Jurnal Mekanikal
June 2010, No. 30, 77 - 86

MICROWAVE PYROLYSIS OF OIL PALM FIBRES

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ABSTRACT

Malaysia and Indonesia are generating millions of ton of oil palm fibres (OPF) from their oil palm mills as biomass solid wastes which needs proper waste utilization application. The main purpose of the present research was to pyrolyse the OPF biomass into bio-oil using microwave irradiation technique. A domestic microwave of 1000 W and 2.45 GHz frequency was modified to accommodate fluidized bed system. It was found that OPF showed poor microwave absorbing characteristics. Therefore, an appropriate microwave-absorbing material such as biomass char was added to initiate the pyrolysis process. Temperature profiles and bio-oil yield was investigated by varying the ratio of OPF to microwave absorber. It was found that the yield of bio-oil depended on the ratio of OPF to microwave absorber. Particular attention on the temperature profiles was also taken into account during microwave heating of OPF. It can be concluded that microwave technique can save significant time and energy through its rapid and volumetric heating characteristics.

Keywords: *Fast pyrolysis, biomass, microwave heating, oil palm fibres, bio-oil.*

1.0 INTRODUCTION

Rapid heating methods are being developed to convert biomass waste into a value added products. One of such is the microwave heating technique whereby the microwave energy is deposited directly inside the materials which create instantaneous heat. On the other hand, conventional heating methods demonstrate inverse temperature profile whereby the heat is transferred from surface towards the core of the material. They also suffers from certain drawbacks such as heat transfer resistance, heat losses to surrounding, utilization of portion of heat supplied to biomass materials, damage to reactor walls due to electric heating etc. Furthermore, long heating duration results in an undesired or secondary reaction in conventional heating. These secondary reactions are responsible for inferior product quality owing to further cracking of vapor product. Therefore, novel,

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clean and effective biomass technologies are needed to exploit carbon credit mechanism of biomass [1].

Microwave heating technique has gained interest in recent decades for waste treatment such as plastic [2], sewage sludge [3], and biomass [4-7]. The key merit of the microwave induced pyrolysis is the prevention of undesired secondary reactions that leads to formation of impurities in the product by decreasing the yield of desired compounds [8]. Moreover, microwave heating is an attractive technique since it provides volumetric heating mechanism at improved heating efficiencies compared to conventional techniques [9]. Another important characteristic of microwaves is the selective heating of materials whereby no energy is wasted [10].

The fast pyrolysis system has already been developed in our laboratory to obtain bio-oil from various biomasses [11]. Fast pyrolysis as one of the thermo-chemical process converts the biomass into bio-oil (liquid), char (solid) and flue gas in the temperature range of 400 to 600 °C and in absence of oxygen. Through fast pyrolysis the yield of the liquid i.e. bio-oil or bio-fuels is maximized depending on the process conditions. A variety of conventional pyrolysis systems used till date has been covered in publication [12]. Recently microwave pyrolysis has gained momentous attraction because of the earlier mentioned merits. Existing research works on microwave pyrolysis of biomass includes pyrolysis of wheat straw [1], corn stover [4], rice straw [5], fir sawdust [6], biomass [7], coffee hulls [13], and pine wood sawdust [14]. To our knowledge, till date there has been no published work on microwave pyrolysis of oil palm fiber (OPF). Particularly, the effect of microwave absorber on the temperature profile and yield of bio-oil was found lacking in the literature. Therefore, it was interesting to know the performance of OPF under microwave irradiation.

The objective of the present research work is to reveal the conversion of electromagnetic energy into thermal energy to obtain bio-oil from biomass. Moreover, the effect of biomass to microwave absorber such as carbonaceous char ratio in present study on the yield of bio-oil was the main aim of the study. Further, temperature profile of the oil palm biomass under microwave radiation and characterization of the bio-oil produced was given a particular attention. Thermo-gravimetric analysis (TGA) of OPF has also been presented.

2.0 EXPERIMENTAL PROCEDURES

2.1 Materials

Table 1: Proximate analysis of oil palm biomass

Biomass	Volatile matter, wt %	Fixed Carbon, wt %	Ash content, wt %	Moisture, wt %
OPF [15]	76.2	16.5	0.95	6.34
OPF [16]	72.8	18.8	8	-

As received oil palm fiber (OPF) biomass was obtained from Kulai oil palm mill situated in Johore state of Malaysia. The proximate analysis of OPF is presented in Table 1. Further, the lignocellulosic content in the OPF is also given in Table 2.

