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The Properties of the Washed Empty Fruit Bunches of Oil Palm

Nurhayati Abdullah* and Fauziah Sulaiman

School of Physics, Universiti Sains Malaysia, 11800 USM Pulau Pinang, Malaysia

*Corresponding author: nurhaya@usm.my

Abstract: *Water washing pre-treatment on empty fruit bunches (EFB) of oil palm was carried out for reducing some ash in the EFB to improve its quality prior to any thermal conversion process to yield better quality fuel. EFB was washed by varying some parameters such as the residence time and the amount of water used to study the effectiveness of washing with water. Total ash was reduced by about 24.9–70.3% in the EFB by various water washing pretreatments done in this work with an average ash content of 2.48 mf wt% achieved. The residence time would be expected to impact washing efficiency, which was described by the correlation between the percentage of ash reduction in the EFB and the incremental electrical conductivity (EC) of waste liquor. The use of tap water versus distilled water was also addressed for economic viability. Water washing pretreatment is an effective procedure in removing potassium and sodium in ash as they are proxies of ash in the EFB. Results showed that about 71% of potassium and 96% of sodium can be removed in ash. A thermogravimetric analysis (TGA) was performed under 100 ml min^{-1} nitrogen with a heating rate of 10ºC min^{-1} to investigate the thermal degradation characteristics of the unwashed and washed EFB.*

Keywords: Empty fruit bunches, fast pyrolysis, water washing pretreatment, bio-oil, washed EFB, thermogravimetric analysis

1. INTRODUCTION

The fundamental formation of potassium and sodium in ash influences the quality of organic yield since ash is a proxy indicator of potassium and sodium, as indicated in a study on straw and sugarcane bagasse.[1,2] Alkali metal content such as sodium (Na), potassium (K), magnesium (Mg), phosphorus (P) and calcium (Ca) are known as ash produced from the chemical breakdown from biomass fuel, by either the thermochemical or biochemical process.[3,4] It was found that the reduction in ash is clearly not proportional to the reduction in alkali metals. However, there is a correlation, and it can therefore be assumed that a given level of ash reduction will roughly correspond to a particular level of alkali metal reduction at least with water washing, as removal of alkali and earth alkali metals dominates the overall reduction in ash content.

Water washing pretreatment is one of the methods used to remove the ash content or other minerals from biomass. Ash varies in different forms of biomass. The ash content in biomass will give a significant effect on biomass energy conversion such as in the pyrolysis process. The major elements in ash compositions of the biomass including empty fruit bunches (EFBs) are listed in Table 1. Table 1 also shows that most of the biomass contained high amount of potassium (K).

Table 1: The ash composition of the biomass.[31]

Biomass	Major element (ppm)							
	Al	Ca	Fe	Mg	Na	K	P	Si
Bagasse	na	1518	125	6261	93	2682	284	17340
Coconut coir	148	477	187	532	1758	2438	47	2990
Coconut shell	73	1501	115	389	1243	1965	94	256
Coir pith	1653	3126	837	8095	10564	26283	1170	13050
Corn cob	na	182	24	1693	141	9366	445	9857
Corn stalks	1911	4686	518	5924	6463	32	2127	13400
Cotton gin waste	na	3737	746	4924	1298	7094	736	13000
Groundnut shell	3642	12970	1092	3547	467	17690	278	10960
Millet husk	na	6255	1020	11140	1427	3860	1267	150840
Rice husk	na	1793	533	1612	132	9061	337	220690
Rice straw	na	4722	205	6283	5106	5402	752	174510
Subabul wood	na	6025	614	1170	92	614	100	195
Wheat straw	2455	7666	132	4329	7861	28930	214	44440
EFB oil palm waste	na	14300	2900	1900	3300	86100	10600	19500

na: not available

The high ash content in biomass promotes secondary reaction of the thermochemical process, thus producing non-homogenous liquids.[5] However, as mentioned elsewhere, the EFB with ash content of less than about 3 mf wt% is capable of producing homogenous bio-oil by fast pyrolysis for petroleum application.[6] According to Fahmi et al.,[7] the pyrolysis oil quality can be improved by washing the high ash feedstock.

Potassium is a catalyst for the pyrolysis process and also serves as a dominant source of alkali in most of the biomass fuel.[8] According to Jones et al.,[9] thermogravimetric studies showed that potassium salts increase the gas and char yields and decrease the primary decomposition of tar products (liquid). Thus, potassium content should be decreased in biomass in order to obtain the highest yield in liquid products via fast pyrolysis process. The reactions in a thermochemical conversion process are mainly due to the presence of potassium.[10] Therefore, the correlation between the percentage of ash reduction and the reduction of potassium and sodium in ash for the washed feedstock is investigated in this work since potassium and sodium are the proxy of ash. The correlation between the percentage of ash reduction and the reduction of potassium and sodium is shown by each reduction. The reduction of potassium and sodium is measured by the Atomic Absorption Analysis method.

The pretreatment can be carried out by using either hot water or cold water to eliminate the ash content in biomass.[11] However, some researchers also use mild acid leaching in their washing treatment on biomass in order to remove all the ash elements.[2] Simple water washing pretreatment will remove a large amount of minerals from the biomass surface.[12] Some of these minerals are possibly due to their contact with soil during the harvest season or during transportation of the biomass itself.

