Journal of Applied Sciences

ISSN 1812-5654

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Journal of Applied Sciences 12 (24): 2527-2533, 2012
ISSN 1812-5654 / DOI: 10.3923/jas.2012.2527.2533

Analysis of the Pyrolytic Fuel Properties of Empty Fruit Bunch Briquettes

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Abstract: Briquetting is technique for mechanically compacting and densifying loose materials into a uniform solid fuel aimed at improving the physical and thermochemical properties such as energy density, moisture content. This study is aimed at investigating the pyrolytic fuel properties of briquettes using DSC thermal analysis. The EFB Briquettes were pulverized and sieved in a Retsch analysis sieve with screen size 800 micron. The Higher Heating Value (HHV) of the fuel was determined using a bomb calorimeter according to the ASTM standard D-2015 technique. Proximate analysis was carried to determine the moisture content, volatile matter, fixed carbon and ash content of the fuel. The powdered EFB briquette was analyzed using a Perkin Elmer DSC 7 thermal analyzer under pyrolysis conditions from 30 to 500°C at a constant heating rate of 10°C min^{-1} with nitrogen (N_2), flow rate of 25 mL min^{-1} as sweeping gas. The calorific changes during DSC analysis were recorded and analyzed. A Scanning Electron Microscope (SEM) was used to investigate the surface composition and particle size distribution of the fuel. Surface analysis revealed that 57% of the fuel particles are in the 151-250 μm range. The HHV value of the fuel obtained was 17.57 MJ kg^{-1} and specific heat C_p = 1,397 J kg^{-1} K^{-1}. From the FTIR spectra of the fuel, the product gases as H_2, CO, CO_2, CH_4 and C_mH_n can be reasonably predicted. The results indicated that EFB briquette has good fuel properties and can be utilized as a fuel in biomass pyrolysis.

Key words: Biomass, palm oil, briquettes, pyrolysis, DSC analysis, microscopy

INTRODUCTION

The production of crude palm oil in Malaysia generates significant quantities of solid lignocellulosic biomass as waste. The palm based solid bio-waste comprises; 53% Empty Fruit Bunches (EFB), 32% palm mesocarpfibre and 15% palm shell (Baharuddin *et al*., 2009; Wahid *et al*., 2004; Yusoff, 2006). Current production data estimate around 7 million tonnes of EFB, 4.5 million tonnes of palm fibre and 1.9 million tonnes of shell, are generated annually from palm oil production in Malaysia (Mae *et al*., 2000). With the generation of solid palm waste set to increase 5% annually, socioeconomic and environmental problems such as global warming and the greenhouse effect are imminent. Consequently, urgent practical and sustainable solutions are required to curtail the future potential effects of increased solid bio-waste from palm oil production. While efforts are currently focused on the utilization of Empty Fruit Bunch (EFB) as fuel in boilers to generate steam and electricity in palm oil mills; a large fraction is still simply incinerated or burnt in open air (Mahlia *et al*., 2003; De Souza *et al*., 2010; Lahijani and Zainal, 2011). The low efficiency of current conversion technologies has led to increased emission of large quantities of CO_2 and other greenhouse gases (GHGs) such as NO_x, SO_x into the atmosphere. Consequently researchers in Malaysia are currently exploring novel, efficient and sustainable process technologies to convert palm oil based waste into a renewable source of energy. The efficiency of EFB fibres currently used as boiler fuel in palm oil mills can be improved by briquetting. This is a technique for mechanically densifying biomass by compacting loose materials into a uniform solid fuel. This is known to improve the physical and thermochemical properties such as homogeneity, energy density, energy content, moisture content as well as storage and handling of the fuel (Grover and Mishra, 1996; Bhattacharya and Shrestha, 1990; Bhattacharya *et al*., 1989). These properties make briquettes ideal fuels for application in biomass conversion technologies such as gasification and pyrolysis which are considered promising technologies for converting CO_2 neutral biomass into clean energy for future applications (McKendry, 2002; Demirbas, 2001). Pyrolysis is the thermal decomposition of biomass into solid charcoal, liquid (pyrolysis oil) and H_2 rich gases in