Table 2: Lignocelllulosic constituents of oil palm solid biomass materials [17]

Biomass	Cellulose	Hemicellulose	Lignin
Oil palm shells	31.0	20.0	49.0
Oil palm fibres	40.0	39.0	21.0
Oil palm empty bunches	40.0	36.0	24.0

2.2 Experiment set-up

Figure 1 depicts the experimental set-up of the modified microwave pyrolysis system of 1 kW and 2.45 GHz frequency. Microwave cavity was modified in order to accommodate the fluidized bed quartz glass reactor (0.1 m I.D. × 0.15 m height) facilitated with perforated steel distributor plate of 1 mm holes. Glass condensers in series were used to cool down the generated vapors during pyrolysis process into bio-oil. Temperature of the process was measured using two K-type metallic thermocouples connected to Pico data acquisition system (TC-08 USB) acquired from U.K. and further this was linked to personal computer for continuous recording of real time temperature. Thermocouple T1 was inserted inside the bed region and T2 was held just above the bed surface. Thermo-gravimetric analysis (TGA) of OPF was carried out in a TGA/DTG system from Perkin Elmer, U.S.A. (Diamond TG/DTA and Pyris software) with nitrogen as carrier gas and heating rate of 25 °C/min.

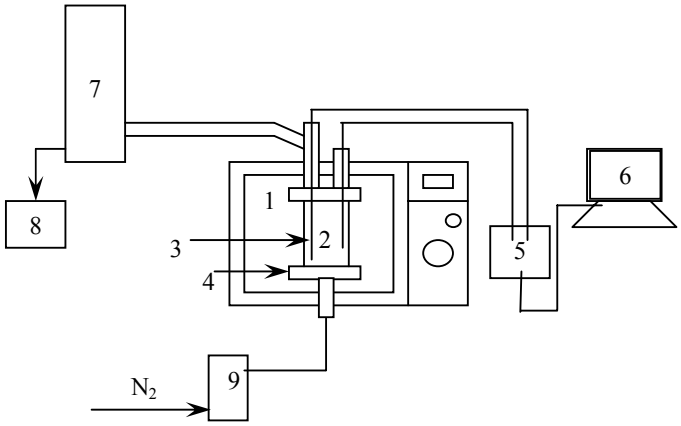

Figure 1: Schematic diagram of microwave pyrolysis process: 1. microwave cavity, 2. quartz glass fluidized bed reactor, 3. thermocouples, 4. distributor plate, 5. temperature data acquisition system, 6. personal computer, 7. condensing unit, 8. bio-oil collector, 9. Rotameter

2.3 Method

For each experiment, OPF was mixed with microwave absorber (in this study biomass char) at different proportions (1:0, 1:0.25, 1:0.5, and 1:1) and charged into the fluidized bed quartz reactor. The microwave absorber was the char which was obtained from conventional fast pyrolysis of oil palm shell biomass of about 100 to 200 μm size. This carbonaceous char can absorbs microwave readily and can act as microwave susceptors for poor or non microwave absorbing materials. Microwave power (450 W) and time radiation of 25 min was kept constant. Before commencement of experiments the fluidized bed reactor system was purged with nitrogen gas at about 20 liter per minute (LPM) to create an inert environment. This inert gas can also help as carrier gas for the vapor generated during the pyrolysis process as well as to avoid any explosion or hazard during the microwave pyrolysis. The vapor generated out of the reactor was condensed using cold water at about 5 to 7 °C. Bio-oil remained in the equipments was also determined via weight difference of the equipments before and after the experiments. Thus total bio-oil, and solid char residue was weighted at the end of the experiments to investigate the yield, while the yield of the flue gas emitted was measured through difference. All the experiments were repeated twice to confirm the values obtained.

3.0 RESULTS AND DISCUSSIONS

3.1 Thermo-gravimetric analysis

The TGA curve as shown in Figure 2 showed initial weight loss in OPF of about 10 wt % till 100 °C and it was due to removal of moisture from the material. It was observed that significant loss in OPF mass started at around 175 °C after which the major decomposition of OPF occurred till 425 °C. After this, the gradual loss in mass occurred until 900 °C. Hence, the TGA profile revealed three steps in loss of mass in the OPF. First was due to removal of moisture, second due to release or burning of volatile matters and lastly the gradual decomposition was because of char burning. This can also be explained by DTG analysis curve as shown in Figure 2. The main reaction took place at the release of volatile matters and it shows two step reaction mechanisms with two peaks in DTG curve from 225 °C to 450 °C.

