

Microwave Induced Pyrolysis of Oil Palm Biomass by using Layer Microwave Absorber in Reverse Flow Double Cylinder Reactor

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ABSTRACT

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Empty fruit bunches (EFB), mesocarp fiber (MF) and oil palm shell (OPS) from oil palm biomass have undergone microwave pyrolysis with layer microwave absorber (MA) method by using reverse flow double cylinder reactor. Microwave pyrolysis has been carried out under fixed microwave power of 800W with parameters of temperatures (400-600°C) and MA loading percentages (30-50wt.%). The performance in terms of products yield and liquid product quality (phenolic compounds) has been studied. Results show that 500°C is the optimum temperature to produce highest liquid pyrolysis product yield for all the three types of oil palm biomass. At the optimum temperature, the highest liquid product yield was obtained at the MA loading percentage of 30wt.% for the case of MF and OPS, while 40wt.% for the case of EFB. The highest liquid product yields were recorded 37.02wt.%, 37.01wt.% and 37.96wt.% for the case of EFB, MF and OPS respectively. Besides, gas yield from EFB, MF and OPS at 500°C lie within the range of 22-34wt.%. Under the optimum temperature and MA loading percentages, total phenolic compound of GS-MS area 78.91% was obtained in the case of EFB while 75.41% and 81.75 % of GC-MS area for the case of MF and OPS.

Keywords:

Empty fruit bunch, mesocarp fiber, oil palm shell, microwave pyrolysis, layer microwave absorber placement, pyrolysis liquid product

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1. Introduction

Being one of the tropical countries with equatorial climate, approximately 85% of total global palm oil production was dominated by Malaysia and Indonesia [1, 2]. The increase in the demands of vegetables oil due to the growth of living standard and population globally has caused the rise of palm oil production in these regions [2, 3]. Interestingly, 90% of the by-product from the palm oil production process consist of solid oil palm waste while only 10% of the product was edible [2]. Hence, the management of such massive amount of solid oil palm biomass is crucial to avoid the

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negative impact of disposal to the environment [4]. Here, recycling as well as energy recovery from the waste has been claimed to be the fundamental principle of general waste management [4].

In recent years, thermochemical process such as microwave (MW) pyrolysis seems to be a potential method to convert oil palm biomass into bio-char, liquid product, and synthesis gas. MW heating in pyrolysis was mentioned to be a rapid, non-contact, and in-core volumetric heating method that greatly reduced the pre-pyrolysis time and heat loss, in which has been encountered in the case of conventional pyrolysis (electric-powered furnace) [5, 6]. However, the application of MW heating in biomass pyrolysis has encountered the problem of low MW absorption capability of biomass. Hence, the application of MW heating that involves oil palm biomass requires microwave absorbers (MA) materials in order to make use of MW as heating source. MA materials actually absorb and convert the MW energy into thermal energy and later the energy is transmitted to the oil palm biomass during pyrolysis [5, 7-15].

In the case of the absence of MA, solid oil palm biomass requires considerable time and MW power to achieve pyrolysis temperature at the low heating rate and this situation shows the inefficient use of MW power [5, 16]. While in the case of intimately mixing of biomass and MA, Mushtaq, Abdullah [17] stated that the hotspot problem has caused the non-uniform heating and subsequently deteriorates the MW pyrolysis process. Previously, researchers [5, 17, 18] have applied the layer MA placement method in MW pyrolysis of OPS and have carried out some parametric studies. The highest liquid product yield of 31.03wt.% has been produced under the MA loading of 35wt.% and MW power of 300W without controlling the pyrolysis temperature. The MW pyrolysis temperature of their study was uncontrolled and therefore, the temperature could raise until 700°C [18]. The results indicate that the layer MA placement has improved the biomass conversion, liquid product production, and product selectivity, in which liquid products were found to be rich in phenol contents [17]. The aim of the present study is to understand the effect of layer MA placement on MW pyrolysis of oil palm empty fruit bunch (EFB), mesocarp fiber (MF) and oil palm shell (OPS) under various temperatures and MA loading percentages in terms of liquid product yield. Temperature profiles of MW pyrolysis of all the three solid oil palm biomass at the optimum condition are presented and discussed. Besides, the liquid product obtained from the optimum temperature and MA loading percentage is characterized by using Gas Chromatography-Mass Spectrometry (GC-MS) analysis to analyse its chemical characteristics.

