Effect of Temperature and Residence Time on Torrefaction of Municipal Sewage Sludge

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Abstract*— Malaysia, a developing country with a constant rise in population, recorded an increase in municipal and industrial wastes with approximately 23,000 tonnes of wastes were produced each day in Malaysia (Behzad, Ahmad, Saied, Elmira, & Bin, 2011). This resulted in an increase of total waste generated. With the common practice of waste disposal through landfill, and incineration, these contributes to another environmental issue; the pollution. To achieve a sustainable environment, new alternatives was found by researchers to treat wastes. Torrefaction is one of thermal process that is applied on biomass, to convert it into three useful products, char, bio-oil and condensable gases. In current research, char from lignocellulosic biomass was proven as the good alternative in the fuel industry. However, the characteristics of non-lignocellulosic biomass makes it possible for researchers to conduct a studies on it. Hence, the objective of this study is to produce char from municipal sewage sludge by using torrefaction method. In this study, municipal sewage sludge obtained from Indah Water Konsortium, located in Johor, Malaysia, has been used as the raw material. A characterization study was performed on the raw and dried sewage sludge. The study was conducted to record the moisture content, ash content and high heating value (HHV) of the raw sewage sludge. Next step is where biomass is being placed in a covered crucible and heated in a muffle furnace. The heating process operates under pressure of 1 atm and temperature of 200°C, 250°C and 300°C with residence time of 20, 30 and 60 mins for every temperature. The effects of torrefaction temperature and residence time on mass and energy yields, and HHV of torrefied sludge were investigated. Mass and energy yield of torrefied sludge were investigated to study the weight and energy loss of raw sewage sludge after torrefaction. HHV of the char was determined to study the energy value stored in torrefied sludge. From the results, the HHV, mass and energy yields were decreased as the torrefaction temperature and residence time increased. Highest peak was at temperature of 250°C and residence time of 60 mins. Thermogravimetric analysis was performed on high HHV of char. It was found that hemicellulose will degrade first at temperature of approximately above 200°C. An average reaction order of 0.59, 6.9852 kJ/mol of activation energy, E_a, and pre-exponential factor of 0.3772, were found to be the kinetics parameters of torrefaction on municipal sewage sludge.

***Keywords*— Energy yield, Kinetics, Mass yield, Municipal sewage sludge, Torrefaction.**

I. INTRODUCTION

Unplanned industrialization and urbanization with rapid population growth have led to tremendous increase in municipal solid wastes (Ramachandra, Bharath, Kulkarni, & Sheng, 2017). Common disposal methods for these waste are through incineration, and landfill. However, these methods is not good enough to be accepted, since the energy used for waste incineration is much consumed than energy recovered (Pulka, Wiśniewski, Gołaszewski, & Białowiec, 2016). The authorities is well informed, and they discussed on finding another way to dispose these waste rather than dumping it through the landfill. Limitations of land area for development also triggers the government to find other to dispose waste. With these issues, researchers have found a thermal treatment process to be applied on the wastes (Agrafioti, Bouras, Kalderis, & Diamadopoulos, 2013; Dhungana, Dutta, & Basu, 2011; Kwapinski et al., 2010). Pyrolysis is a thermal treatment process where high temperature is applied on the biomass with the absence of oxygen (Chen *et al.*, 2018). Three methods of pyrolysis are torrefaction, carbonization and hydrothermal carbonization. The pyrolysis of biomass will result to three products, biochar (solid products), bio-oil (liquid products) and biogas (gaseous products) (Manyà, Azuara, & Manso, 2018).

However, various applications on biochar have been studied. In the article of Weber and Quicker (2018), the applications of biochar are presented in various industry, including their use in medical, energy, and agricultural industry as well as a replacement in fossil fuel. Human overpopulation caused the reducing of fossil fuels and limitations on the energy sources. Current researchers are conducting studies to find which type of biomass that will perform better in the fuel industry.

Biochar or char, is a good alternative to substitute with wastes disposal methods as the application is beneficial to the fuel production and being used as one of the energy sources (Agrafioti et al., 2013). The lignocellulosic components such as lignin, hemicellulose and cellulose in biomass have contributed to the yield of torrefied products to make it useful. These composition varies depending on the types of biomass. In the production of biochar using torrefaction method, the types of biomass that are commonly used are lignocellulosic biomass and non-lignocellulosic biomass. Lignocellulosic biomass contains lignocellulosic components and are mainly woody plant while non-lignocellulosic biomass can be found in municipal wastes and poultry litter with low content of lignocellulosic components (Stefanidis et al., 2013).