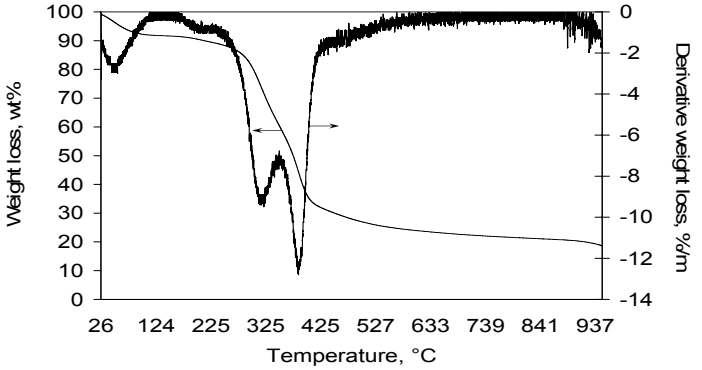

Figure 2: TGA and DTG analysis of OPF

The first peak occurred in the temperature range of 225 to 350 °C while second peak in the range of 350 to 450 °C. However, the maximum rate of mass loss was found at around 320 °C for the first peak while at around 385 °C for the second peak. This can be explained by degradation of lignocellulosic content present in the OPF such as cellulose, hemicellulose and lignin. It was reported that the first step or peak was due to degradation of hemicellulose and followed by the degradation of cellulose for the second step of peak. The gradual loss in mass at the end of the decomposition of OPF can be exhibited mainly due to degradation of lignin. Similar study was performed by Guo and Lua [15] for OPF. However, their study showed some distinct temperature results than present TGA work. This could be due to several reasons such as heating rate, particle size as well as the lignocellulosic content may influence the TGA profiles. Nevertheless, the two step reaction of OPF during volatile release in this study was in agreement with that of literature [15]. It was found that cellulose generally degrade at temperature of around 360 °C [18]. However, it depends on the types of cellulose or biomass.

3.2 Microwave Pyrolysis of OPF

3.2.1 Temperature Profile

Figure 2 shows the temperature profile of OPF microwave pyrolysis without any microwave absorber. It can be observed that absence of microwave absorber i.e. 0 %, results in the maximum bed temperature (T1) to about 80 °C only. Apparently, no pyrolsis or evolution of vapor took place at this stage, even though the water evaporation was noticed. This proves that the OPF was poor absorber of microwaves. The increase in temperature at this stage was due to presence of water in form of moisture inside the biomass. According to TGA profiles at this temperature about 10 wt% of OPF mass may be reduced. Initially the temperature increased linearly but later on the increase was gradual and started to decrease after 20 min of time as can be depicted in Figure 3a and Figure 3b. This is because of water content in the biomass which creates sudden heat in the OPF at initial stage. However, after the moisture was removed there exist no other constituents, which can cause the heat since they do not absorb the microwaves readily.

The addition of microwave absorber i.e. biomass carbonaceous char not only helped in increase of temperature as shown in Figure 4 but also initiated the pyrolysis process by generating the vapors. The commencement of microwaves and generation of vapors were instantaneous. Microwave absorber played a role in absorbing the microwave and transferring it the heat to the OPF materials. A significant increase in the bed temperature, T1 was observed compared to surface temperature T2. This shows that the core or centre of the OPF bed was at higher temperature compared to the surface due to penetration behavior of microwaves into the materials. Therefore, it was reported [19] that the sample internal temperature might be ten or hundred degrees higher than that of surface. Another reason of high temperature might be the physio-chemical characteristics of the OPF. Since OPF are much lighter and thin in diameter or thickness resulting in a high rate of heat transfer in short period of time. Therefore, a sharp increase in temperature to about 1200 °C within few seconds can be observed as shown in Figure 3, which is difficult to achieve in conventionally heated pyrolysis process.

Maximum temperature of about 480 °C was recorded in 7-8 min for microwave pyrolysis of rice straw at 450 W without any microwave absorber or additives [5]. But in the present research work multimode ON/OFF microwave system was used which might have established this temperature profiles. Table 3 shows the average, maximum and minimum temperature recorded during microwave pyrolysis at different ratio of OPF to microwave absorber for 20 mins time interval.