2. Methodology

2.1 Preparation of Materials

The oil palm biomasses were acquired from an oil palm mill which located in the northern part of state of Johor, Malaysia. Three types of solid oil palm biomass obtained from oil palm mill which includes empty fruit bunch (EFB), mesocarp fiber (MF) and oil palm shell (OPS) have been used as feedstock in the present study. The raw biomass was ground by using an automatic hammer mill grinder and sieved into the particle size of less than 3.38mm. Since biomass is generally recognised as a material with poor MW absorbance, a microwave absorber (MA) material was required in order to achieve the desired temperature more quickly [9]. In the present study, the commercial charcoal activated granulated (Brand: HmbG Chemicals) obtained from a local company has been applied as MA material and it has been cut and sieved to obtain the particle size of 3-5mm. To assure the humidity consistency of the MA and to avoid the excessive water content in the liquid product yield, both sieved oil palm biomasses and MA were dried in the oven for 12 hours before storage. Both the proximate and elemental analysis data for EFB, MF and OPS are tabulated in Table 1 and 2, respectively. Based on Figure 1 (a) and (b), thermogravimetric (TG) and derivative thermogravimetric

(DTG) graph of EFB, MF and OPS have been presented and it can be noticed that the major volatile decomposition of the three oil palm biomass was completed within the temperature range of 400-600°C. The claim was supported by Omar *et al.*, [9] who stated that 120-600°C was the temperature range for the release of volatile matter.

Table 1

Proximate analysis

Oil palm biomass	EFB (wt.%)	MF (wt.%)	OPS (wt.%)
Moisture	1.22	5.813	6.01
Volatile	75.79	63.80	64.00
Fixed carbon	19.73	23.73	26.96
Ash	3.27	5.60	3.04

Table 2

Elemental analysis and heating value of the raw materials

Oil palm biomass	EFB (wt.%)	MF (wt.%)	OPS (wt.%)
Carbon (C)	42.86	42.61	46.86
Hydrogen (H)	6.31	6.09	6.01
Nitrogen (N)	0.78	1.60	0.45
Sulphur (S)	0.50	0.26	0.20
Oxygen (O)	49.55	49.43	46.48
Heating value (MJ/kg)	16.51	17.23	19.53

2.2 Apparatus and Microwave Pyrolysis Experimental Procedures

Experimental rig consists of reactor, thermocouple, N₂ gas supply, pyrolysis vapour condensing unit is illustrated in Figure 2. The internal cylinder was used as the inlet for inert gas injection and thermocouple placement, while oil palm biomass and MA were placed in the space between two cylinders (shaded part) as shown in Figure 3. A thermocouple type-R, temperature data logger unit and a laptop were used to record the real-time temperature throughout the experiments. Moreover, the type-R thermocouple's tip was immersed in the layer of biomass to specifically measure the biomass layer's temperature instead of MA layer.

A fixed amount of 55g of oil palm biomass has been applied as input material throughout all the experiments. The biomass and MA were separated into four layers each and the arrangement is illustrated in Figure 3. The loaded reactor was then inserted from the top of a modified domestic SAMSUNG MW oven with maximum MW power capacity of 800W. Nitrogen (N₂) gas with 99.98% purity was supplied in order to create the inert environment for pyrolysis and at the same time acted as a carrier gas [14]. Initially, 1 liter per minute (LPM) of N₂ gas was injected for purging process up to 15 minutes before adjusted to 0.5LPM for the MW pyrolysis experiment.

During the experiment, the heating temperature of the feedstock was continuously monitored and controlled by a proportional-integral-derivative (PID) thermostat. While for the liquid product collection, two water-cooled glass condensers which connected to a water chiller (Brand: Lauda Alpha RA-8) were placed in between the vapour's outlet and collecting flask in order to condense the pyrolysis vapour into liquid product.

The MW irradiation was set constant for 1 hour and bio-char will be lefted to be cooled to room temperature. After that, the pyrolysis products were collected and weighted while the yields of liquid product and bio-char were determined by using Eq. 1. The yield of bio-gas was obtained from the difference between the total weight of input biomass and summation of the weight of bio-char and liquid product yield.

$$\text{Bio-oil yield (wt.\%)} = \frac{\text{Weight of liquid pyrolysis product}}{\text{Weight of solid oil palm biomass}} \times 100 \quad (1)$$

Liquid product samples were later sent for characterization analysis. In the present study, variation of MA loading percentage and pyrolysis temperature has been carried out on three types of solid oil palm biomasses which are EFB, MF, and OPS for parametric study. Table 3 shows the variation of operating conditions among two manipulated parameters in experiments.

