Bio-oil derived from empty fruit bunches

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Abstract

The fast pyrolysis of washed and unwashed empty fruit bunches (EFB), a waste of the palm oil industry, is investigated in this study. Firstly, the composition and particle size distribution of the washed and unwashed feedstock were determined and the thermal degradation behaviour was analysed by TGA. Then a 150 g/h fluidised bed bench scale fast pyrolysis unit was used to study the impact of key variables: reactor temperature in the range 425–550 °C and feedstock ash content in the range 1.03–5.43 mf wt%. The properties of the liquid product were analysed and compared with wood derived bio-oil and petroleum fuels. It was found that the maximum ash content of washed feedstock that still yields homogenous liquids is less than about 3 mf wt%. The experiments also indicated that the fast pyrolysis of washed EFB with a low ash content gave similar yields as commonly obtained for wood.
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1. Introduction

Fast pyrolysis is a thermal decomposition process that occurs at moderate temperatures in which the biomass is rapidly heated in the absence of oxygen or air to produce a mixture of condensable liquids, gases and char [1]. It is one of the most recent renewable energy processes and promises high yields of liquid with a minimum of gas and char if it is carefully controlled. Normally for woody feedstocks, temperatures around 500 °C together with short vapour residence times are used to obtain bio-oil yields of around 70%, and char and gas yields of around 15% each [2]. Bio-oil is a high density oxygenated liquid that can be used as a substitute for liquid fossil fuels in some applications. It can be burned in diesel engines, turbines or boilers, though further work is still required to demonstrate long term reliability [3]. It may also be used for the production of speciality chemicals, currently mainly flavourings. Renewable resins and slow release fertilisers are other potential applications, which have been the subject of research [4].

Bio-oil is perceived to offer logistical and hence economic advantages over other thermal conversion processes, as it is a liquid with a relatively high density of around 1.2 kg/l [4]. A preliminary assessment [5] indicates that two truckloads of wood chips can be replaced by a single tanker load of bio-oil with the same energy content, but that there is no advantage compared to wood pellets.

The palm oil industry is currently expanding rapidly and yields large amounts of poorly utilised waste biomass [6]. A pilot plant utilising BTG technology and EFB as the feedstock for bio-oil production has also recently started up [7]. 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 [6] and it was found that in all cases the liquid product separated into two phases presenting difficulties for fuel applications. In this work, washed EFB was pyrolysed in a fluidised bed reactor with a nominal capacity of 150 g/h, with the objective of determining the effect of ash reduction on yield and

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the maximum ash level giving a homogenous bio-oil. The results are compared to those from pyrolysis of various feedstocks with different ash contents.

2. Feedstock preparation and properties

The EFB used in the experiments was supplied by the Malaysian Palm Oil Board (MPOB). Samples received were relatively dry having less than 10 mf wt% moisture, and were in the form of whole bunches. Particle size reduction was required to allow fast pyrolysis of the EFB on the available 150 g/h system. The bunches were first manually chopped into smaller pieces that could be fed into a shredder. After that, a Fritsch grinder with a screen size of 500 μm was used in order to reduce the feedstock size to less than 500 μm. The distribution of feed particle size after grinding is given in Table 1. The ash content of each size fraction was determined using National Renewable Energy Laboratory (NREL) Standard Analytical Method LAP005, and the mass average of the size fractions of 5.39% compares well, within the accuracy of the measurements, with the original sample sent by MPOB, which had an ash content of 5.36%.

After extensive feeding trials for unwashed feedstock, it was found that only particles between 250 μm and 355 μm are easily fed. Both the size fractions below and above this range frequently led to blockage of the available feeder. As

shown in Table 1, this size fraction had an ash content slightly below the mass average of the whole EFB sample. Further details can be found in Abdullah [6].

Key properties of EFB, both measured for this research and from the literature, are given in Table 2. 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 [3]. The carbon and hydrogen content are comparable to woody biomass, as is the measured heating value. The lowest HHV in the literature [8] 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 [9] – a value of 10 MJ/kg is quoted for dry oil palm matter, which is clearly too low for a ligno-cellulosic biomass on a dry basis – or [10] – values for wet FFB are used for dry FFB.

