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Kinetics of Microwave Co-Pyrolysis of Palm Oil Industry Solid Waste and Polyethylene Terephthalate Waste



Novi Caroko^{1,3,*}, Harwin Saptoadi², Tri Agung Rohmat²

- ¹ Doctoral Student of Dept. of Mechanical and Industrial Engineering, Faculty of Engineering, Universitas Gadjah Mada, Indonesia
- Dept. of Mechanical and Industrial Engineering, Faculty of Engineering, Universitas Gadjah Mada, Indonesia
- Dept. of Mechanical Engineering, Faculty of Engineering, Universitas Muhammadiyah Yogyakarta, Indonesia

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ABSTRACT

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Comparison of co-pyrolysis kinetics of palm oil solid waste (shell and fiber) and Polyethylene Terephthalate (PET) waste has been performed in a microwave thermogravimetric analyzer. The ratio of biomass (shell or fiber) with PET was varied at 100:0, 75:25, 50:50, 25:75, and 0:100. All variations used 50 % of material absorber from the overall sample mass. The results showed that the addition of PET to the oilpalm shell and fiber affects maximum temperature, heating rate, average mass-loss rate, and activation energy. In shell - PET mixture compositions of 75:25, 50:50, and 25:75, maximum temperature decreased 22.47%, 30.80%, and 34.46%, heating rate decreased by 11.78%, 32.60% and 42.69%, average mass-loss rate decreased by 1.62%, 7.25%, and 13.38% than pure shell. In fiber – PET mixture compositions of 75:25, 50:50, and 25:75, maximum temperature decreased 27.50%, 32.22%, and 37.33%, heating rate decreased by 29.60%, 34.03%, and 53.94%, the average mass-loss rate decreased by 21.35%, 37.88% and 39.36% than the pure fiber. Both pure biomasses have the lowest value of activation energy compared to other test variations. Co-pyrolysis of a mixture of palm oil industry solid waste and PET waste showed a synergistic effect when biomass and PET are mixed. It suggests that microwave co-pyrolysis is a promising method for biomass and plastic waste processing.

Keywords:

Heating rate; Microwave co-pyrolysis; Average mass-loss rate; Maximum temperature; Oil-palm shell and fiber

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1. Introduction

The world energy consumption growth rate increased by 2.2% in 2017, which was the fastest growth since 2013. In the last ten years, the average growth was 1.7% per year. Coal and natural gas are the biggest energy consumption followed by renewables and oil. Since 2010, the growth of global oil consumption in the past ten years increased 1.7 million barrels per day which equals 1,8%. Natural gas consumption has increased to 96 billion cubic meters. Coal consumption has risen 25 million tons

E-mail address: novicaroko@yahoo.co.id (Novi Caroko)

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^{*} Corresponding author.



of oil equivalent (MTOE) or 1%. Global nuclear generation has risen by 1.1%. Renewable energy is regarded to be the biggest record increase (69 MTOE) of 17%, greater than the average of the last ten years [1].

Every year, over 300 million tons of plastics generated. This is substantially larger than 1.5 million tons in 1950. The worldwide rise in the use of plastics is approximately 4% per year [2]. The use of standard plastics as packaging products poses several environmental issues, for example, environmental pollution caused by non-degradable and non-recyclable compounds such as Polyethylene Terephthalate (PET) [3]. Every year, 1.15 to 2.41 million tons of plastic wastes are predicted to flow into the ocean [4]. Indonesia's oil palm plantation area was 11,914,499 hectares with a manufacturing capability of 33,229,381 tons in 2016 [5]. Regarding the elevated demand of Crude Palm Oil (CPO), the resource of vegetable oil as well as supplies of biofuel, a large land area was provided. Palm oil processing produces waste such as fibers and empty fruit bunches that are usually disposed of or burned [6].