Table 3

Variation of two parameters in the microwave pyrolysis of oil palm biomass

Biomass	MA (wt. %)	Temperature (°C)
EFB, MF and OPS	30	400
		500
		600
	40	400
		500
		600
	50	400
		500
		600

3. Results and Discussion

3.1 Heating Profile

Figure 4 illustrates the real time continuous temperature profile throughout the MW pyrolysis operation of EFB, MF, and OPS. It can be noticed that the temperature profile achieved a better consistency in temperature fluctuation compared to studies that controlled MW power only [5, 7, 8, 10, 12-14, 17-22]. The fluctuation was able to be limited under $\pm 10\%$ from the setting temperature. It is because when the temperature has reached the setting temperature, PID thermostat will cut off the MW power supply and switch on the supply again when the temperature starts to drop under the setting temperature. The situation was similar to the case that altered the MW power, in which experiencing the cyclic ON/OFF due to the factory default setting of domestic MW oven. The situation has also been mentioned in the other microwave pyrolysis studies [5, 7, 8, 10, 12-14, 17-22].

3.2 Microwave Pyrolysis Product Yield

3.2.1 Effect of pyrolysis temperature

According to the graph of thermogravimetric (TG) and derivative thermogravimetric (DTG) shown in Figure 1, major volatile decomposition has completed within the temperature range between 400-600°C. Therefore, in order to determine the temperature for highest liquid product yield, temperature of 400°C, 500°C, and 600°C have been chosen as the range of temperature to be studied.

Figure 5 has demonstrated the pyrolysis products yield that has been obtained under different pyrolysis temperature and MA loading percentages. 500°C was found to be the temperature that produced the highest liquid product yield regardless of MA loading percentage. The highest liquid product yield of approximately 37wt.% has been produced from EFB and MF, while 38wt.% of liquid product was obtained in the case of OPS. The finding was found similar to the study carried out by Abubakar and Ani, in which the temperature of 500°C was considered to be a suitable temperature

for fast pyrolysis and temperature that is lower than that was reported to be the cause of incomplete pyrolysis [21].

On the other hand, the yield of gaseous product was noticed to increase with an increase in temperature. The situation was noticed when the temperature was increased from 400°C to 600°C for all the materials involved in this study. In addition, the finding is on par with the study done by Hossain et al which stated that more hydrogen-rich gas could be produced when there is an increase in pyrolysis temperature [23]. The reason for the gaseous product yield increment was due to the increase in temperature, in which potentially promotes the secondary reaction to be more frequently happened that further converts the volatile vapour into the gaseous products instead of liquid product. This can be observed from the case of EFB under the temperature of 600°C. The lowest char yield (28.9wt.%) and highest gas yield (34.1wt.%) suggest that the high temperature actually causes an increase in gaseous product yield. By comparing the case of 500°C and 600°C under the same MA loading of 40wt.%, the increment in gas product further confirms the occurrence of pyrolytic vapours secondary cracking.

In contrast with the trend found in the case of gaseous products, the char yield was found to decrease with an increase in temperature for all types of oil palm biomass. As mentioned above, the increase in pyrolysis temperature is able to produce higher gaseous products yield while it actually reduces the char formation at the same time [23-25]. If compared to the increment in gaseous product yield, the temperature increment from 400°C to 600°C has caused more significant reduction in terms of char yield (wt.%). The results indicate that there is slightly greater effect of temperature on the primary biomass decomposition rather than enhancement in secondary reaction (volatile vapour to gaseous product). If compared to the case of EFB and MF, the case of OPS shows slightly higher in terms of liquid product yield and bio-char yield, that is probably because OPS has comparatively lower ash content [26] and higher fixed carbon content, as shown by Table 1 (proximate analysis of the raw OPS).

3.2.2 Effect of microwave absorber layers loading percentage

In the present study, the effect of MA loading percentage as a function of temperature was investigated. In general, optimum MA loading percentage is unable to be generalized for different types of biomass due to the difference in terms of MA placement method and biomass physio-chemical characteristics [10, 14]. In the present study, longer time was taken to achieve the desired temperature when there was an increased in layer MA loading percentage. This situation has been consistently observed in all the experiments carried out in the present study. There was a delay of not more than two minutes to achieve the desired temperature when MA loading was increased from 30wt.% to 50wt.%. In contrast, Mushtaq, Abdullah [17] have found that the effect of increase in MA loading on the heating rate was not significant in their optimization study. However, different studies that used the technique of mixing up the biomass with MA have revealed that the increase in MA loading tends to increase the heating rate but the rate started to decrease at sufficiently high MA loading (75wt.% loading) [18]. It is shown that the difference in MA-biomass configuration and operating conditions affect the pyrolysis heating rate.