Abdullah and Gerhauser conducted a study on the fast pyrolysis of washed and unwashed EFBs.[6] They used 100 g of feedstock for each washing pretreatment. All the washing experiments were performed at ambient temperature in Malaysia (26°C–28°C). They found that the most effective method to remove ash was soaking the feedstock of size 250–355 μm in 7 l of distilled water for 24 h. The ash content of the washed feedstock was 1.03 mf wt%.

Jenkins et al. studied the ash reduction in rice straw and wheat straw for reducing slagging and fouling in furnaces and other thermal conversion systems.[1] 100 g of whole straw samples which were hand-sprayed for 1 min with tap water and then submerged for 24 h in 7 l of distilled water. In the meantime, another 100 g of milled straw samples were flushed with 20 l of tap water and distilled water and 50 g of milled straw was flushed with 7 l of distilled water. They found that the flushing treatment was quite effective for wheat straw, yielding averages of 5.0 mf wt% and 4.2 mf wt% of ash for tap water and distilled water, respectively. They also found that the best ash removal using water was obtained by soaking the whole straw for 24 h in distilled water. Moreover, the effect of soaking was more effective in wheat straw, with the overall ash being reduced by almost 50% and the levels of chlorine, sodium, and potassium significantly reduced.

Di Blasi et al. investigated the degradation of the characteristics of straw and washed straw.[11] They carried out the water washing pretreatment because water washing affects the decomposition of straw. The straw was soaked and submerged in distilled water with 100 ml of water for 1 g of straw. The residence time of the experiment was about 2 h or 7200 s. They found that the ash content in the unwashed straw decreased to 2.20 mf wt% after washing pretreatment was performed on it.

From their investigation, hot water treatment is more effective than cold water treatment which required longer resident time or rain leaching where the ash content was lowered. Fahmi et al. studied the effect of alkali metals on combustion and pyrolysis of Lolium and Festuca grasses, switchgrass and willow.[13] All the grasses were subjected to varying washing conditions to investigate the effect of decreasing metal content in grasses. The objective of their work was to reduce the amount of minerals, which could catalyse the combustion or the gasification process. All the samples were subjected to a stirring method for 2 h at room temperature continuously, 24 h at room temperature, and 2 h at 60°C continuously to improve the leaching and minimize the hemicellulose hydrolysis. They used de-ionised water for the washing stage of the experiment.

Piyali Das et al. carried out a research on the influence of pretreatment for deashing sugarcane bagasse on pyrolysis products.[2] The washing experiments were aimed to find the maximum extraction of ash. The washing treatments used water leaching, acid leaching with Hydrochloric (HCl) solution and Hydrofluoric (HF) solution. A total of 12.5 g of sample and 150 ml of leachate were used for water treatment experiments and 25 g of sample was employed in 150 ml of acid leaching. For all the pretreatments, mixtures were leached and occasionally stirred for a predetermined time at 25°C. For the acid treatment, the samples were soaked in acid and filtered, and then washed with distilled water. They found that the treatment with HCl solution led to an apparent increase of the percentage of ash, which was attributed to the relatively high removal of other components in the bagasse and corresponded to the highest mass reduction of the biomass itself. However, pretreatment by water leaching has a moderate effect on ash removal.

Yang et al. examined the effect of minerals on biomass by using corn straw as a sample with different pretreatment methods.[14] The analysis of the pretreatment was analysed using a thermogravimetric analyser (TGA) and a Fourier transform infrared (FTIR) spectrometer. The samples were pretreated by washing process with water at 60°C and 0.5% nitric acid (HNO_3) solution. All the washing treatments were performed by soaking and churning each sample of 1 g in 100 ml solution for 12 h. After washing with different methods, the corn straw samples were filtered, and then dried at 105°C for 10 h.

For the acid washed corn straw, the samples were flushed with distilled water until they became neutral before they were filtered and dried at 105ºC for 10 h. The process has removed 55% of ash content in corn straw by acid washing and around 25%–35% by water washing. The removal of K was over 95% by both water and acid washing but the removal of Ca was only 78% by acid washing and almost zero by water washing. It showed that water washing almost removed most of the potassium content in the corn straw.

The palm oil industry is currently expanding rapidly and yields large amount of poorly utilised waste biomass. Fast pyrolysis of EFB is therefore currently an important subject for further investigations. Results for the production of bio-oil derived from unwashed EFB were presented elsewhere and it was found that in all cases, the liquid product separated into two phases presenting difficulties for fuel applications.[6,15,16] However, it was found that the maximum ash content of washed EFB that produced homogenous liquids is less than about 3 mf wt% by pyrolysis process.[6,15,16] The aim of this work is to investigate the water washing pretreatment on EFB in order to remove some ash, thus improving the quality of EFB which may be used to produce homogenous bio-oil in fast pyrolysis process for fuel applications.