In order to find new energy sources for waste processing technologies development is necessary [7]. Pyrolysis is one method of waste processing that is deemed good to be evolved as it can convert waste into various types of higher economic value. Pyrolysis breaks down waste into three primary products: char, liquid, and gas. Since there is no residue generated during the pyrolysis process, it is regarded as an environmentally friendly technique [8].

Most of the researches conducted microwave pyrolysis that used single material and no experiments were made to blend two materials. Several studies have been performed on the copyrolysis of biomass and polymer waste. However, the use of microwaves as the heat source is still minimal. The duration of pyrolysis, microwave power, and sample size may affect the pyrolysis features of a blend of biomass and polymer waste [9]. TGA analysis was used to obtain an overview of the thermal behavior of the materials. The use of TGA analysis also helped define the effect of PET addition on microwave co-pyrolysis of biomass and PET mixture.

2. Experimental Section

2.1 Material

This research used raw materials in the form of biomass (shells and fibers obtained from a palm oil factory in Riau, Indonesia) and Polyethylene Terephthalate (PET, obtained from a plastic industry in Yogyakarta, Indonesia). The biomass raw material was analysed following ASTM D7582-12. Amounts of extractives with organic solvents, lignin, holocellulose, and a-cellulose were determined. Before chemical analysis, the samples were ground by a rotary speed mill (P-14, Fritsch) and then sieved to collect samples in 40–80 mesh size. After that, the samples were oven-dried at 105 \pm 2°C. To determine the amount of the extractives, 5 g oven-dried sample was extracted with 120 ml mixture of ethanol and toluene (1/2, v/v) by a Soxhlet extractor for 6 h. Amounts of Klason lignin, holocellulose, and a-cellulose were determined by ordinary methods [8,9]. The material absorber used is coconut shell charcoal obtained from a coconut shell charcoal industry in Yogyakarta, Indonesia. All biomass (shell and fiber), Polyethylene Terephthalate (PET), and material absorber feedstocks were dried at 105 °C for 2 hours and then ground into 1–2 mm particle size. Microwave absorber use is adapted to the kinds and quantity of the heated material. There are kinds of material absorbers that react like carbon, but there are kinds of material absorbers that do not react entirely like silicon carbide [10]. About 30 g of a mixture of biomass, PET, and material absorber was used in each experiment. All variations used 50 % of material absorber from the overall sample mass. The ratio of biomass (shell or fiber) with PET was varied at 100:0, 75:25, 50:50, 25:75, and 0:100.



2.2 Experimental Apparatus and Procedures

The tests were conducted by using a microwave oven. An electronic balance (Capacity: 0.25 kg, Precision: 0.0001 g) was located on the top of the oven. Weight data were collected and recorded by a data acquisition system during the experiment.

Co-pyrolysis experiments were carried out using an Electrolux microwave EMM2308X model with 800 W microwave output power and frequency of 2.45 GHz. An open Pyrex reactor (300 mm height, 100 mm ID) was used in this experiment. The sample was placed in a sample container made of Pyrex with a capacity of 50 ml. The process of co-pyrolysis was carried out at a microwave output power of 800 W and held for 60 minutes. A sample of 30 g was inserted at room temperature and left until it reached the maximum temperature. The system schematic diagram is displayed in Figure 1.

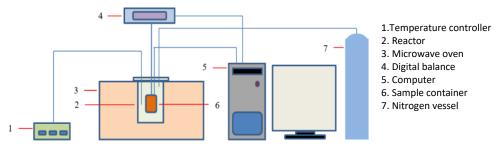


Fig. 1. Schematic diagram of experimental set-up of thermogravimetric analysis

2.3 Microwave Co-Pyrolysis Experiment

The biomass thermal degradation conducted (shell or fiber) with PET was assessed. About 30 g of the sample was loaded into a Pyrex crucible for each experiment and heated for 1,600 seconds with 250 ml/min of nitrogen flow rate. Biomass (shell or fiber) with PET powder was first mixed with the mass ratio of 100:0, 75:25, 50:50, 25:75, and 0:100. All variations used 50 % of material absorber from the overall sample mass, and then, to conduct the Thermogravimetry Analysis (TGA) test.