Among the three oil palm biomass, OPS has achieved the highest heating rate for all the three MA loading percentages under the optimum temperature of 500°C. It is because OPS is physically thicker than the other two fibrous biomass. Hence, the OPS has higher tendency to generate superheated steam pressure from its bound moisture content within the biomass when interacting with microwave irradiation. The result was supported by the studies done by Mushtaq [27] where heating rate comparison under MW irradiation was made between the OPS nuts and powdered OPS.

Besides, 40wt.% of MA loading percentage was found to be the optimum to produce highest liquid product yield in the case of EFB. Here, the achievement in terms of highest liquid product yield was consistent across the temperature range of 400-600°C. Meanwhile, both MF and OPS have achieved the highest liquid product yield at 30wt.% of MA loading while 50wt.% of MA loading did not show any significant improvement in terms of liquid product yield in the present study. In most cases, slightly reduction in liquid product yield could be observed for the case of 50wt.% MA loading. The decrease in liquid product yield is supposed to be caused by the excessive MA loading that leads to hotspot that happened at MA layer. Hotspot is a common issue that often reported in the previous studies that applied the intimately mixing of both biomass and MA in the reactor [18].

The comparison among several studies with highest liquid product yield achieved in different MA placement method is shown in Table 4. Based on the comparison, it was found that liquid product yield obtained from the present study has been improved by using the layer MA placement. The improvement was most obvious for the case of EFB, in which approximately 16wt.% of improvement has been noticed if compared to the intimately mixed method. In addition, the layer MA method has demonstrated its potential even though there are more factors like carrier gas flow rate, biomass particle size, MW power, biomass layer thickness and other operating conditions are yet to be tested and optimized.

Table 4

Comparison in terms of bio-oil yield between present and the past studies that operated under different MA placement methods.

MA placement method	Highest bio-oil yield (wt.%)		
	EFB	MF	OPS
Present study	37.02	37.01	37.96
Intimately mixed	21.00 ²⁰	23.22 ^{15,19}	31.47 ²²
Stirred bed	-	-	28.00 ^{21,28}
Not using MA	27.22 ³²	-	-

3.3 Liquid Product Characterization

In the following sections, the results of the physical characterization and Gas chromatography–mass spectrometry (GC-MS) analysis for liquid products obtained from the optimum temperature and MA loading percentage (in terms of highest liquid product yield) are presented and discussed.

3.3.1 Physical characterization

The liquid products that produced from the MW pyrolysis of EFB, MF and OPS in the present study were found to be non-homogeneous, in which two visibly layers were noticed. The watery upper layer appeared to be dark brown in colour while the more viscous lower layer is black colour. Moreover, the upper layer liquid product that made up of at least 71% seems to be the major constituent of all the liquid products obtained from the present study. The weight percentage of the lower layer of the liquid product of EFB, MF and OPS were 19.25wt.%, 18.08wt.%, and 28.52wt.%, respectively.

In terms of viscosity, the upper layer of liquid product was found to be much less viscous if compared to the lower layer, in which numbers of studies have called it as the aqueous phase [29] or watery phase [28]. In the present study, the viscosities of the upper layer liquid product were found to be 1.85cP, 2.23cP and 2.35cP for EFB, MF and OPS respectively at the temperature of 27°C. The results seem to be more viscous if compared to the previous conventional fixed bed pyrolysis

study that has also produced inhomogeneous liquid product, in which 1.7cP (for EFB), 1.5cP (for MF) and 1.9cP (for OPS) obtained [30]. Meanwhile, in the present study, the viscosity of the lower layer liquid product of EFB, MF and OPS produced were recorded as 24.09cP, 24.71cP and 25.13cP, respectively, which is more viscous if compared to upper layer liquid product. In terms of density, both upper and lower layers of liquid product produced from all types of oil palm biomass have higher density if compared to heavy fuel oil which typically recorded as 855 kg/m³. The densities of all the liquid products produced in the present study were fall within the range of 1044-1069kg/m³ for upper layer and 1090-1106kg/m³ for lower layer liquid product. The relatively high density liquid product was claimed to be due to the presence of oxygen in the heavy organic molecules while phenol is one of the example.