2. EXPERIMENTAL

2.1 Preparation and Feedstock Properties

The EFB used in the experiments was obtained from a local palm oil industry in the northern part of Penang, Malaysia. Samples received were wet and in the form of whole bunches. These samples were taken after the sterilisation and stripping process of fresh fruit bunches in the palm oil mill. Key properties of EFB, both measured for this research and from the literature, are given in Table 2. The carbon and hydrogen contents are comparable to woody biomass, as is the measured heating value. The lowest HHV in the literature may be due to confusion between values quoted on a dry basis as opposed to a wet basis, a problem apparent elsewhere in the literature, for example, a value of 10 MJ kg^{-1} is quoted for dry oil palm matter, which is clearly too low for a ligno-cellulosic biomass on a dry basis – or [19] – values for wet FFB are used for dry FFB.[17,18]

The EFB constitutes a lignocellulose material which consists of chemical component of 57.8% cellulose, 21.2% hemicellulose and 22.8% lignin. The results showed that the EFB contain high volatile contents (76.85 mf wt%), very low nitrogen (2.18 mf wt%) and sulphur (0.92 mf wt%) contents with a high HHV (20.54 MJ kg^{-1}). The trace element of sulphur and nitrogen showed that the EFB is an environmentally friendly biomass. It was also found that the EFB had an ash content of 5.19 mf wt%. The high ash content implies that the high alkali metal especially potassium (K) and sodium (Na) are known to have an actively catalytic nature to promote secondary reaction of the pyrolysis process.[6,15,16] The high ash and potassium values are noteworthy, as it is well known that ash, and in particular potassium, lead to reduced liquid yields in fast pyrolysis.[20]

The proximate analysis was performed to determine the moisture, ash, volatiles and fixed carbon contents. The ASTM standard test methods for measuring moisture, volatile and ash contents are ASTM E871, E872-82 and National Renewable Energy Laboratory (NREL) Standard Analytical Method LAP005, respectively. The fixed carbon contents were calculated by finding the difference. Ultimate analysis was performed for the determination of basic elemental composition of the EFB using the combustion analysis method for carbon, hydrogen, nitrogen and sulphur. Oxygen however was again determined by the difference. The HHV of the EFB was determined by bomb calorific experiment according to the ASTM D2015 standard test method.

Table 1: Properties of EFB (mf wt%).[17,19]

Component /property	Literature values	Measured	Method
Cellulose	59.7, 38.1	57.8	–
Hemicellulose	22.1, 16.8	21.2	–
Lignin	18.1, 10.5	22.8	–
Elemental analysis			
Carbon	48.9, 48.8	46.36	Combustion analysis
Hydrogen	7.33, 6.3	6.44	
Nitrogen	0.7, 0.78	2.18	
Sulphur	0.68, 0.2	0.92	
Oxygen	40.2, 36.7	38.91	By difference
K	2.41, 2.24	–	Spectrometry
K_2O	3.08–3.65	–	–
Proximate analysis			
Moisture	–	4.68	ASTM E871
Volatiles	87.3, 75.7	76.85	ASTM E872
Ash	3.02, 4.3	5.19	NREL LAP005
Fixed carbon	9.6, 17	18.07	By difference
HHV (MJ/kg)	19.0, 17.86	20.54	Bomb calorimeter
LHV (MJ/kg)	17.2	–	–

2.2 Drying Process of EFB Waste

Initially, samples were dried using a conventional oven to obtain moisture content of less than 10 mf wt% as normally done in most laboratory experiments and commercial processes for thermal conversion technologies such as pyrolysis.[6,15,16] According to McKendry[3] and Cuiping et al.,[21] biomass fuel with low moisture content is more suitable for thermal conversion technology while biomass fuel with high moisture content is more suitable for biochemical processes such as fermentation. The EFB samples in the form of whole bunches were first dried at temperature of 105ºC for 24 h. Then, they were continually dried and monitored every hour in order to find the correlation between moisture content and the period of drying. The drying process continued until the weight of the whole bunches was stabilised in which the moisture content became less than 10 mf wt%. Then, the whole bunches were manually chopped and fed into the shredder in order to reduce the size to 1–3 cm before employing water washing experiments. For the analyses of the moisture and ash contents, the samples were ground to powder form.

2.3 The Pretreatment of Water Washing

The water washing pretreatment was carried out in order to reduce some of the ash in the EFB. The variables of the water washing pretreatment considered were the residence time of EFB, the amount of water for every 100 g of feedstock, and the methods of washing. For all experimental runs, tap water at ambient temperature of 25°C–28°C was used.

2.3.1 Residence time

Most of the results from previous studies showed that soaking the biomass for 24 h can contribute to the most reduction of ash content. According to Jenkins et al.,[1] the best ash removal using water at ambient conditions was obtained by soaking the biomass for 24 h in distilled water to facilitate ash removal easily. This is in agreement with Abdullah et al.[6], where they found that the ash content was reduced by almost 81% by soaking the feedstock for 24 h in distilled water. However, in this study the minimum residence time needed in the washing treatment was investigated in the range of 5 min to 40 min in order to achieve the desired ash content.