2.4 Kinetic Study

Co-pyrolysis acquired the evolution with weight loss temperature (TG) and the weight loss rate (DTG) for the TGA analyses [11]. The expression used to calculate the DTG:

$$\frac{dW}{dt} = -\frac{1}{W_0} \left(\frac{dW_t}{dt} \right) \tag{1}$$

The difference in weight loss (ΔW) was frequently described to explain the synergistic impacts among co-reactants based on the synergistic effect of each material during pyrolysis [11-14].

$$\Delta W = W_{blend} - (x_1 W_1 + x_2 W_2) \tag{2}$$

The activation energy, kinetic parameters, and pre-exponential factor of biomass co-pyrolysis with polymers can be defined by integral technique. [11-18]. In addition, co-pyrolysis was presumed to be a first order reaction. [11-15,18]. The calculation of kinetics was based on the equation of Arrhenius. It is therefore possible to explain the reactions concerning biomass-plastic co-pyrolysis as the following formula:



$$\frac{dx}{dt} = A \exp\left(-\frac{E}{R_0 T}\right) (1 - x) \tag{3}$$

where A is pre-exponential factor; E is activation energy; T is temperature; t is time; x is weight loss percentage or co-pyrolysis conversion calculated by the equation.

$$\chi = \frac{W_0 - W_t}{W_0 - W_f} \tag{4}$$

where W_0 is the original test sample mass; W_t is the mass at time t and W_f is final mass at the end of co-pyrolysis. The equations were rearranged and integrated for a constant heating rate H during co-pyrolysis, H = dT/dt.

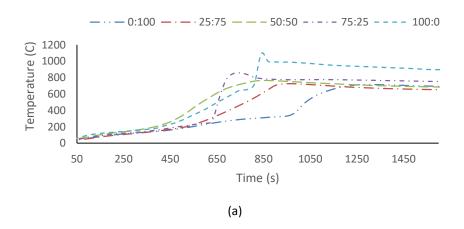
$$ln\left[\frac{-ln(1-x)}{T^2}\right] = ln\left[\frac{AR_u}{HE}\left(1 - \frac{2R_uT}{E}\right)\right] - \frac{E}{R_uT}$$
(5)

The expression $\ln[AR_u/HE(1-2R_uT/E)]$ in the proposed equation is principally constant for most E values and for the temperature range of co-pyrolysis. Therefore, a straight line will be obtained if the left side is plotted against 1/T. The activation energy E can be determined from the slope, -E/R_u. Moreover, the pre-exponential factor A can also be defined by the temperature at which W_t = (W₀ + W_f)/2 replaces T in the intercept term of the above equation.

3. Results and Discussion

3.1 Temperature Profile

Figure 2 shows the microwave pyrolysis temperature profiles of a blend of biomass (shell or fiber) with PET with variations of 0:100, 25:75, 50:50, 75:25, and 100: 0 at 800 W. The shell temperature escalated quickly to approximately 105 °C in about two minutes mainly due to moisture heating. The temperature subsequently increased slowly from about 120 to 182 °C, showed the capacity of materials to heat under microwave areas, reduced as a result of moisture evaporation [19]. Then, the temperature nearly displayed a linear rise from 182 to 1,091°C in about 14 minutes and eventually stayed principally stable at 890 °C due to the balance between heat loss and heat produced. All experiments showed a resemblance to the temperature profile. The difference in temperature profile is caused by the material's capacity to absorb microwaves.