To reduce the ash content of the EFB by varying amounts a range of simple water washing techniques was employed, work that is described in greater detail elsewhere [6]. The treatments used for this work are briefly described in Table 3. It is worthwhile to note that freshly sterilised empty fruit bunches typically have a water content of 60% on a wet basis. Water washing as described in [6] therefore does not have to bear a large additional cost burden from drying, as drying is required in any case.

3. Experimental procedure

A fluidised bed bench scale fast pyrolysis unit operating at atmospheric pressure was employed for nearly all runs. Fig. 1 shows a schematic diagram of this unit, which consists of three main parts, namely the feeder, reactor and product collection system. The reactor is a stainless steel (type 316) cylinder with a length of 260 mm and a diameter of 40 mm. The heating medium in the reactor is inert sand of size between 355 μm and 500 μm. The

Table 1
Particle size distribution of EFB

Feed particle size (μm)	Mass fraction	Average ash (mf wt%)
Less than 250	22	7.44
250–355	30	5.29
355–500	42	4.82
More than 500	6	4.72
Mass average	–	5.39

Table 2
Properties of EFB (mf wt%), cited literature [8,10,19–26]

Component/property	Literature values	Measured	Method
Cellulose	59.7, 38.1–42.0	–	–
Hemicellulose	22.1, 16.8–18.9	–	–
Lignin	18.1, 10.5–11.7	–	–
Elemental analysis			
Carbon	48.9, 48.8, 49.2–50.6	49.07	Combustion analysis
Hydrogen	7.33, 6.3	6.48	
Nitrogen	0.0, 0.7, 0.78–1.19, 0.2, 0.8, 0.44	0.7	
Sulphur	0.68, 0.2	<0.10	
Oxygen	40.2, 36.7	38.29	By difference
K	2.41, 2.24	2.00	Spectrometry
K_2O	3.08–3.65	–	
Proximate analysis			
Moisture	–	7.95	ASTM E871
Volatiles	87.3, 75.7	83.86	ASTM E872
Ash	3.02, 7.3, 4.3	5.36	NREL LAP005
Fixed carbon	9.6, 17	10.78	By difference
HHV (MJ/kg)	19.0, 17.86, 15.5, <10	19.35	Bomb calorimeter
LHV (MJ/kg)	17.2	–	–

Table 3
Ash content of washed EFB according to washing method

| Ash content (mf wt%)	Water washing method
1.03	Soak 100 g of feedstock of size 250–355 µm for 24 h at ambient temperature in 7 l of distilled water
2.14	Soak 100 g of feedstock of size 2–3 cm for 20 min at ambient temperature in 5 l of distilled water
3.05	Soak 100 g of feedstock of size 2–3 cm for 10 min at ambient temperature in 5 l of distilled water
3.68	By manual agitation of 100 g of feedstock of size 2–3 cm for 1 min at ambient temperature in 5 l of distilled water
5.43	Unwashed feedstock that was not subjected to any washing. The ash content of this batch differs fractionally from the mass average of the batch referred to in Table 1, and is modestly higher than the ash content of the size fraction of unwashed biomass that could be fed for fast pyrolysis
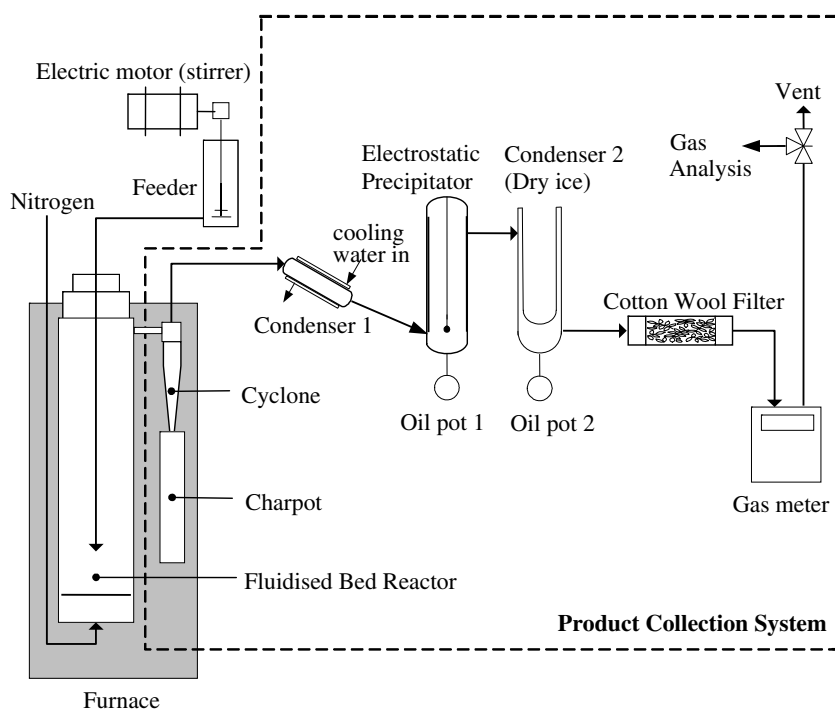
Fig. 1. Flow diagram of the 150 g/h fluidised bed pyrolysis system.