2.3.2 Amount of water for 100 g of feedstock

Jenkins et al.[1] used 7 l of distilled water for soaking and another 20 l for spraying the feedstock. Meanwhile Abdullah et al.[6] used 5–7 l of distilled water for soaking the feedstock. Their results showed that the ash content was reduced in 7 l of distilled water. Some researchers used 100 ml and 150 ml of water with 1 g of feedstock. In this study, 2–5 l of water was used with 100 g of feedstock.

2.4 The Procedures and Analytical Equipment

The following are the analyses performed on the unwashed and washed EFB to investigate their properties:

2.4.1 Atomic emission spectroscopy

The atomic emission spectroscopy analysis (AES) is a chemical analysis to determine the elements in a biomass feedstock. This method uses the intensity of light emitted from a flame at a particular wavelength. The intensity of the emitted light is proportional to the number of atoms of the element. In this analysis, Jenway PFP 7 Flame Photometer was used to determine the alkali and alkaline earth metals in terms of the emission wavelength and the colour of flame produced[22] as shown in Table 3. The flame photometer was performed on the ash of the unwashed and washed EFB.

Table 2: The element of alkali metals measured by flame photometer.

Element	Emission wavelength	Flame colour
Sodium (Na)	589	Yellow
Potassium (K)	766	Violet
Barium (Ba)	554	Lime Green
Calcium (Ca)	622	Orange
Lithium (Li)	670	Red

2.4.2 Thermogravimetric

The thermogravimetric analysis (TGA) is a thermal analysis technique performed to determine the changes in the weight of a sample as a function of temperature. The analysis is highly dependent on three measurements, which are the weight, temperature and temperature change.[23] In this work, the Pyris 1 TGA, which is a computer-controlled analyser was used. The TGA was carried out on the unwashed and washed EFB to interpret their qualitative composition of hemicellulose, cellulose, lignin and thermal degradation behaviour. Each TGA analysis was performed utilising 100 ml min^{-1} nitrogen with a heating rate of 10°C min^{-1} in the temperature range of 100°C –600°C.

2.4.3 The electrical conductivity

The electrical conductivity analysis was measured using a HI 8733 multi-range conductivity meter to measure the ability of water or solution to conduct an electrical current.[24] It depends on the concentration of the ion (higher concentration will result in higher electrical conductivity), the temperature of the solution (higher concentration will result in higher electrical conductivity), and specific nature of the ions (higher specific ability and valence will result in higher electrical conductivity).

2.5 Experimental Plan

Water washing pretreatment is one of the methods of reducing the ash content in biomass. It is required to enhance the pyrolysis reaction sufficiently to produce homogenous bio-oil. Water washing has become an important procedure to produce good quality biomass prior to any thermal conversion process to yield better quality fuel. In this work, the main objective of water washing pretreatment is to find the minimum parameter for washing to produce good quality EFB with ash content of less than about 3 mf wt%.[6,15,16] Tap water was used throughout the experiments.

EFB of size 1–3 cm was used in all experiments after shredding process was done. Effects of residence time, amount of water and employment of soaking and stirring methods were investigated in order to find the minimum parameter needed to reduce the ash. For all experiments, 100 g of EFB was used each time. The impacts of varying the amount of water from 2–5 l and residence time from 5 to 30 min were studied. The water washing treatments employed are summarised by number in Table 4.

Table 3: Summary of water washing treatments.

Treatment no.	Treatment
0	Untreated, sample not subjected to washing
1	Soaking for 5 min in 5 l water
2	Soaking for 10 min in 5 l water
3	Soaking for 20 min in 5 l water
4	Soaking for 30 min in 5 l water
5	Soaking for 5 min in 3 l water
6	Soaking for 10 min in 3 l water
7	Soaking for 15 min in 3 l water
8	Soaking for 20 min in 3 l water
9	Soaking for 25 min in 3 l water
10	Soaking for 30 min in 3 l water
11	Soaking for 5 min in 2 l water
12	Soaking for 5 min in 2.5 l water
13	Soaking for 5 min in 3 l water
14	Soaking for 5 min in 3.5 l water
15	Soaking for 5 min in 4 l water
16	Soaking for 5 min in 5 l water

3. RESULTS AND DISCUSSION

3.1 The Drying Process

Three whole EFBs, which were initially very wet, were weighed using a digital balance before they were dried. Table 5 shows the decreasing weight of the three EFB after 24 h, 48 h and 68 h of drying in a conventional oven. It was found that the weight of all samples stabilised after 68 h of drying as shown in Figure 1.

Table 4: The weight EFB waste after specified time of drying.