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the absence of oxygen (McKendry, 2002). The rate of biomass pyrolysis is influenced by biomass properties and operating parameters such as moisture content, temperature, heating rate, particle size and catalysts (Chen *et al*., 2003; Di Blasi *et al*., 1999; Li *et al*.,2004; Lappas *et al*., 2002). Previous studies on biomass pyrolysis have focused on the parametric of biomass pyrolysis using thermal analytical techniques such as Differential Scanning Calorimetry (DSC) and Thermogravimetric analysis (TGA) (Rath *et al*., 2003; Stenseng *et al*., 2001; He *et al*., 2006). Studies by Mahlia *et al*. (2003), Lahijani and Zainal (2011), Mohammed *et al*. (2005), Sulaiman and Abdullah (2011) and Abdullah *et al*. (2010) have all highlighted the potential of EFB as a feedstock fuel for hydrogen production. Studies by Yang *et al*. (2004); Yang *et al*. (2006) have investigated the pyrolysis of palm oil waste using Thermogravimetric Analysis-Fourier Transform Infrared (TGA-FTIR) for the production of hydrogen. Other applications of EFB include for the production of chemicals, biofuels (Kassim *et al*., 2011; Kelly-Yong *et al*., 2011; Bari *et al*., 2010) and agricultural inputs such as organic manure (Sung *et al*., 2010; Affendy *et al*., 2011). Nonetheless, the thermochemical, kinetic mechanisms and pyrolytic fuel properties of biomass fuels requires further investigation. Studies on the fuel properties on EFB briquettes as a fuel for pyrolysis have not been previously reported in literature. Therefore, this study is aimed at investigating the pyrolytic fuel properties of EFB briquettes using DSC analysis, FTIR and SEM microscopy for clean energy applications for the future.

MATERIALS AND METHODS

The EFB briquettes with average dimensions (4.9×2.5×0.8) cm used in this study were acquired from FeldaSemenchuSdnBhd, Johor, Malaysia. Fig. 1, shows the briquettes produced from Empty Fruit Bunches (EFB).The EFB briquettes were pulverized in a high speed crusher machine (Kimah Malaysia, Model RT 20) fitted with a 1 mm screen. The resulting briquette powder was sieved for a second time in a Retsch analysis sieve (D-42759, Haan Germany, screen 800 micron) to obtain particles with particle size <800 µm reportedly (He *et al*., 2006) ideal for DSC analysis. The proximate analysis of fuel was carried out to using ASTM standard techniques for determining the moisture content, volatile matter, ash content and fixed carbon content of biomass fuels. The Higher Heating Value (HHV) of the EFB briquette was analyzed using a bomb calorimeter (IKA calorimeter system, Model C2000) according to ASTM standard D-2015 technique. Using a table top SEM scanning electron microscope (Model Hitachi TM 3000) high resolution micrographs were obtained at a magnification of x50 and x100 using Mode A analytical settings to analyze the surface composition and particle size distribution of the fuel.

FTIR, Fourier Transform Infra-Red spectroscopic (Model Perkin Elmer Spectrum One) analysis was carried on the fuel and the IR spectra recorded from 4000 to 500 cm^{-1}. Subsequently, the pyrolysis of the powdered EFB briquette was carried out using a computerized Perkin-Elmer DSC 7 thermal analyzer. The powdered