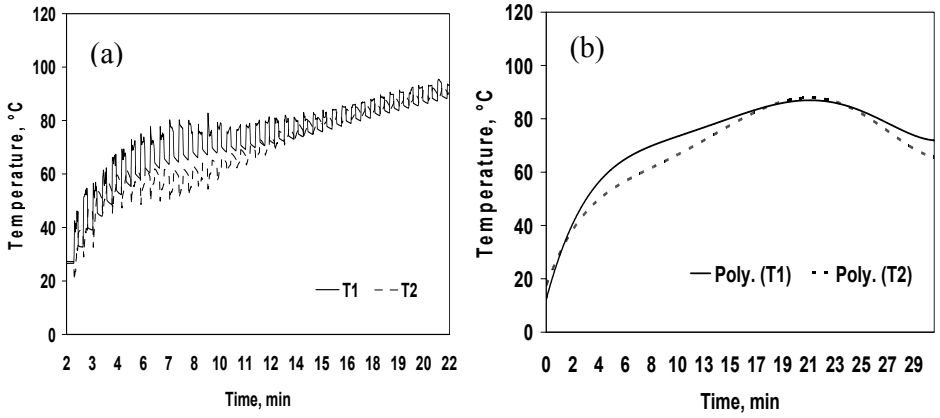

Figure 3 : Temperature profile of OPF under microwave radiation without any microwave absorbers, (a) real time temperature, (b) polynomial regression analysis; Microwave power – 450 W

Domínguez [3] mixed carbon char to achieve high temperature for sewage sludge pyrolysis. They reported that the char obtained from pyrolysis of biomass are good microwave receptors. Other study [20] used carbon grains of about 0.5 wt % with oil shale sample to pyrolyse under microwave radiation to allow uniform temperature or heating. Repeatedly, carbonaceous materials are reported to be good microwave absorbers [9-10]. It was found that in present study biomass char played an effective role in raising temperature to a significant value. Moreover, this biomass char is by-product of the pyrolysis process with cheap cost and environmentally sustainable. The formation of char during pyrolysis process of OPF starts to absorb more microwaves readily and forms a sustainable process.

The sinusoidal nature in the temperature profiles of Figures 3 and 4 shows the discrete or cyclic ON/OFF working nature of the microwave. Basically, all domestic microwave system works in this type of mode. This means that the temperature increases to higher value when the magnetron is in ON mode and drops to a certain level when the magnetron is in OFF mode. Hence, the polynomial regression analysis of was conducted on the temperature profiles as shown in Figure 4. The maximum temperature attended at 1:0.25 OPF to microwace absorber ratio was around 800 °C while at 1:1 ratio it was around 225 °C. Lower temperature profile at higher OPF to microwave absorber might be due to localized heating or hot spot which are common in multimode systems.