sand fills the reactor to a depth of approximately 8 cm and expands during fluidisation to 12 cm. The fluidising gas is nitrogen, which is preheated prior to entering the base of the reactor.

The char is carried out of the reactor body by the fluidising gas flow, which is known as 'blow-through' mode [11]. The char is then separated from the product stream in a cyclone. The condensable vapours are collected in the liquid products collection system, which consists of two cooled condensers, an electrostatic precipitator and a cotton wool filter. The incondensable gases leave the system through a gas meter and are then sampled by gas chromatography (GC) to assess the quantity and type of gas produced. The GC utilised is a Varian Micro GC with helium as the carrier gas and two columns (molar sieve and poropak). Calibration is against calibration gases supplied by BOC at concentrations of 0.5 vol%, 1 vol% and 3 vol%. Due to dilution with the fluidising gas nitrogen, carbon

monoxide and carbon dioxide are typically between 0.5 vol% and 1.5 vol%, while hydrogen and the low molecular weight hydrocarbons are well below 0.5 vol%. These latter gases do not contribute materially to the mass balance closure and therefore do not need to be measured to the same level of accuracy. Due to the use of helium as the carrier gas, the concentration of hydrogen is particularly difficult to measure accurately at low concentration with the GC used for this work.

4. Experimental plan

To investigate the effect of ash content, a number of washed feedstocks subjected to a range of water washing techniques of varying severity (see Table 3) were pyrolysed. Five experiments were carried out at a reactor temperature of 500 °C and hot vapour residence times of 1.02–1.06 s considering the following ash contents: 1.03 mf wt%,

2.14 mf wt%, 3.05 mf wt%, 3.68 mf wt% and 5.29 mf wt%. The results are compared to those from pyrolysis of various feedstocks with different ash contents.

To investigate the effect of temperature, fast pyrolysis experiments were carried out at a vapour residence time of 1.01–1.04 s on feedstock of size 355–500 μm covering the temperature range of 425–550 °C in 25 °C increments. The ash content of the feedstock used for these runs was 1.03 mf wt%. The potassium content was 0.13 mf wt%. Simple water washing was sufficient to remove over 90% of the potassium and over 80% of the ash.

One pyrolysis experiment was carried out at a temperature of about 500 °C and at a residence time of 1.1 s on washed feedstock of size 355–600 μm with an ash content of 1.06 mf wt% using a larger pyrolysis system (1 kg/h) to study the impact of a different product collection system and scale.

5. Thermogravimetric analysis of washed EFB and unwashed EFB

The thermal characteristics of washed and unwashed EFB were analysed with a computerised Perkin–Elmer Pyris 1 thermogravimetric analyser. Thermogravimetric analysis (TGA) was performed under 100 ml/min nitrogen with a heating rate of 10 °C/min. The thermal degradation characteristics of unwashed and washed feedstock are displayed in Figs. 2 and 3 by thermogravimetry (TG) and dif-

Fig. 2. Thermogravimetric analysis of washed and unwashed EFB.