On the other hand, relatively higher water content was found in the upper layer of the liquid product if compared to the lower layer. In the case of EFB, 33.43% of water content was obtained from the upper layer while 8.93% for the lower layer. The high water content in the upper layer leads to the undefined heating value due to the ignition failure during the operation by using the bomb calorimeter. Furthermore, high water content in the sample causes the dilution in energy density of the upper aqueous layer of liquid product [31]. The situation can be observed from the heating values obtained for both liquid product layers in the previous conventional pyrolysis study in which the value were recorded as 12.18MJ/kg (for upper layer) and 27.97MJ/kg (for lower layer) [30]. In the present study, the lower layer of liquid product for the case of OPS contained highest amount of water (14.12%) and that has caused the liquid product heating value to be the lowest (23.94MJ/kg) if compared to EFB (25.11MJ/kg) and MF (25.65MJ/kg). Overall, the heating value of the liquid product produced in the present study is slightly lower if compared to the inhomogeneous liquid product produced by the conventional pyrolysis [28, 30].

3.3.2 Gas chromatography-mass spectrometry characterization

The chemical analysis of liquid product produced from EFB, MF and OPS is depicted in Table 5. Both upper and lower layer of liquid product have been separated and sent for GC-MS analysis to determine their chemical composition. The results show that the phenol content in the liquid product produced by MW pyrolysis of EFB, MF and OPS were generally higher than that if compared to the conventional pyrolysis [32-39]. This finding has shown the potential of using layer MA placement in MW pyrolysis to provide better performance in phenol production as replacement for petroleum-based phenol.

Phenol content in the lower layer liquid product (viscous layer) was higher if compared to upper layer liquid product. Phenolic compounds noticed in the liquid product were actually the product of lignin decompositions [14]. Therefore, due to the comparatively higher lignin content in raw OPS [30], highest phenol content of 47.52% was obtained among the three types of oil palm biomass and followed by 39.32% and 37.48% for the case of MF and EFB, respectively. Other than phenol, 2,6-dimethoxy-phenol was also found to be one of the major chemical compounds. The increase in MA loading from 30wt.% to 50wt.% tends to increase the total phenolic content in the liquid product but fluctuation trend is noticed in phenol content. The increasing trend of phenolic content is suggested to be caused by the catalytic effect of MA and it has been proven that the application of layer MA placement still can produce the quality liquid product if compared to the mixed method.

Table 5

Chemical compounds in upper and lower layer of bio-oil from MW pyrolysis of EFB, MF and OPS under optimum temperature of 500°C (maximum bio-oil yield).

MA loading percentage	EFB		MF		OPS	
	40wt.%		30wt.%		30wt.%	
Chemical compounds	% area		% area		% area	
Bio-oil layer	Upper	Lower	Upper	Lower	Upper	Layer
Phenol	14.95	37.48	28.74	39.32	33.77	47.52
2-methyl-phenol	1.76	4.61	3.82	3.78	4.21	2.26
3-methyl-phenol	3.03	7.88	3.24	5.90	-	-
4-methyl-phenol	-	-	-	-	-	3.17
2,3-dimethyl-phenol	-	1.56	-	-	-	-
2,4-dimethyl-phenol	0.50	-	0.83	2.15	-	-
2,5-dimethyl-phenol	-	1.08	-	-	-	-
2,4,6-trimethyl-phenol	-	-	-	1.92	-	-
2-methoxy-phenol	2.87	4.50	4.48	5.78	4.79	6.66
3-ethyl-phenol	1.19	2.90	-	2.47	-	-
4-ethyl-phenol	-	2.15	1.53	-	-	-
2-methoxy-4-methyl-phenol	0.89	-	2.32	3.38	3.38	5.06
2-methoxy-4-propyl-phenol	-	1.24	-	0.81	-	1.12
2-methoxy-4-(1-propenyl)-(E)-phenol	-	1.68	-	-	-	1.95
4-ethyl-2-methoxy-phenol	1.29	4.15	1.89	3.57	2.07	5.06
2,6-dimethoxy-phenol	3.50	7.84	5.48	6.33	6.30	6.94
2,6-dimethoxy-4-(2-propenyl)-phenol	-	-	0.64	2.44	-	2.01
3,4,5-trimethyl-phenol	-	1.84	-	-	-	-
Total phenolic compound	29.98	78.91	52.97	77.85	54.52	81.75

4. Conclusions

The ability to hold the temperature within temperature fluctuation of $\pm 10\%$ throughout the pyrolysis process has shown the feasibility of the layer MA placement in MW pyrolysis of EFB, MF, and OPS. All setting temperatures of 400°C, 500°C, and 600°C were achieved after mere 6.5 minutes. This situation has shown the capability of layer MA placement to provide relatively high heating rate in order to promote vigorous volatile release that is preferable for liquid product production. Besides, 500°C was found to be the optimum temperature in terms of highest liquid product yield. This finding has shown the reliability of layer MA placement method to rise the temperature up to 500°C with minimum hotspot. Hotspots are often been noticed in the case of intimately mixed MA placement that creates the problem of serious uneven heating issue.