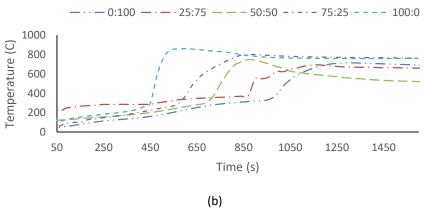
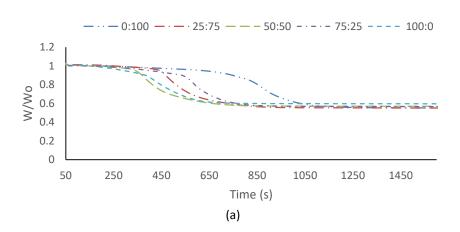


Fig. 2. Temperature profiles of microwave co-pyrolysis of a mixture of shell (a) and fiber (b) with Polyethylene Terephthalate (PET)

3.2 Mass-loss Profile

The mass-loss profiles of the shell (a) and fiber (b) with Polyethylene Terephthalate (PET) copyrolysis with variations of 0:100, 25:75, 50:50, 75:25, and 100:0 is shown in Figure 3. For all variations, thermal decomposition reaches 40% with the final mass of 0.6 w/w0. The decomposition of biomass occurred immediately after the moisture evaporation was completed. The variations of the mass profile were probably produced by the thermal effects of the microwave. The hotspot is a thermal effect in heterogeneous reactions due to solid-to-vapor transformation by non-uniform electromagnetic field distribution or selective microwaves heating [20]. It may lead to greater temperatures in local areas within the sample than the average temperature. Selective heating for biomass material could occur because of the existence of metal ions. The presence of hotspots can seem to react at a reduced temperature [21]. Whereas in the biomass (shell and fiber) - PET mixture, the fastest mass reduction occurs in variations of 100:0 and the slowest occurs in variations of 0:100. There is no mass reduction after 1100 seconds with the final mass around 60%. This is due to the constant temperature gained after reaching a maximum temperature as a result of most of the biomass had been converted to char when the process completed [22].





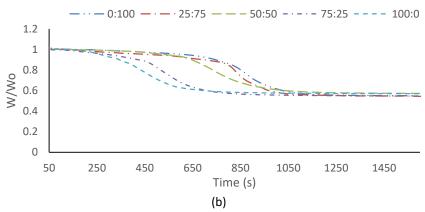


Fig. 3. Mass-loss profiles of microwave co-pyrolysis of a mixture of shell (a) and fiber (b) with Polyethylene Terephthalate (PET)

3.3 Co-pyrolysis of Materials and Their Blends

DTG curves can also be seen in Figure 4. Biomass (shell or fiber) and PET co-pyrolysis occurs in one stage. Thermal decomposition of shell-PET with variation of 100:0 occurs at 120-840 seconds, 75:25 at 150-990 seconds, 50:50 at 210 - 810 seconds, 25:75 at 360-1020 seconds, and 0:100 at 600-1230 seconds, while on fiber-PET variations are 100:0 at 210-870 seconds, 75:25 at 450-970 seconds, 50:50 at 600-1100 seconds, 25:75 at 750-1100 seconds, and 0:100 at 600-1230 seconds. The addition of PET will have an impact on the long process of decomposition. In both variations, the mixture composition of 25% PET and 75% of biomass has the best dTG/dt value compared to other variations. This result shows that biomass and PET have a beneficial synergetic impact on the reduction of dTG/dt. Individually, bio-mass is more easily decomposed compared to plastic [23].

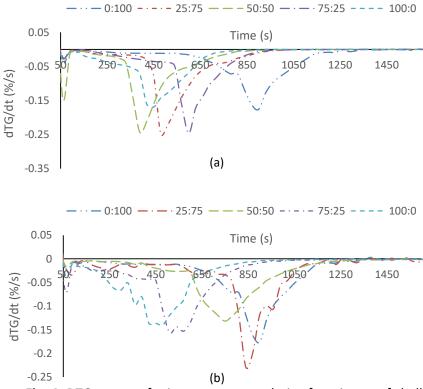


Fig. 4. DTG curves of microwave co-pyrolysis of a mixture of shell (a) and fiber (b) with Polyethylene Terephthalate (PET)