Fig. 1(a-b): (a) Empty fruit bunch (EFB) and (b) Empty fruit bunch (EFB) briquette sample was placed in an aluminium crucible with a lid and heated in the DSC furnace from 30 to 500°C at a constant heating rate of 10°C min^{-1} using nitrogen (N_2) (flow rate of 25 mL min^{-1}) as sweeping gas. After each run the furnace was cooled and the DSC traces of each run obtained for each sample. The changes observed during DSC analysis were recorded and analyzed in MS EXCEL to obtain the calorific requirement from which the specific heat capacity (C_p) of the fuel can be deduced. The calorific requirement of biomass pyrolysis is a measure of the heat required to heat the sample and to complete the reaction. It can be calculated from the formulae (Jalan and Srivastava, 1999; Liliedahl and Sjostrom, 1998; Sharma and Rao, 1998; Rath *et al*., 2002):

$$Q = C_{p,b} \int m_b \, dT + C_{p,ch} \int m_{ch} \, dT + Q_P \qquad (1)$$

Although the calculation method for determining the calorific requirement of biomass from Eq. 1 is widely accepted, it is complex and has numerous limitations. According to a study by He *et al*. (2006), the complexity of the calculation method is due to the large changes in biomass temperature during pyrolysis which in addition to the lack of reliable property data for biomass feedstock such as C_p accounts for the discrepancies in the calculations and the difficulty in quantifying the specific heat and reaction heat Q_p.

However, the calorific requirement of biomass can also be deduced experimentally by DSC thermal analysis by integrating the DSC curves obtained during thermal analysis as expressed mathematically in Eq. 2 (He *et al*., 2006):

$$Q\big|_{m,o} = \int_0^t \big|_{m,o} m_b C_{p,b} \frac{dT}{dt} + m_b \dot{H}_P \, dt \qquad (2)$$

where, Q is calorific requirement; $Cp_{,b}$; Cp_{ch}; specific heat capacity of biomass and char respectively; $m_{,b}$; $m_{,ch}$; mass of biomass and char respectively; Q_p, reaction heat of biomass; H_p heat flow due to reaction heat of biomass pyrolysis; dT, temperature change; t, time of DSC experiment.

RESULTS AND DISCUSSION

Proximate analysis and heating value: The results of the proximate analysis (wt. %) and heating value (HHV) of the EFB briquette are presented in Table 1. The FB briquette contains a high percentage of volatile matter (>70%), low moisture content (<10%), ash content (<5%) and fixed carbon (<20%).

The low moisture content (8.17%) is an indication that the thermal conversion efficiencyof the fuel will be considerably high. This is consistent with the findings of Basu (2010) which show a direct correlation between moisture content and the heating value of biomass fuels. Furthermore, the low EFB briquette ash content (4.56%) indicates a low risk of slaggingand fouling of the thermal conversion equipment during conversion.The ratio of volatiles to fixed carbon ratio (VM/FC) for the fuel was 4.65, which is in good agreement with the typical values of >4.0 for biomass species.

The HHV of the EFB briquette (~18 MJ kg^{-1}) is lower than the values for coal, due to the low fixed carbon content, high oxygen content and low density of the fuel (McKendry, 2002; Basu, 2010). Thehigher heating value (HHVs) of the fuel was also calculated from the proximate analysis using the Demirbas formulae Eq. 3 as a function of Fixed Carbon (FC) (Demirbas, 1997):

$$\text{HHV} = 0.196\,(\text{FC}) + 14.119 \qquad (3)$$

The calculated HHV value is comparable to the experimental HHV of the EFB briquette sample and can therefore be used as an acceptable approximation of the measured calorimetric value.

DSC thermal analysis: The normalized DSC curve for the pyrolysis of the EFB briquette is presented in Fig. 2. The