Sample	Initial weight (g)	Weight after first 24 h (g)	Weight after second 24 h (after 48 h) (g)	Weight after third 20 h (after 68 h) (g)
Sample 1	1403.27	539.55	468.77	466.30
Sample 2	1412.56	636.57	515.78	510.02
Sample 3	2528.62	1448.95	1134.25	1046.27

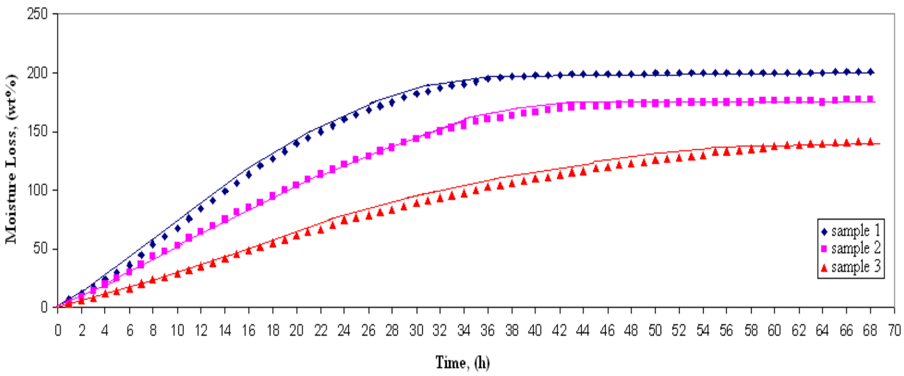

Figure 1: The moisture loss during drying process of the EFB.

Basically, the effects of moisture loss are similar to weight loss behaviour.[25] The moisture loss is calculated based on the dry basis. It was observed that as time increased, moisture loss also increased but started stabilising at 42 h during the drying process. During the heating process, water was removed from the biomass sample gradually until it reached equilibrium where most of the water has been evaporated. The moisture content of the samples was also measured as shown in Table 6.

Table 5: The moisture content of EFB.

Sample	Initial moisture content (mf wt%)	Moisture content after 24 h (mf wt%)	Moisture content after 29 h (mf wt%)	Moisture content after 68 h (mf wt%)
Sample 1	160.1	15.1	8.4	0.5
Sample 2	121.9	23.4	9.8	1.1
Sample 3	74.5	27.8	8.4	7.4

The initial moisture content for the three bunches of EFB was 160.1 mf wt%, 121.9 mf wt% and 74.5 mf wt%, respectively. After 29 h of drying, the moisture content of all samples was less than 10 mf wt%. By 68 h, only sample 3 maintained at high moisture content because the sample was the biggest and almost doubles the initial weight of the other two samples; hence it has the ability to hold more water. Figure 2 illustrates the decreasing moisture content in the drying process.

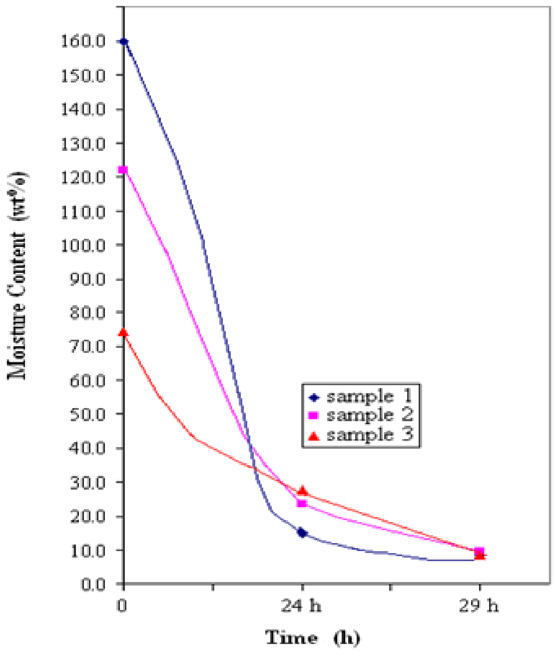

Figure 2: The decreasing moisture content of the EFB.

The results showed that the moisture content was 15.1 mf wt%, 23.4 mf wt% and 27.8 mf wt%, respectively for each sample after 24 h of drying. The moisture content became less than 10 mf wt% for all samples after 29 h of drying. It is a known fact that biomass moisture content of less than 10 mf wt% is essential for thermal conversion technologies such as pyrolysis process.[26,27]

3.2 Impact of Residence Time

Results of ash analyses for various treatments are listed in Table 7. The results showed that tap water is considered effective in reducing the ash content of the biomass. By soaking the feedstock in tap water for 5 min was enough to reduce the ash content to 2.66 mf wt%. It shows clearly in Table 7 that by soaking the feedstock for about 5 min or more in tap water at ambient temperature for most of the time was enough to achieve the minimum requirement of ash content of less than about 3 mf wt%.

Table 6: Ash content in unwashed and washed EFB

Treatment no.	Treatment	Ash content (mf wt%)
0	Untreated	5.19
1	Soaked 5 min, 5 l	2.66
2	Soaked 10 min,5 l	2.21
3	Soaked 20 min, 5 l	1.66
4	Soaked 30 min, 5 l	1.54
5	Soaked 5 min, 3 l	2.98
6	Soaked 10 min, 3 l	2.59
7	Soaked 15 min, 3 l	2.43
8	Soaked 20 min, 3 l	2.17
9	Soaked 25 min, 3 l	1.97
10	Soaked 30 min, 3 l	1.89
11	Soaked 5 min, 2 l	3.19
12	Soaked 5 min, 2.5 l	3.15
13	Soaked 5 min, 3 l	2.98
14	Soaked 5 min, 3.5 l	2.82
15	Soaked 5 min, 4 l	2.79
16	Soaked 5 min, 5 l	2.66

Interestingly, ash content of 1.54 mf wt% obtained after soaking the feedstock for 30 min in tap water at ambient temperature was the lowest. Therefore, it is expected that soaking at a much longer time will reduce more ash. Referring to Jenkins et al.[1] who studied the ash reduction in rice straw and wheat straw by water washing. They found that the flushing treatment they employed was quite effective in removing ash but the effect of soaking for 24 h was more effective with the overall ash being reduced by almost 50%. For the purpose of conserving water and time, the investigation was further continued for residence times of 5–30 min with as minimum amount of water needed to achieve ash content of less than about 3 mf wt%.