Liquid pyrolysis product yield obtained in the present study was noticed to be higher than previous studies that applied intimately mixing method and stirred bed method. The situation shows the potential of layer MA method to minimize the uncertainty caused by hotspot problem that often occurred in other MA placement method. Besides, phenol-rich liquid pyrolysis product has also been produced from EFB, MF and OPS MW pyrolysis in the present study. Phenol and total phenolic compounds were noticed to increase with an increase in MA loading due to the catalytic effect. Meanwhile, the highest phenol contents of 37.48%, 39.32% and 47.52% were obtained in the liquid product for EFB, MF and OPS, respectively under optimum temperature and MA loading. Although

every layer of oil palm biomass seems to be pyrolysed by observation, the pyrolysis level in each layer has not been focused in the present study. Hence, the temperature across every layer of biomass would be one of the main subjects to be focused in the future.

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Appendix A: Figures

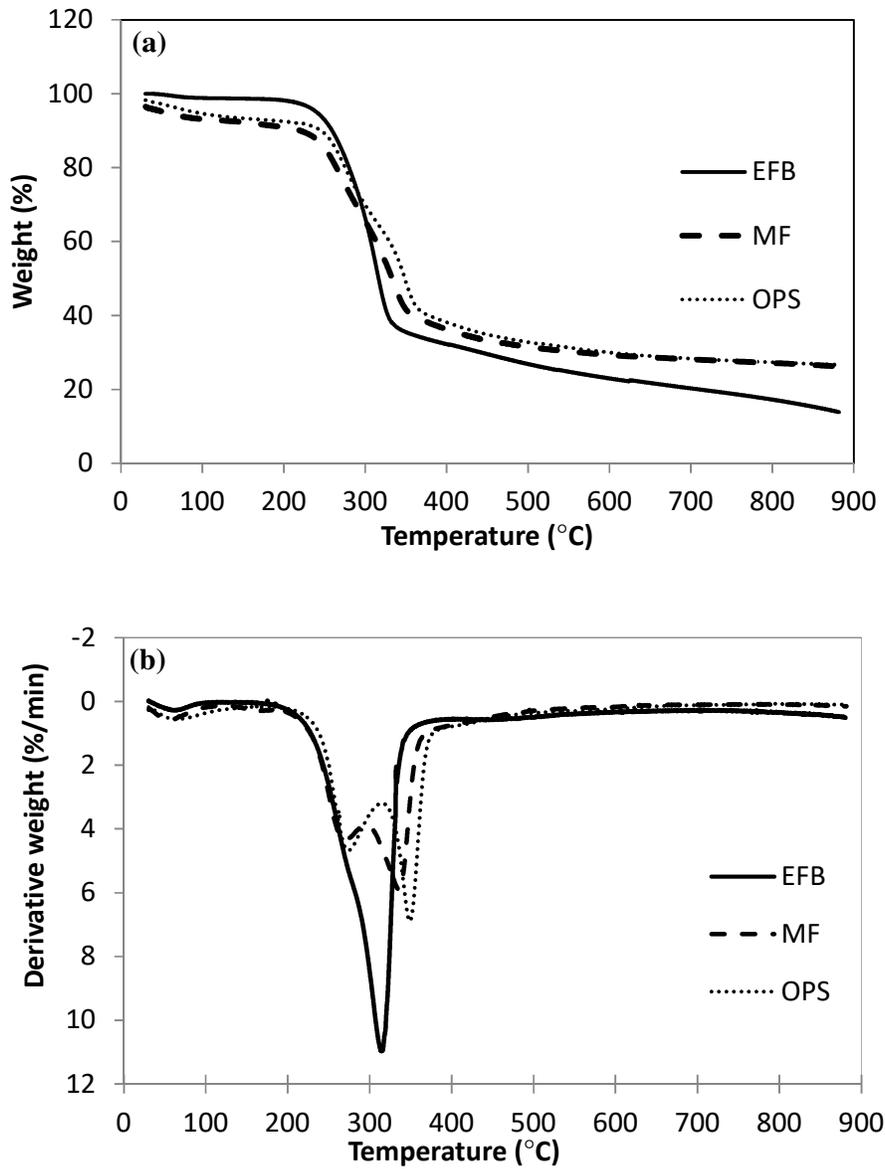


Fig. 1. (a) Thermogravimetric (TG) and (b) derivative thermogravimetric (DTG) curve for the raw EFB, MF and OPS