Fig. 3. Differential thermogravimetric analysis of washed and unwashed EFB.

ferential thermogravimetry curves (DTG), respectively. The washing procedure employed for the washed sample gave an ash content of 1.15 mf wt% after two hours of stirring 100 g of EFB in 7 l of distilled water at 90 °C. The ash content of the unwashed sample was 5.43 mf wt%. The thermal behaviour of the two samples differs substantially. The unwashed sample shows a small DTG peak around 200 °C, which may be indicative of extractives [12]. No such peak is apparent for the washed sample. A possible explanation is that the high washing temperature may have led to the loss of extractives during washing. For simple soaking at room temperature, the peak around 200 °C is not lost.

The unwashed sample shows a single peak at 355 °C, while the washed sample has its greatest weight loss at 383 °C. Furthermore there is a hump at around 320 °C for the washed sample, which is not visible in the DTG curve of the unwashed sample. Similar trends have been reported by other authors for low ash and high ash biomass feedstocks [12–15]. It is generally supposed that the temperature shift is due to the catalytic effect of the ash and that this leads to a single DTG peak for both cellulose and hemicellulose for high ash biomass, while the hump apparent in the DTG curve for low ash biomass is indicative of hemicellulose and the actual peak is due to the decomposition of cellulose.

The total weight loss between 100 °C and 450 °C is 77.4% for the washed sample and only 65.8% for the unwashed sample. There are two explanations. Firstly, the ash itself will largely not volatilise and therefore contribute to the char yield. Secondly, the ash is also believed to contribute to greater char production through its catalytic activity.

A number of other washed samples with intermediate ash contents have been subjected to TGA analysis. The resulting curves vary smoothly between the two extremes presented in Figs. 2 and 3. The total weight loss between 100 °C and 450 °C and temperature of maximal weight loss are listed in Table 4.

The TGA results provide an independent verification of the catalytic effect of the ashes removed by washing. As the variation in the TGA curves and the fast pyrolysis organics yield with ash content is smooth and comparable, they represent a quick and cheap way to assess the effectiveness of washing.
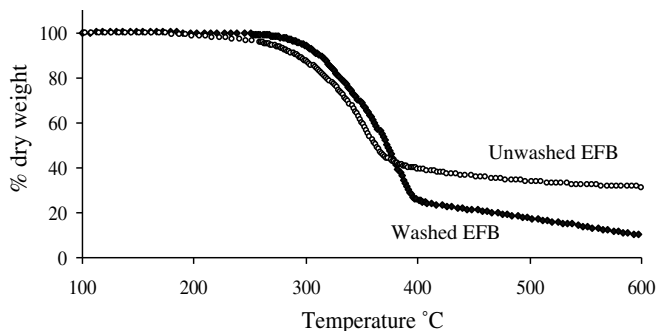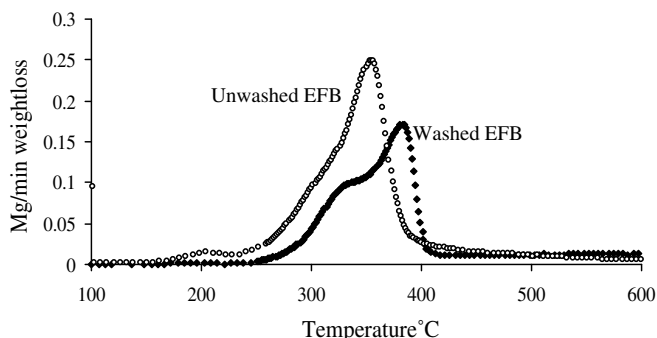
Table 4
Thermogravimetric analysis data for a range of ash contents

Ash content (mf wt%)	Temperature of peak weight loss (°C)	Weight loss between 100 °C and 450 °C (%)
5.43	355	65.8
3.68	358	69.4
3.05	361	70.6
2.14	366	74.4
1.15	383	77.4

6. Results and discussion

6.1. Effect of reactor bed temperature on product yields

The product yields resulting from varying the reactor temperature for washed EFB are shown in Figs. 4–6. All yields are given on a dry feedstock basis. The gas yield rises with temperature, while the char yield declines. The water yield is approximately constant over the temperature range considered, while there is a maximum for the organics and liquids yields of 61.3% and 72.4%, respectively at roughly 500 °C.