Figure 3 illustrates the correlation between the percentage of ash reduction in the EFB and the incremental electrical conductivity (EC) of waste liquor after soaking it in 5 l of water over a range of residence times (treatments 1–4 of Table 7). It was found that the percentage of ash reduction in the EFB has increased drastically for the first 5 min. Ash is easily reduced due to faster diffusion when the concentration of water is low for the first 5 min and then diffusion gets slower as the washed water becomes saturated with minerals. It also showed that the highest ash reduction was obtained at longer residence time until it reached equilibrium, as was also found by Piyali Das et al.[2] who argued that the residence time would be expected to impact washing efficiency. Furthermore, results showed that the increase of ash reduction is mirrored by the increase in electrical conductivity of the waste liquor. This may be due to the increase in ash reduction that caused the increase in alkali metal and organic ions contained in waste liquor.

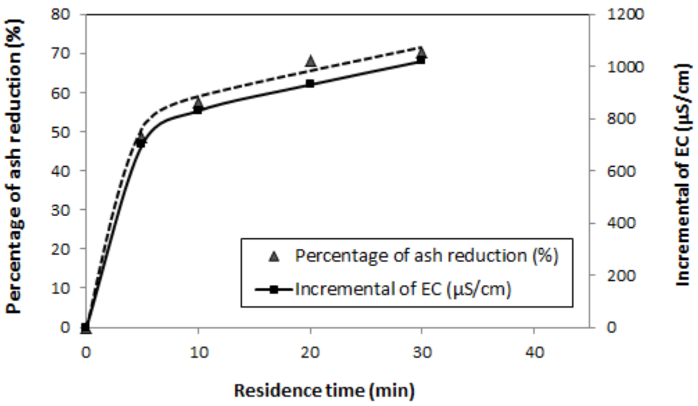

Figure 3: The percentage of ash reduction in the EFB and incremental electrical conductivity of waste liquor for using 5 l of water.

In order to further investigate the parameter for washing pretreatment, the experiments were continued by reducing the amount of water to 3 l. Approximately 3 l of water was used for 100 g of EFB at ambient temperature over a range of residence time from 5 to 30 min. The results showed that 3 l was enough to reduce the ash content to less than 3 mf wt% as shown in Table 7 (treatments 5–10).

Figure 4 shows that for the first 5 min, the percentage of ash reduction drastically increased as before when using 5 l of water. It shows that ash reduction and the incremental EC of waste liquor patterns was somewhat similar to the experiments done with 5 l of water. The longer the residence time, the more reduction of ash and the higher the incremental EC of waste liquor occur. It is expected that the waste liquor will become saturated and will be in equilibrium at a point after 40 min.

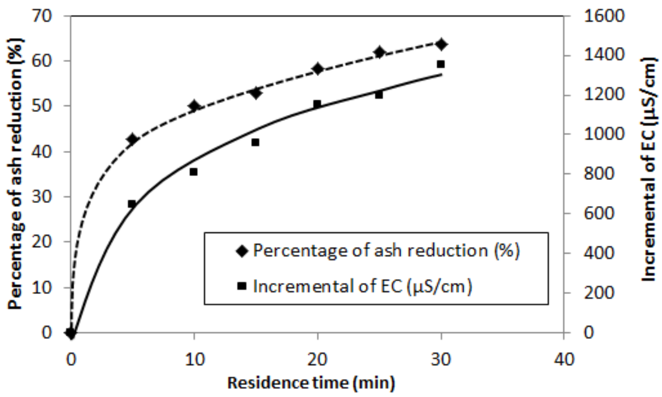

Figure 4: The percentage of ash reduction in the EFB and incremental electrical conductivity of waste liquor for using 3 l of water.

3.3 Impact of Amount of Water

Figure 5 shows the results of the washing experiments by soaking the EFB for 5 min in water of varying volume between 2–5 l at ambient temperature listed as treatment 11–16 as in Table 7. The percentage of ash reduction increased with increasing water volume while the EC of the waste liquor decreased. The increasing water volume had lowered the concentration of waste liquor, thus decreasing the EC. Moreover, the diffusion of mineral contents will be enhanced until it reached the equilibrium state. The results also showed that the percentage of ash reduction reached almost 50% by using 5 l of water suggesting that the increase of water volume will decrease the concentration of waste liquor, and consequently, ash will be easily diffused.