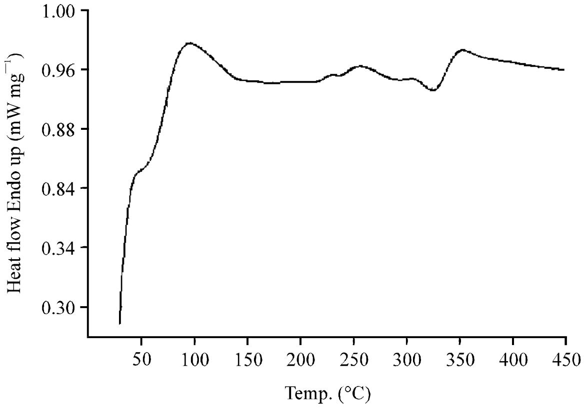

Fig. 2: DSC curves of the pyrolysis of the EFB briquette

Table 1: Properties of EFB briquette

Fuel	Moisture content$_{ad}$	Volatile matter$_{ad}$	Ash content$_{d}$	Fixed carbon$_{ad}$	HHV (MJ kg^{-1}) E	HHV (MJ kg^{-1}) C
EFB briquette (wt. %)	8.17	71.83	4.56	15.44	17.57	17.05

ad: Air-dried basis, d: Dry basis, E: Experimental, C: Calculated graph only presents the pyrolysis reaction profile of the EFB from 30 to 450°C as no visible mass loss was observed beyond this end temperature. From the DSC curves, it can be deduced the pyrolysis process occurs in stages as indicated by the multiple DSC peaks observed in Fig. 2. Studies by Stenseng *et al.* (2001); He *et al.* (2006); Basu (2010) showed that pyrolysis occurs in four stages namely:

- Drying (~100°C),
- Initial stage (100-300°C) for heating of biomass
- Intermediate stage (200-600°C) for biomass degradation and char aggregation
- Final stage (300°C>) for secondary cracking of volatiles into char and non-condensable gases

However, there are no distinct boundaries between these processes during biomass (Basu, 2010) pyrolysis.

In addition to heating rate and residence time, temperature plays an important role in the biomass pyrolysis. During pyrolysis the biomass fuel is heated to a maximum (peak) temperature known as the pyrolysis temperature, an important parameter that influences the composition and yield of the pyrolysis product (Basu, 2010).

Table 2 presents data on the onset, peak and burnout temperature of the different stages of the pyrolysis of EFB briquette during DSC thermal analysis. The drying stage of the pyrolysis process is due to the moisture content of the EFB briquette as presented in Table 1. The onset of drying was observed at 58°C however moisture removal was completed at 140°C. During the second stage heating of the biomass (140-220°C) no peaks were observed during the process indicating there is no significant weight loss in the briquette during this stage of pyrolysis.

However, during the degradation process (220-330°C), three endothermic peaks where observed during heating. This may either be due to non-inform distribution of fuel particles of different sizes observed in the sample or the effect of sample weight on the DSC analysis as size and distribution of fuel particles can influence heat and mass transfer during pyrolysis.

The thermal decomposition of EFB briquettes fuel particles began at 220°C (with a maximum mass loss rate at 260°C) which is in agreement with the findings of Yang *et al.* (2004) for EFB fiber (raw material for making EFB briquettes). The degradation of fuel particles in the temperature range (220-330°C) can be attributed to the decomposition of hemicellulose and cellulose (Yang *et al.*, 2004; Basu, 2010). A single endothermic peak was observed for the heating and aggregation of char formed during the pyrolysis process. The decomposition of lignin is the main process occurring during this final stage of pyrolysis (Basu, 2010). The onset of char aggregation began at 328°C with a characteristic peak at 344°C. The temperature of the char aggregation of EFB briquette is the highest temperature achieved during the pyrolysis process and is therefore the pyrolysis peak temperature of the fuel.