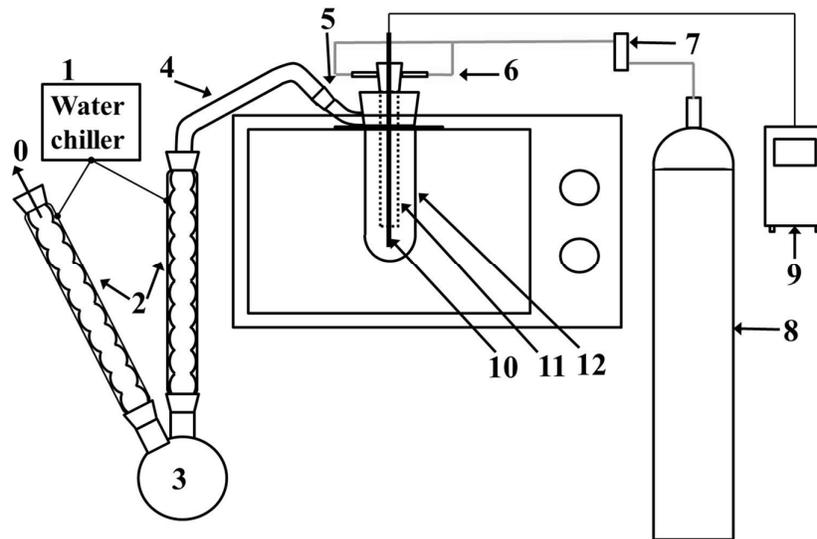


Fig. 2. Schematic diagram of the microwave pyrolysis of oil palm biomass by using reverse flow double cylinder reactor: (0) Gas outlet; (1) Water chiller; (2) Glass condensers; (3) Bio-oil collecting flask; (4) Glass connecting tube; (5) Vapour outlet; (6) N₂ gas inlet; (7) Flow meter; (8) N₂ gas tank; (9) Proportional-integral-derivative (PID) thermostat; (10) Thermocouple; (11) Internal reactor cylinder; (12) Outer reactor cylinder.

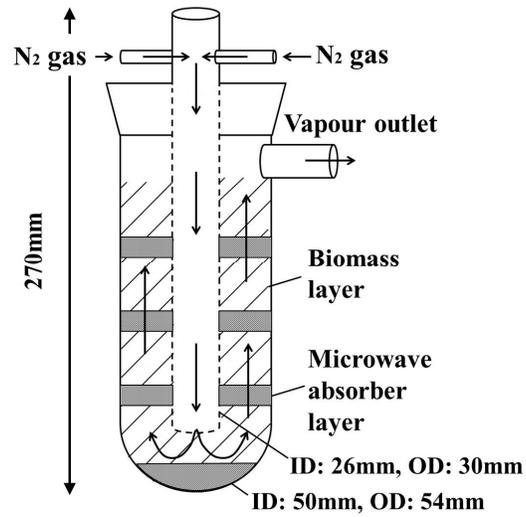


Fig. 3. Illustration of biomass and MA placement configuration with carrier gas flow direction. ID: Internal diameter OD: Outer diameter.

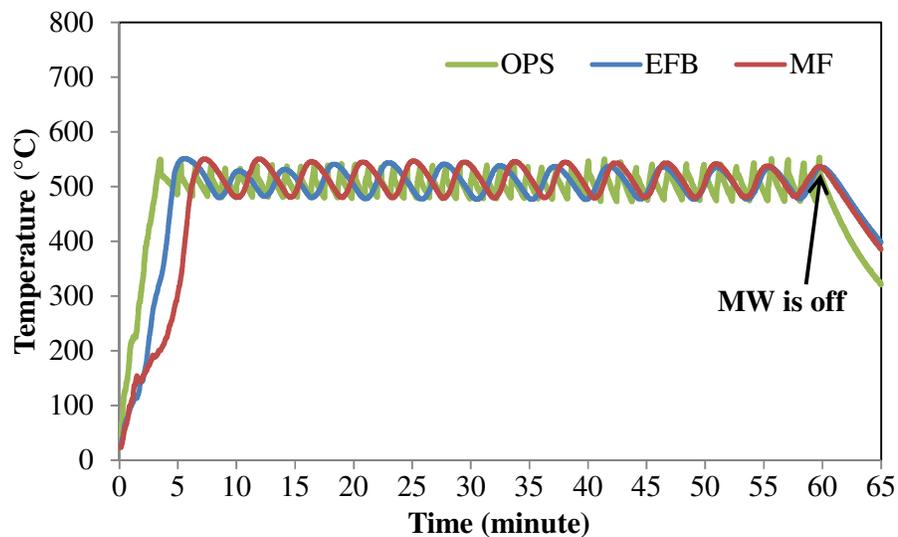


Fig. 4. Temperature profile during operation of MW pyrolysis for OPS, EFB and MF at 500°C.