Worasuwannarak, Sonobe and Tanthapanichakoon, (2007); Stefanidis *et al.*, (2013) have stated in their research, that the production of torrefied products (biochar, bio-oil and condensable gases) were determined through these constituents. Usually the connections between cellulose and lignin have led to the high yield of char, while the connections between hemicellulose-cellulose and hemicellulose-lignin would not give much difference on the yield of biochar.

Apart from that, parameters such as torrefaction temperature and residence time are also important in producing a biochar. According to Agrafioti *et al.*, (2013); Tripathi, Sahu and Ganesan, (2015), torrefaction temperature and residence time will give different effects on the applications of torrefied products. At temperature ranging from 200°C to 400°C, the torrefied products can be used in various applications. While at low temperature, torrefied products is useful in soil applications. At an elevated temperature, porosity in biochar performs well in the removal of heavy metals in soil (Agrafioti et al., 2013; Tag, Duman, Ucar, & Yanik, 2016). Furthermore, decomposition of constituents is heavily dependent on the residence time of biomass through the thermal process.

Several studies about effects of temperature and residence time through various methods on biochar were limited to lignocellulosic biomass (Kwapinski et al., 2010; Suman & Gautam, 2017; Weber & Quicker, 2018; Yaacob, Rahman, Matali, Idris, & Alias, 2016; Zhang, Hu, Zhang, & Xiong, 2016), but not on non-lignocellulosic biomass, such as municipal sewage sludge (Poudel, Ohm, Lee, & Oh, 2015; Pulka, Manczarski, & Koziel, 2019). As a conclusion, firstly, this study focuses to produce biochar from municipal sewage sludge. Secondly, to investigate the effect of torrefaction temperature and residence time on torrefied municipal sewage sludge obtained; in terms of high heating value (HHV), mass and energy yields. Lastly, an analysis on the constituents present in biomass were conducted through thermogravimetric analysis and studying the kinetics of torrefaction on municipal sewage sludge.

II. METHODOLOGY

A. *Materials*

Municipal sewage sludge obtained from Indah Water Konsortium, Johor, Malaysia was used as the raw material in this study. After collection, the samples were dried in a drying oven to determine the moisture content. The samples were dried for 24 hours, at temperature of 90°C. Table 1 shows the characterization study of the sewage sludge sample used. Determination of moisture content as shown in Equation (1).

Table 1 Characterisation Study of Municipal Sewage Sludge

Moisture content (%)	52.84
Ash content (%)	74.73
High heating value, HHV (MJ/kg)	10.103

Determination of moisture content :

$$\text{Moisture content (\%)} = \frac{\text{dried sample} - \text{wet sample}}{\text{dried sample}} \times 100\% \quad (1)$$

B. *Research methodology*

The dried sewage sludge were grounded and sieved to be torrefied in a muffle furnace under temperature ranging from 200°C to 300°C. The residence time was varied at 20 minutes, 30 minutes and 60 minutes. For each experiments, constant mass of 50g dried sewage sludge was used. Under operating pressure of 1 atm, a 50 ml ceramic crucibles was placed in a muffle furnace at the centre, and covered with aluminium foil. It was done to minimize the presence of oxygen. After heating, the crucible was then cooled in desiccator and weighted, until two consecutive masses were obtained. The apparatus of muffle furnace set up is shown in Figure 1.

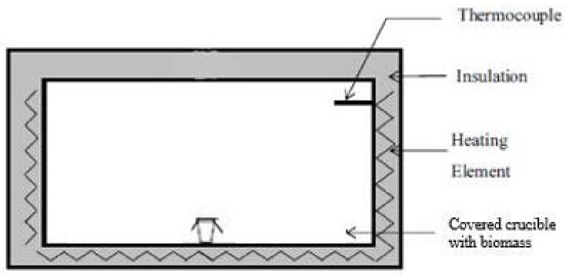

Fig. 1 A schematic arrangement of covered crucibles with biomass in muffle furnace

For each torrefied sludge obtained, mass yield, energy yield and HHV were determined. Determination of HHV is to measure the energy content of the torrefied sludge. The HHV were measured using a bomb calorimeter (IKA – Works C5000 Control Germany), at operating pressure of 35 bar and operating temperature of 1000°C.