3.4 Maximum Temperature

Maximum temperature in both types of biomass (shell or fiber) and Polyethylene Terephthalate (PET) mixtures will increase with the increasing percentage of the bio-mass. The maximum temperature in the composition of 100:0 shell-PET is reached at 1107.00 °C, while in the shell – PET mixture compositions of 75:25, 50:50, 25:75, and 0:100 was approximately 858.17, 766.02, 725.50, and 638.31 °C, respectively (Figure 5). The maximum temperature in 100:0 fiber-PET composition was reached at 860.74 °C, while in the fiber – PET mixture compositions of 75:25, 50:50, 25:75, and 0:100 was approximately 802.52, 750.32, 693.70, and 638.30 °C, respectively. It means that all variations of the mixture can reach the pyrolysis temperature (400-700 °C) but there is a difference in the maximum temperature achieved. The difference in maximum temperature is caused by the ability of the material to absorb microwaves [9]. The lignocellulosic content of biomass also affected microwave pyrolysis heating efficiency. Generally, with increasing polysaccharide content, peak temperature has risen. This should be attributed to hemicellulose and cellulose thermal reactivity [24]. At 75:25, 50:50, and 25:75 shell-PET compositions, maximum temperature decreased 22.47%, 30.80%, and 34.46% than pure shell. For 75:25, 50:50, and 25:75 fiber-PET compositions, maximum temperature decreased 27.50%, 32.22%, and 37.33% compared with pure fiber.

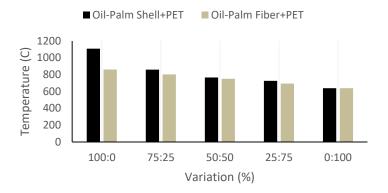


Fig. 5. Correlations between material composition and maximum temperature

3.5 Heating Rate

As can be seen in Figure 6, both types of mixtures biomass (shell and fiber) and PET have a heating rate value which decreases with the increasing percentage of PET with the maximum value found in variations of 100:0 and the minimum value is found in variations of 0:100. In all the variations, the shell-PET mixture has a higher heating rate than the fiber-PET mixture. The higher fixed carbon content has better absorption of microwaves [25]. Based on these results, raising the heating rate can be done by adding a fixed carbon content to the pyrolysis raw material. At 75:25, 50:50, and 25:75 shell-PET compositions, the heating rate decreased by 11.78%, 32.60% and 42.69% than the pure shell. For 75:25, 50:50, and 25:75 fiber-PET compositions, the heating rate decreased by 29.60%, 34.03% and 53.94% than pure fiber.



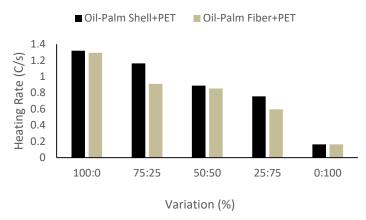


Fig. 6. Correlations between material composition and heating rate

3.6 Average Mass Loss Rate

It can be concluded based on Figure 6 and 7 that better heating rate effects on the better average mass-loss rate value. In the study of Barneto *et al.*, [26] heating rate affects the average mass-loss rate. However, considering the heating rate of both shell and fiber in the 100:0 variations, fiber average mass-loss rate should be better than the shell. Therefore, this difference is possibly due to the higher moisture content of fiber compared to shell resulting in the higher average mass-loss rate of fiber than that of the shell. At 75:25, 50:50, and 25:75 shell-PET compositions, average mass-loss rate decreased by 1.62%, 7.25% and 13.38% than pure shell. For 75:25, 50:50, and 25:75 fiber-PET compositions, average mass-loss rate decreased by 21.35%, 37.88% and 39.36% than pure fiber. The difference in average mass-loss rate of 75:25, 50:50, and 25:75 shell-PET compositions are 0.00366, 0.00669, and 0.00963 g/s. For 75:25, 50:50, and 25:75 fiber-PET compositions are 0.00061, 0.00213, and 0.00651 g/s than before both of biomass and PET mixed. It means there is a synergistic effect on the average mass-loss rate when biomass and PET are mixed.