The yields of methane and carbon monoxide rise much faster with temperature than is the case for carbon dioxide. Similar results for the impact of reactor temperature on gas composition are reported by other authors for the fast pyrolysis of woody biomass, for example [16].

6.2. Effect of ash content of feedstock on product yields

The impact of varying EFB ash content through simple water washing is illustrated in Fig. 7. Lowering the ash content results in a substantial increase in organics yield, while reaction water, char and gas yields all decline. A single phase oil was obtained with an EFB ash content of less than roughly 3 mf wt%. Lowering the ash content from 2.04 mf wt% to 1.03 mf wt% gave an increase in organics

Fig. 4. Yields of char, gas and reaction water as a function of temperature for washed feedstock.
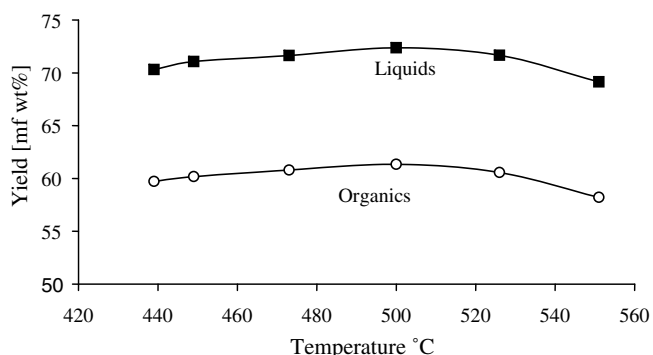
Fig. 5. Yields of organics and all liquids as a function of temperature for washed feedstock.
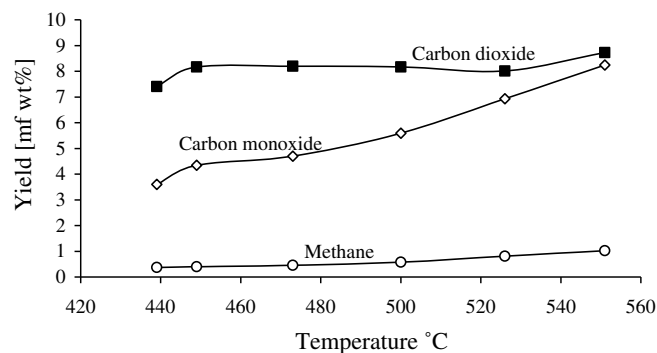
Fig. 6. Yields of methane, carbon monoxide and carbon dioxide as a function of temperature for washed feedstock.

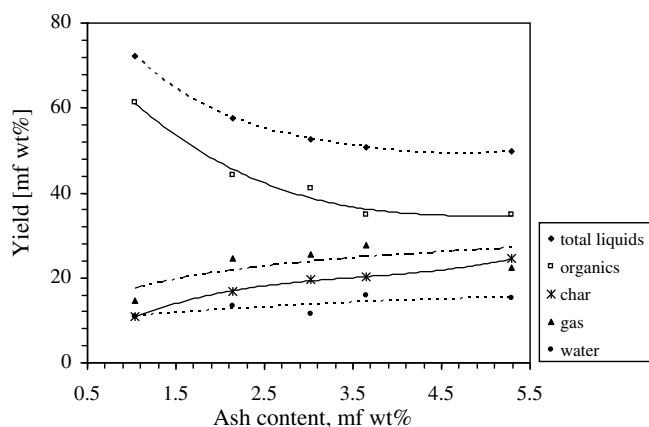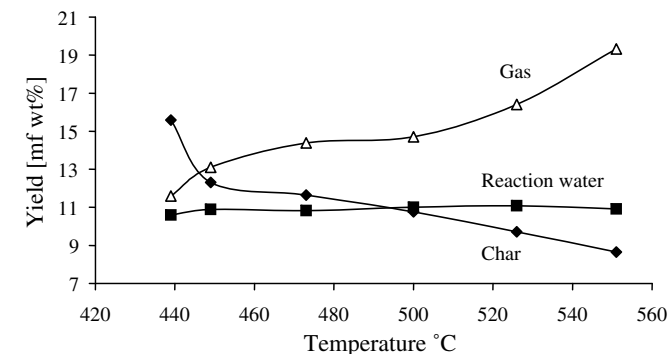Fig. 7. Impact of ash content on product yields.