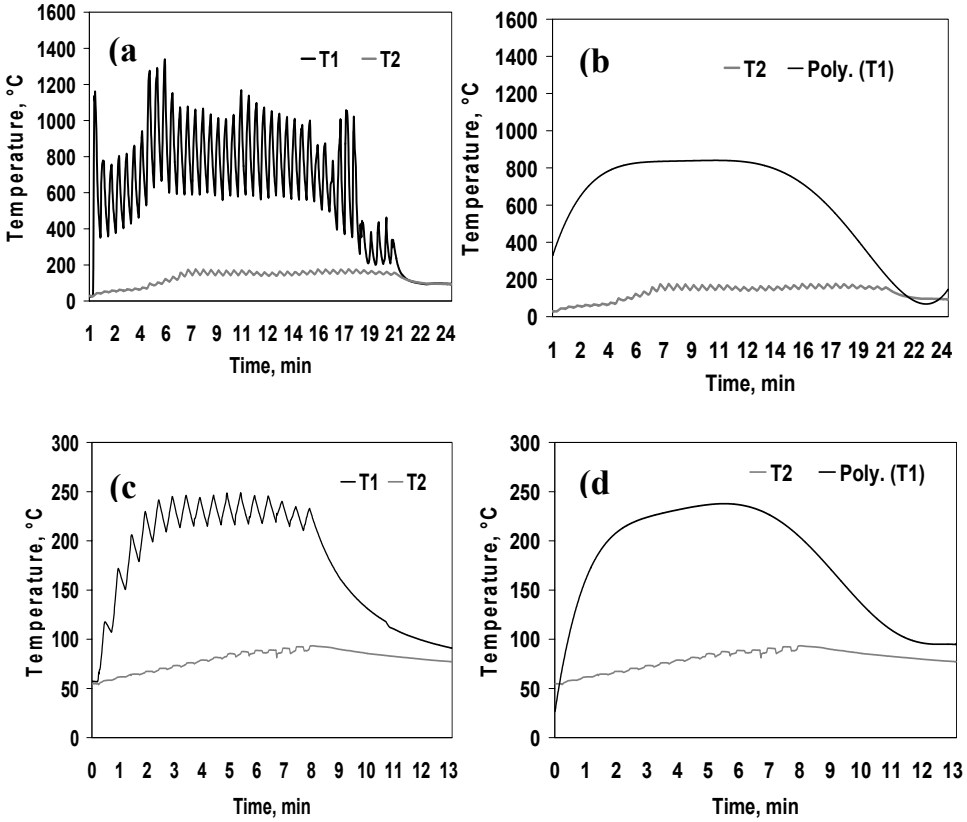

Figure 4: Real time temperature profile of OPF biomass to microwave absorber ratio (a) and (b) 1:0.25, (c) and (d) 1:1 at microwave power of 450 W

Table 3 : Average, maximum and minimum temperature at different biomass to microwave absorber ratio

Ratio MA:OPF	**Tavg*, °C**		**Tmax*, °C**		**Tmin*, °C**	
	T1*	**T2***	**T1**	**T2**	**T1**	**T2**
1:0	72	70	95	94	26	20
1:0.25	597	125	1340	176	27	27
1:0.5	560	140	1365	1000	27	25
1:1	177	79	249	93	57	54

*: Tavg - Average temperature; Tmax - Maximum temperature; Tmin - Minimum temperature; T1 – Bed inside temperature; T2 – Bed surface temperature

3.2.2 Effect of microwave absorber on the product yield

The amount of microwave absorber in the OPF biomass influenced the pyrolysis products yield as shown in Figure 5. Thus, maximum bio-oil yield was obtained at biomass to microwave absorber ratio of 1:0.5. It was assumed that as the amount of microwave absorber is small, OPF could not get enough heat to pyrolysis the material. Conversely, higher amount of microwave absorber might have lead to localized heating of char or OPF material alone. Nevertheless, temperature at which the pyrolysis takes place also plays an important role in the product yields [13]. Yet the exact reason for this has to be investigated and understood since the heating characteristics of microwave is very rapid. Similar results of microwave absorption media influence on pyrolysis of fir sawdust was found by [6]. In their study ionic liquid and glycerol was used as microwave absorber to conduct the pyrolysis in domestic microwave and optimal fir sawdust to ionic liquid ratio at which highest bio-oil yield was obtained was about 1:2. This ratio was much higher compared to present study i.e. about 1:0.5. Presence of minerals in the biomass char might enhance the microwave absorbivity.

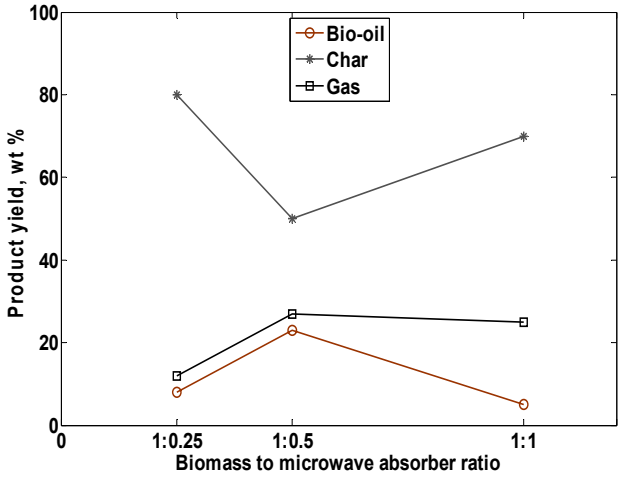

Figure 5: Effect of microwave absorber on yield of OPF microwave pyrolysis product

4.0 CONCLUSIONS

Microwave pyrolysis of as received OPF biomass was carried out to produce bio-oil. Sinusoidal temperature profiles were observed during microwave pyrolysis of OPF in a domestic microwave system due to cyclic operation of magnetron. Microwave absorbers were found to increase the pyrolysis temperature largely within seconds thus saving considerable time and energy. Further, it also helped to initiate the pyrolysis process since without the microwave absorber no pyrolysis process took place. Product yields were observed to be dependent on the OPF to microwave absorber ratio. Thus, microwave pyrolysis can be successfully applied to produce bio-oil from OPF and other related biomass with the help of

microwave absorbers such as biomass char which can become a sustainable process.

ACKNOWLEDGEMENTS

The authors are grateful for Fundamental Research Grant (FRGS) funding from Ministry of Higher Education (MOHE), Malaysia to carry out the research activity.

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