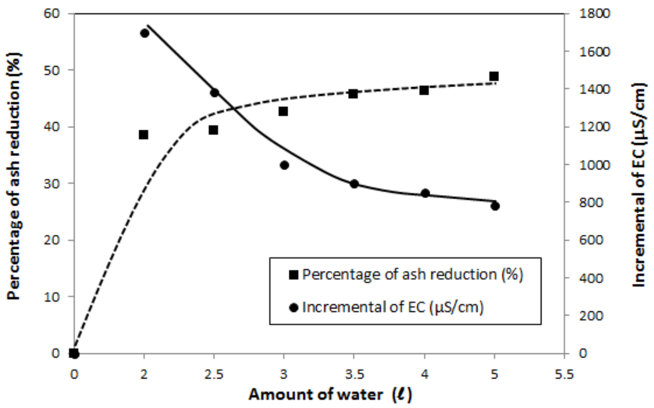

Figure 5: The percentage of ash reduction as a function of water amount and the incremental EC of waste liquor for soaking in 5 min.

Jenkins et al.[1] also achieved a much higher ash reduction for straw with the same amount of feedstock as this study when the volume of water increased from 7 l to 20 l. Furthermore, Di Blasi et al.[11] investigated the influence of water washing treatments on the degradation characteristics of wheat straw and they found that almost 60% of ash had been reduced by soaking 100 g of wheat straw of size 2–3 cm for 2 h in 10 l hot distilled water at temperature of 90°C.

3.4 Elemental Composition

The correlation between the percentage of ash reduction and the composition of potassium and sodium for seven samples (treatments 0 and 5–10 as of Table 7) are figured in Figure 6.

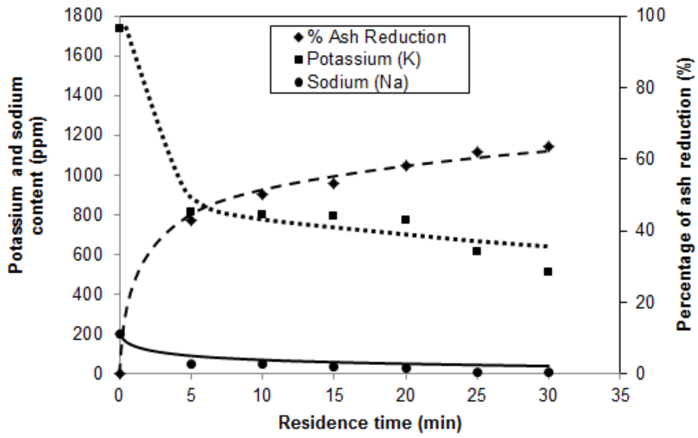

Figure 6: The correlation between the percentage of ash reduction and composition of potassium and sodium in washed EFB.

Figure 6 shows that water washing pretreatment is effective in reducing the potassium and sodium contents in the EFB. It is well known that potassium and sodium are proxies of ash. The potassium content were very high in the unwashed EFB and was reduced drastically in the first 5 min after undergoing soaking in all washed samples. Interestingly, the decreasing potassium and sodium contents are directly related to the decreasing ash content as suggested in the literature.[1,2]

The results from Figure 6 showed that around 50% of potassium and 75% of sodium were reduced in the ash of EFB by soaking the samples in 3 l of water for 5 min. As for soaking in 20 min, potassium was also reduced by around 55% and sodium was reduced by around 85%. Therefore, biomass with high alkali metal contents especially potassium can be dissolved very well of up to 70% by washing it with water.[13,14]

3.5 Thermogravimetric Analysis (TGA)

The thermal characteristics of the unwashed and washed EFB were analysed with the Pyris 1 TGA, which is a computer-controlled analyser. The TGA was performed under 100 ml min^{-1} nitrogen over a continuous increase in temperature from 100°C–600°C at a heating rate 10°C min^{-1}. The thermal degradation characteristics of the unwashed and washed EFB are shown in Figure 7 and Figure 8 by the thermogravimetry (TG) and DTG, respectively.

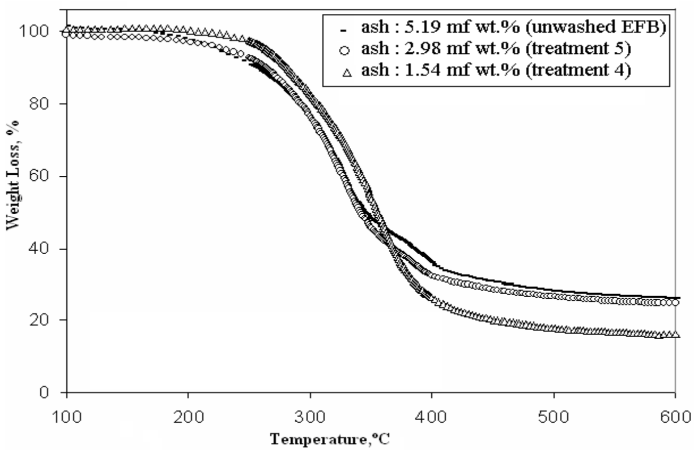

Figure 6: TG curves of unwashed and washed EFB.