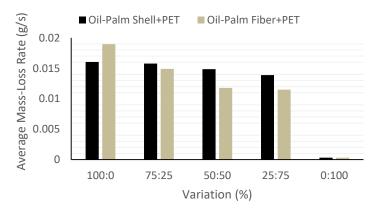


Fig. 7. Correlations between material composition and average mass-loss rate

3.7 Activation Energy

As shown in Table 1, the value of activation energy in both types of mixtures bio-mass (shell and fiber) with PET has the same trend, namely the value of activation energy that increases with the increasing percentage of PET. The variation of 0:100 (pure PET) has the highest activation energy



which is 121.95 kJ/mol compared to other test variations. In each variation of the mixture, pure fiber has higher activation energy when compared to pure shell. This is caused by fixed carbon content in bio-mass. Table 2 presents the content of fixed carbon in a shell is higher than fiber. This can be correlated to the study of Huang *et al.*, [24], that fixed carbon content affects the activation energy.

Table 1Kinetic parameters of microwave pyrolysis of a mixture of shell and fiber with Polyethylene Terephthalate (PET)

Biomass-PET feedstock	Conversion fraction	E (kJ/mol)	R ²
Shell-PET		-	
100:0	0.14	10.04	0.98
75:25	0.16	12.75	0.99
50:50	0.14	16.11	0.97
25:75	0.11	23.78	0.96
Fiber-PET			
100:0	0.10	22.03	0.97
75:25	0.08	25.26	0.97
50:50	0.17	29.19	0.94
25:75	0.21	38.83	0.88
0:100	0.15	121.95	0.96

Table 2Compositions of oil palm solid waste feedstocks

Compositions of oil paint solid waste feedstocks				
Characteristics	Oil-Palm Shell	Oil-Palm Fiber		
Proximate analysis* (wt.%)				
Moisture	6.04	7.08		
Volatile	85.18	84.32		
Fixed carbon	7.34	2.84		
Ash	1.43	5.76		
Lignocellulosic analysis (wt.%)				
Holocellulose	57.00	46.33		
Alva cellulose	28.67	22.24		
Lignin	38.76	34.10		

^{*} Air dry based

4. Conclusions

The influence of composition variation of raw material on maximum temperatures, mass loss rate, heating rate, and energy activation was substantial. Either maximum temperature, heating rate, mass loss rate, and activation energy has a linear relationship with the composition ratio of biomass (shell and fiber) and PET. The heating effectiveness was also influenced by the combustible content of biomass, particularly the content of fixed carbon. The results showed that the addition of PET to shell and fiber affects maximum temperature, heating rate, average mass-loss rate, and activation energy. In shell – PET mixture compositions of 75:25, 50:50, and 25:75, maximum temperature decreased 22.47%, 30.80%, and 34.46%, heating rate decreased by 11.78%, 32.60% and 42.69%, average mass-loss rate decreased by 1.62%, 7.25%, and 13.38% than pure shell. In fiber – PET mixture compositions of 75:25, 50:50, and 25:75, maximum temperature decreased 27.50%, 32.22%, and 37.33%, heating rate decreased by 29.60%, 34.03%, and 53.94%, the value of average mass-loss rate decreased by 21.35%, 37.88% and 39.36% than pure shell. Both of biomass has the lowest value of activation energy compared to other test variations. Co-pyrolysis of a mixture of palm oil industry



solid waste and PET waste showed a synergistic effect when biomass and PET are mixed. It suggests that microwave co-pyrolysis is a promising method for biomass and plastic waste processing.

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