Table 2: Temperature profile of EFB briquette pyrolysis

	Temp. (°C)		
Pyrolysis stage	Onset	Peak	Final
Drying of biomass	58	70	140
Heating of biomass	140	-	220
Biomass degradation	220	260	330
Char aggregation	328	344	450

From the DSC analysis, the calorific enthalpy and specific heat capacity of the fuel was deduced. Mathematically, this is expressed as (Brown, 1938):

$$\Delta H = \int_{T_1}^{T_2} C_p \, dT \tag{4}$$

where, C_p is the specific heat capacity of the fuel which can be calculated from the calorific enthalpy ΔH of the fuel. From the MS EXCEL calculations and analysis, the value of $\Delta H = 1080\ J\ g^{-1}$. The calculated $Cp = 1397\ J\ kg^{-1}\ K^{-1}$. It should be noted that the value of calorific requirement was calculated based on the initial weight of the briquette fuel. According to (He *et al.*, 2006) the heat required for drying the sample is an integral part of the calorific requirement of biomass pyrolysis.

Fourier transform infra-red spectroscopy (FTIR): The typical products of biomass pyrolysis are gases, liquids and solids depending on the heating rate, temperature and residence time as represented in Eq. 5 (Basu, 2010):

$$\text{Biomass} \xrightarrow{\text{Heat}} \text{Gas}(H_2, CO, CO_2, C_mH_n) + H_2O + \text{tar} + \text{char} \tag{5}$$

The elemental and functional group composition of the fuel was analyzed using FTIR spectroscopy. According to the study by Bassilakis *et al.* (2001), the distribution of elements and functional groups in the fuel can be a vital tool for predicting the distribution and composition of the pyrolysis products. The FTIR spectra for the EFB briquette fuel is presented in Fig. 3.

The broad O-H stretching vibrations between 3200 and 3400 cm^{-1} and 1050 and 1150 cm^{-1} indicate the presence of alcohols. The medium intensity band

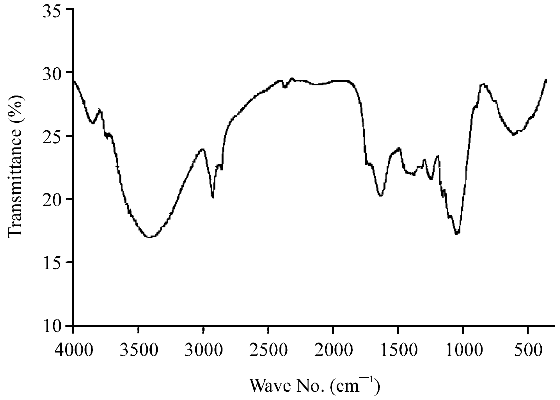

Fig. 3: FTIR spectra of EFB briquette

observed in 2368 cm^{-1} may be due to nitrile C-N functional group, possibly indicating the presence of nitrogen in the elemental composition of the fuel. However, this can only be confirmed by a detailed ultimate analysis. The FTIR spectraalso indicated two bands of strong intensity in the region 2800 and 3000 cm^{-1} typical of C-H stretching vibrations found in CH_3 and CH_2 and the C-H deformation vibrations usually observed between 1350 and 1475 cm^{-1} for alkenes. The broad intensity band between 1600 and 1750 cm^{-1} indicates the presence of C = O stretching vibrations typical of ketones and aldehydes. In addition the bands observed between 1000 and 1300 cm^{-1} may also be due to the presence of ether groups. The weak absorption bands observed between 600 and 900 cm^{-1} of the finger print region could not be assigned to specific functional groups or elements. From the FTIR analysis of the fuel, the following gases as H_2, CO, CO_2, CH_4, C_mH_n can be reasonably predicted for the pyrolysis of the fuel analyzed.

SEM analysis: Figure 4 presents the high resolution SEM micrographs of EFB briquette at a magnification of 50x and 100x. This was carried out to analyze the surface structure, composition and average particle size distribution of the fuel before pyrolysis.

After grinding, the fuel particles were sieved using 800 micron Retsch sieve to obtain particles less than 0.8 mm. However, the surface and particle size distribution analysis using SEM micrographs indicated the presence of a combination of spherical, cylindrical shaped agglomerates, fibers and asymmetrically dispersed fuel particles ranging in size from 98 to 318 μm. The fuel particles were grouped in multiples of 50 from 0 to 350 μm as shown in Table 3. From the SEM micrograph (magnification×50) it was observed that 57% of the fuel surface comprised particles in the 151-250 μm range, 14% of the particles were greater than 251 μm and 7% below 100 μm.