yield on a dry basis from 44.32% to 61.34%, while unwashed EFB yielded 34.71% under the same conditions. As mentioned earlier, it is generally accepted that ash, and particularly potassium, is catalytically active and favours secondary reactions leading to a reduction in organics yield, and corresponding increases in reaction water, gas and char yields [3,17]. In comparison to the data presented by [3], however, the drop in organics yield between 1 mf wt% and 2 mf wt% ash is much steeper in the work here presented. A possible explanation is that water washing results in a selective reduction of potassium, while the selection of feedstocks considered by [3] is likely to include some with a relatively higher potassium content for ash contents around 1 mf wt%.

The yield of carbon dioxide rises substantially with increased ash content. At 500 °C and for unwashed EFB it is 15% and three times as high as the yield of carbon monoxide. For the same conditions, EFB washed to an ash content of 1.03 mf wt% gives a carbon dioxide yield of only just over 8%, about one and a half times as high as the yield of carbon monoxide.

6.3. Feedstock comparison

A comparison of EFB with results from the literature for other biomass feedstocks is presented in Fig. 8 and
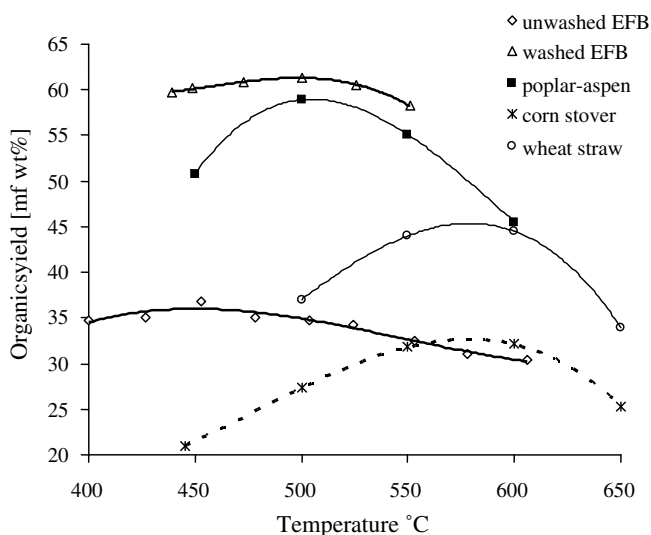
Fig. 8. Organics yield as a function of temperature for a number of feedstocks.

Table 5
Comparison of EFB with other biomass feedstocks

Feedstock	Unwashed EFB	Washed EFB	Poplar aspen	Corn stover	Wheat straw
Reference	This work	This work	[18]	[11]	[11]
Cellulose (mf wt%)	59.7	–	42.3	31.0	32.4
Hemicellulose (mf wt%)	22.1	–	31.0	43.0	41.8
Lignin (mf wt%)	18.1	–	16.2	13.0	16.7
Moisture (mf wt%)	7.48–8.96	6.04–6.54	5.0	6.5	9.0
Ash (mf wt%)	5.36	1.03	0.39	11.0	4.6
Yields (mf wt%)					
Char	24.52	10.76	11	42.2	22
Gas	22.31	14.70	14	13.7	19
Organics	34.71	61.34	58.9	27.3	37
Total liquids	49.8	72.36	74.1	43.1	54

Table 5. The product yields for washed EFB are similar to those for a low ash wood, such as poplar, while the product yields for unwashed EFB are much closer to those for higher ash feedstocks, such as wheat straw [18,11].