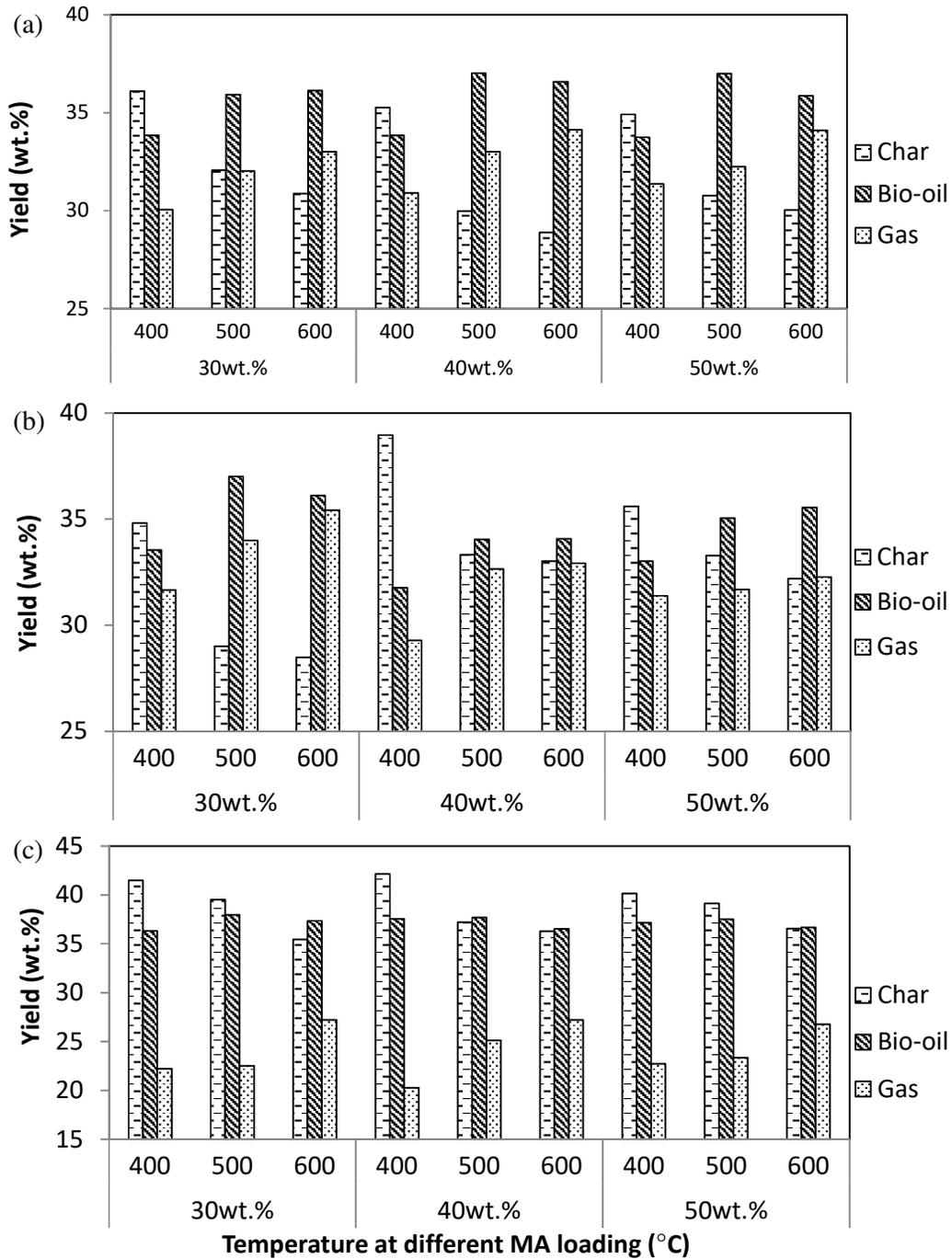


Fig. 5. Products yield under different temperature and MA loading percentage for MW pyrolysis of (a) EFB, (b) MF, (c) OPS