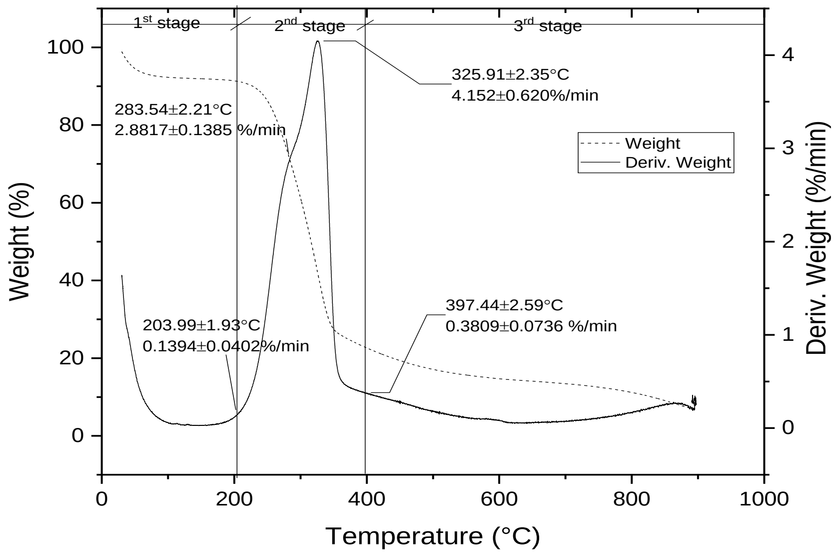

(c)

Fig. 5. Decomposition parameters (a), and decomposition process for the case of unwashed EFB (b), and washed EFB (c), (summarize in Table 5)

Table 5
Summary on specific temperature of different species based on DTG curves

Species/materials	Initial temperature, T_i (°C)	Final temperature, T_f (°C)	Maximum decomposition temperature, T_m (°C)
Unwashed EFB	181.25±0.29	383.33±0.54	302.22±0.43
Washed EFB	203.99±1.93	397.44±2.59	325.91±2.35

For the case of species in the present study, it can be observed that the temperature range for the unwashed EFB decomposition is from 181.25±0.29 to 383.33±0.54°C (with temperature of maximum decomposition occurs at 302.22±0.43°C, see Figure 5(b). But, after washing pre-treatment, the range is from 203.99±1.93 to 397.44±2.59°C (with maximum temperature occurs at 325.91±2.35°C, see Figure 5(c). Based on the results, it is revealed that catalytic effect is more pronounced for the case of unwashed EFB if compared to the case of washed EFB. In addition, the initial, final and maximum decomposition temperatures were clearly shifted for the case of washed

EFB due to washing effect that reduces the catalytic effect of the biomass. These scenarios were also found in a few previous studies [11,30,31] regardless of techniques, water medium, and type of species. In this study, it can be clearly observed that no shoulder (correspond to the hemicellulose degradation) was recorded for the case of unwashed EFB (see Figure 5(b)). Nevertheless, shoulder was recorded for the case of washed EFB (see Figure 5(c)), that occurs at temperature of 283.54±2.21°C. These findings reveal that the hemicellulose in unwashed EFB was decomposed intensively due the catalytic effect if compared to the case of washed EFB, in which the hemicellulose was slowly decomposed. The no shoulder trends were also observed by Acharya, Pradhan and Dutta [32] due to high decomposition intensity of hemicellulose in miscanthus and wheat straw biomass. In terms of cellulose degradation, it is observed to start decompose at 302.22±0.43°C (for unwashed EFB, see Figure 5(b)) and 325.91±2.35°C (for washed EFB, see Figure 5(c)). The lignin degradation for the unwashed EFB (see Figure 5(b)) was observed to start at 383.33±0.54°C while mild decomposition occurs at 451.40±0.64°C (occurs in third stage of decomposition). This occurrence could be possibly due to the decomposition of excessive lignin [24]. In contrast to that, the lignin for the case of washed EFB only starts to decompose at 397.44±2.59°C.

Based on the figure, small peak (or bump) at temperature below 100°C for all cases (unwashed and washed EFB) demonstrates the release of moisture from biomass. This occurrence has been detected in both TG and DTG study for biomass [9,33]. In terms of maximum decomposition rate for EFB, it was found that the rate slightly increases after washing pre-treatment. The maximum decomposition rate for washed EFB and unwashed EFB are 4.15±0.62 %/min (at 325.91±2.35°C) and 4.11±0.32%/min (302.22±0.43°C), respectively. Similar trend was also observed by Deng *et al.,* [34].

4. Conclusions

An investigation on the optimum condition for water washing of oil palm biomass at various parameters has been performed. In the present study, the optimum condition is considered based on the ash content reduction. The optimum result was obtained when the washing pre-treatment was performed at biomass-to-water ratio of 1:20, duration of 1 hour, temperature of 90°C and speed of 180rpm. Meanwhile, the effect of stirring speed is found to be contradict with the results obtained in the previous study, thus implies the interdependence between stirring speed and biomass-to-water ratio. Overall, water washing pre-treatment offers positive impact towards cleaner-energy fuel subsequently improved oil palm biomass properties (i.e higher ash content reduction, and decreased N and S contents). However, water washing pre-treatment causes EFB to be less reactivity. It is believed that the overall properties of washed EFB can be improved when it is further treated with thermochemical conversion process.

Acknowledgement
The authors acknowledge the Ministry of Higher Education Malaysia and Universiti Teknologi Malaysia for giving cooperation and full support in this research activity. The authors wish to thank Research Management Center (RMC) for Tier 1 GUP grant (VOT No.: Q.J130000.2524.20H24) from Ministry of Higher Education Malaysia and Universiti Teknologi Malaysia.

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