6.4. 1 kg/h reactor

Fig. 9 shows a schematic flow diagram of the 1 kg/h fluidised bed pyrolysis system. It consists of a stainless steel reactor, a fluid bed gas preheater, a screw feeder, a solid–gas cyclone separator, a quench column, an electrostatic precipitator (ESP) and a dry ice condenser. The operation of the 1 kg/h system is similar to the 150 g/h system which also employs nitrogen as the fluidising gas and sand as the bed material.

The sand bed was heated to the desired temperature of about 500 °C. The washed feedstock of size 355–600 μm was fed into the bubbling hot sand in the reactor. The reaction products were passed through two cyclones where the char was removed. The remaining vapours were quenched with cooled recycle isopar, a proprietary isoparaffin supplied by Exxon Mobil Corporation. To remove aerosols
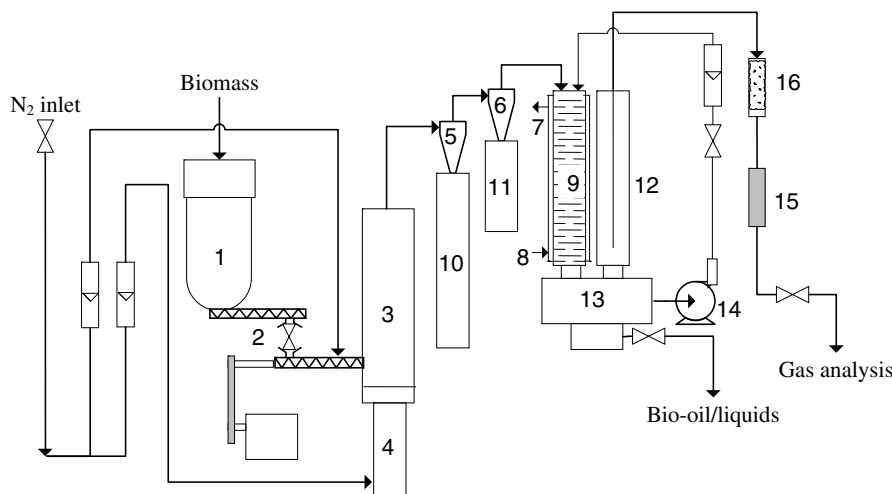
Legend :
1. Feed container
2. Screw feeder
3. Fluidised bed reactor
4. Gas heater
5. Cyclone 1
6. Cyclone 2
7. Cooling water out
8. Cooling water in
9. Quench column
10. Char pot 1
11. Char pot 2
12. Electrostatic precipitator (ESP)
13. Tank
14. Pump
15. Cotton filter
16. Dry ice condenser

Fig. 9. Flow diagram of the 1 kg/h fluidised bed pyrolysis system.

not captured by the quench column the gas was then passed through an electrostatic precipitator which is very effective at capturing aerosols. The liquids collected by the quench column and ESP drain into a common storage tank, which also contains the isopar. Isopar is lighter than bio-oil. Bio-oil therefore sinks to the bottom of the tank, while isopar accumulates at the top, where the pump inlet for the quench column recycle pump is located.

After the run it was found that a tarry mass had stuck to the wall of the ESP. 22.8% and 74% of the total pyrolysis liquid yield were collected on the ESP walls and in the tank, respectively. Subsequently the ESP was modified to allow continuous washing of its walls with isopar. No further runs on the 1 kg/h system have, however, been performed using EFB as the feedstock.

In Table 6, results for similar conditions employing the 150 g/h system and the 1 kg/h system are shown. The ratio of carbon monoxide to carbon dioxide and the char yield are virtually identical between the two runs. The water yield appears substantially lower on the 1 kg/h system, the gas yield substantially higher, and the organics yield moderately lower. There are several reasons for caution, however. Firstly, in later experiments on the 1 kg/h system it was found that the dry ice condenser was not always very efficient, which could largely explain the differences in the reaction water and organics yields. Secondly, there were only four good GC measurements of the gas composition and these showed strong variability between them. The most likely explanation appears to be fluctuations in the feed rate, which on average was higher than calibrated and appeared to be visibly influenced by pressure fluctuations emanating from the reactor. Gas appeared to alternately enter the feeder hopper, bringing feeding to a halt for one or two seconds, and leaving it, taking the light EFB feedstock with it. Thirdly, the assumption that isopar and bio-oil will completely separate is questionable, particularly so for any vegetable oil fraction contained in the EFB derived bio-oil, which could conceivably be extracted by the isopar.