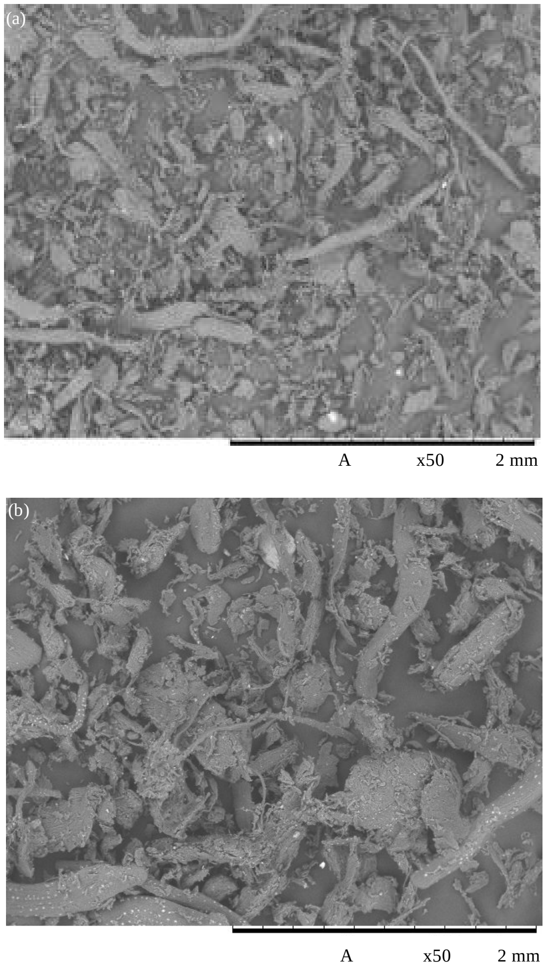

Fig. 4(a-b): SEM micrographs of EFB briquette at (a) 50x and (b) 100x magnification

Table 3: Particle size distribution of EFB briquette

Particle size distribution (μm)	Distribution at x50 (%)
0-50	0.00
51-100	7.14
101-150	21.43
151-200	28.57
201-250	28.57
251-300	7.14
301-350	7.14

The presence of agglomerated fuel particles may be due to the influence of the briquette binder added during briquette manufacture or the high lignin content ~22% is found in EFB fiber (Saka, 2005). It is generally accepted that particle size plays an important role in heat transfer during pyrolysis. Consequently, small sized fuel particles usually characterized by large surface areas are heated faster than large sized particles. This is corroborated by Di Blasi (1996) whose findings showed that gas yield and composition are influenced by heating rate of biomass particles.

Lv *et al.* (2004) showed increased gas yield, heating value, carbon conversion efficiency for smaller sized fuel particles. Conversely, the multiple peaks observed in the DSC can be explained by the irregular distribution of fuel particles of varying sizes resulting in a non-uniform heating profile during pyrolysis.

CONCLUSION

The HHV, DSC analysis, SEM microscopy of the EFB briquette was carried out. The specific heat and the calorific requirement for EFB briquette pyrolysis was calculated from DSC analysis. It was observed that 57% of the surface of the fuel consisted of particles in the 151-250 μm range. The presence of these large particles is a limiting factor for heat and mass transfer during pyrolysis. FTIR analysis was used to predict the pyrolysis gas products. The fuel properties observed indicates that the EFB briquette can be utilized as a fuel for biomass pyrolysis.

ACKNOWLEDGMENTS

The financial support of the GUP Grant, VOT No. 02J94 from the Ministry of Higher Education (MOHE), Energy Research Alliance (ERA) and UniversitiTeknologi Malaysia (UTM) and the assistance of the Research Management Centre (RMC) is greatly appreciated.

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