The washing procedure employed for the washed EFB gave an ash content of 2.98 mf wt% (treatment 5) and 1.54 mf wt% (treatment 4) whereas for the unwashed EFB was 5.19 mf wt% which is represented as the percentage of weight loss as a function of temperature shown in Figure 7. It shows that from 200°C–400°C, the weight loss for all samples dropped significantly. The total weight loss was 92.8% for the unwashed EFB, 94.5% for the EFB with ash content 2.98 mf wt% and 98.7% for the EFB with ash content 1.54 mf wt%. The high weight loss for all samples is reasoned by the thermal degradation of polymer blocks of biomass such as hemicellulose, cellulose and lignin.[28]

The differential thermogravimetric (DTG) curves of the unwashed and washed EFB presented in Figure 8 are indicated by the derivative weight loss as a function of temperature. The main DTG peak is dominated by the composition of cellulose, hemicellulose and lignin. Yang et al.[29] had previously studied that the decomposition of hemicellulose, cellulose and lignin were from 220°C–300°C, 300°C–340°C and 750°C–800°C, respectively. Based on this, the peak appeared from 230°C–400°C representing hemicellulose, cellulose and lignin degradation for the unwashed and washed EFB of this work. This result is in agreement with previous study done by Raveendran et al.[30] and Jensen et al.[31] They showed that clear temperature shifts were observed for several herbaceous biomasses by reducing inorganic content through water washing, and the shift in the decomposition temperature is due to the catalytic effect of the ash.

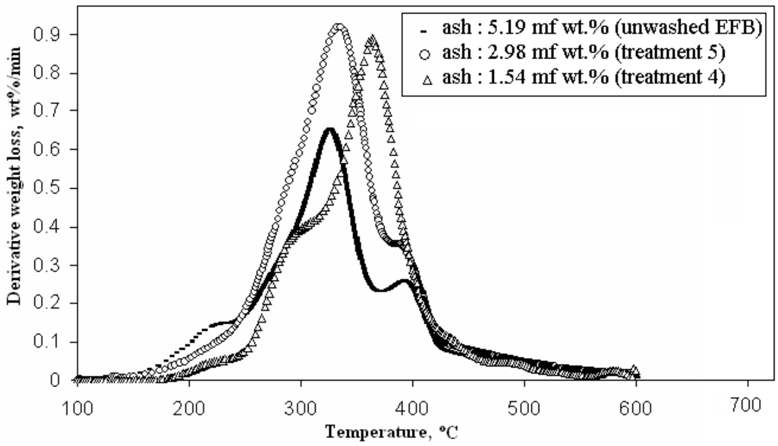

Figure 7: DTG curves of unwashed and washed EFB.

This figure shows that the peak of the washed EFB with ash content 1.54 mf wt% is clearly shifted to higher temperature of around 370°C compared to the other samples. Meanwhile, the washed EFB with ash content 2.98 mf wt% is slightly shifted to a higher temperature compared to the unwashed EFB of which the peak is around 330°C. The shift of the peak for the washed EFB is due to the reduction of the alkali metals' content and inorganic materials by the water washing pretreatment.

The TGA results provided an independent verification of the catalytic effect in the removal of the ash content by the water washing pretreatment. The variation in the TGA curves with ash content is smooth and comparable, thus, the analysis provides a quick and cheap method for assessing the effectiveness of the washing pretreatments.

4. CONCLUSION

The water washing experiments were carried out to investigate the impact of residence time, the amount of water used and the effectiveness of employment of soaking and stirring methods. The water temperature used in all experiments was between 25°C and 28°C, which is the ambient temperature of Malaysia and the size of the EFB samples was fixed at 1–3 cm. For each of the experiments, 5 ashing tests were carried out which consisted of 3 ashing tests for the washed EFB, 1 ashing test for sediment and 1 ashing test for leachate. The mass balance closure for all washing experiments were mostly above 95%.

The water washing pretreatment in this study was sufficient to remove about 24.9–70.3% of ash where the ash content for the unwashed EFB was 5.19 mf wt%. The washed EFB has an average of 2.48 mf wt% of ash content which is the minimum requirement for obtaining ash content of less than 3 mf wt% if soaked in 3 l of water for 5 min.

Tap water was used in all washing experiments. The ash in tap water used in the Energy Laboratory of research work was found to be 0.0042 mf wt%. In terms of economical advantage, the cost of using tap water is very much less than distilled water. In Malaysia, the cost of using tap water is RM0.00022 per l; compared to distilled water which is RM1.80 per l (US$1.00 ≈ RM3.00). In this study, 3 l of tap water is enough to reduce ash content of 100 g EFB to less than about 3 mf wt% by soaking method which is around 100 times cheaper than using distilled water, making it more lucrative economically.

In general, water washing pretreatment is effective in removing alkali metals in ash. Soaking method is most effective in reducing about 50% of ash content supported by works done of Jenkins et al.[1] and Di Blasi et al.[11] Almost 90% of potassium and 98% of sodium can be removed in ash if soaked in 5 l of tap water for 30 min.

The TGA shows that the unwashed and washed EFB start to decompose at lower temperature of 200–300°C, while the DTG peak of the washed EFB occurs at a higher temperature of around 330–370°C. The peak of the TGA curves of the washed EFB shifted to higher temperature compared to the unwashed EFB due to the reduction of ash that leads to less catalytic effect.

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