6.5. Physical and chemical properties of products

A comparison of key properties for the two phases produced from unwashed feedstock and the two liquid fractions collected on the 1 kg/h system, with those of wood derived bio-oil, light fuel oil and heavy fuel oil is given in Table 7. The organics phase generated from the fast pyrolysis of unwashed feedstock has a very large higher heating value, which at over 36 MJ/kg nearly approaches the range of petroleum fuels. The tars collected by the ESP of the 1 kg/h system are considerably lower, coming in at just over 25 MJ/kg, and the liquid collected in the main product collection tank is just under 19 MJ/kg. Taken together the pyrolysis liquids produced on the 1 kg/h system average roughly 20 MJ/kg. This is not entirely unsurprising, as the high yields of reaction water and carbon dioxide for unwashed feedstock imply that the remaining products must have a larger HHV than the original biomass. High bio-oil yields by contrast mean that the HHV of the pyrolysis liquid will approach that of the original biomass.

Table 6
Comparison of results for 150 g/h and 1 kg/h units

	150 g/h	1 kg/h
Reactor		
Reactor temperature (°C)	500	499
Hot vapour residence time (s)	1.02	1.10
Feed moisture (mf wt%)	6.04	7.25
Ash (mf wt%)	1.03	1.06
Particle size (μm)	355–500	355–600
Run time (min)	64.33	52.23
Input		
Wet feed (g)	75.23	1085.61
Dry feed (g)	70.95	1012.25
Water in feed (g)	4.28	73.36
Yields (mf wt%)		
Char	10.76	11.19
Organics	61.34	57.25
Gas	14.70	23.07
Water	11.01	6.96
Total liquids	72.36	64.21
Closure	97.8	98.47
Gas (mf wt%)		
Methane	0.58	0.63
Carbon dioxide	8.17	13.17
Carbon monoxide	5.59	8.77
Hydrogen	0.07	0.03
Ethylene	0.17	0.27
Ethane	0.10	0.12
Propane	0.02	0.04
Propylene	0.01	0.01

Table 7
Characteristics of pyrolysis oil and petroleum fuel [3]

	Unwashed EFB		Washed EFB		Wood derived bio-oil	Light fuel oil	Heavy fuel oil
	Organics phase	Aqueous phase	From ESP	From tank			
C	69.35	13.83	56.47	41.86	32–48	86.0	85.6
H	9.61	11.47	7.85	7.82	7–8.5	13.6	10.3
N	0.74	0.14	0.22	0.1	<0.4	0.2	0.6
O	20.02	74.56	35.46	50.22	44–60	0	0.6
S	–	–	–	–	<0.05	<0.18	2.5
Water (wt%)	7.9	64.01	6.66	21.68	20–30	0.025	0.1
HHV (MJ/kg)	36.06	–	25.29	18.63	–	–	–
LHV (MJ/kg)	–	–	–	–	13–8	40.3	40.7

7. Conclusions

A series of fast pyrolysis experiments on washed EFB have been successfully performed using a 150 g/h fluidised bed reactor operating at atmospheric pressure. The yield maximum for liquids produced from washed EFB was increased to around 72% compared to just under 50% for liquids produced from unwashed EFB. The ash content of the feedstock has been found to significantly influence the yield of organics. The higher the concentration of ash in the feedstock the lower the yield of pyrolysis liquid. Overall product yields for washed EFB compare well with low ash woody feedstocks. A reduction of ash content to less than about 3 mf wt% is required in order to produce homogenous liquids, that is to avoid phase separation of liquids.

Acknowledgments

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