The mass and energy yields of the torrefied sludge were defined by Equation (2) and (3) below, as used by Poudel *et al.*, (2015). Mass and energy yields were studied to measure amount of weight and energy loss difference for raw sewage sludge and torrefied sewage sludge.

$$\text{Mass yield } (Y_{mass}) = \frac{\text{mass after sample being torrefied}}{\text{mass of raw biomass sample}} \times 100\% \quad (2)$$

$$\text{Energy yield } (Y_{energy}) = Y_{mass} \times \frac{\text{HHV torrefied sample}}{\text{HHV raw sample}} \times 100\% \quad (3)$$

In order to study the kinetics analysis of torrefaction, a thermogravimetric analysis was performed on the HHV of torrefied sludge. Thermogravimetric analysis of torrefied sludge were measured using a Thermogravimetric Analyzer (TGA – Mettler Toledo). The heating rate of this analysis are 10, 20 and 30 °C/min, at temperature ranging from 200°C to 900°C. The initial mass of the sample used was 20mg. All analysis were carried out in Nitrogen gas atmosphere with gas flowrate set at 50 m^3/min.

C. *Kinetics Analysis of Torrefaction*

Kinetics parameters such as rate constant, k, activation energy, E_a, and pre-exponential factor, A, were determined through the kinetics analysis based on the Coats-Redfern method. According to Chen, (2015), the Coats-Redfern method is the most common kinetics method used for thermal decomposition of coal and biomass. The standard equation for rate of reaction of a sewage sludge sample, can be written as (W. Chen & Kuo, 2011; Poudel et al., 2015);

$$\frac{dY}{dt} = k\,(1-Y)^n \quad (4)$$

where Y is the conversion of sewage sludge sample. Y is defined by;

$$Y = \frac{m_i - m}{m_i - m_f} \quad (5)$$

Where, m is the mass of the sewage sludge sample at time t, m_i is the initial mass of the sewage sludge sample and m_f is the final mass of the sewage sludge sample.

With respect of the reaction order (n=1), integrating Eq. (4) gives;

$$\ln\left(\frac{1-Y_o}{1-Y}\right) = k\,(t - t_o); \quad \text{for } n=1$$
(6)

Y_o is the conversion of the sewage sludge sample at the beginning of torrefaction where $t=t_o$. From Eq. (6), when a graph of ln $(1\text{-}Y)^{-1}$ against torrefaction time, $(t – t_o)$ be plotted, the slope of the graph is the rate constant, k. However, when the reaction order is not equal to 1 ($n\neq1$), the integration of Eq. (4) becomes;

$$(1 – Y)^{1-n} – (1 – Y_o)^{1-n} = k\,(n - 1)(t – t_o); \quad \text{for } n\neq1 \qquad (7)$$

Similarly, when Eq. (7) was plotted, $(1 – Y)^{1-n}$, against $(n - 1)(t - t_o)$, the slope of this graph is the rate constant for the respective heating rate.

The rate constant is related to the activation energy, Ea and pre-exponential energy, A, through Arrhenius law, as below; (W. Chen & Kuo, 2011; Poudel et al., 2015)

$$k = A \exp\left(\frac{-Ea}{RT}\right) \qquad (8)$$

Converted into logarithm form will gives;

$$\ln(k) = \ln(A) - \frac{Ea}{RT} \qquad (9)$$

When the graph of ln (k) against T^{-1} was plotted, the slope of the graph was referring to –Ea/R with y-axis intercept of ln (A), where T is the torrefaction temperature (W. Chen & Kuo, 2011; Poudel et al., 2015).

Nomenclature

A	Pre-exponential factor (min^{-1})
E_a	Activation energy (kJ mol^{-1})
k	Rate constant (min^{-1})
n	Order of reaction (-)
R	Universal gas constant (8.314 J mol^{-1} K^{-1})
T	Temperature (°C/Kelvin)
T	Time (min)
Y	Conversion of the SS sample (-)
m	Mass of sample (mg)
Subscript	
o	Beginning of torrefaction
i	Initial state (at 200°C)
f	Final state (at 900°C)

III. RESULTS AND DISCUSSION

A. *Mass yield*

First objective of the study is to produce a char from municipal sewage sludge via torrefaction. Secondly, mass of torrefied char were taken before and after torrefaction, to study on the effects of temperature and residence time on mass yield, energy yield and HHV.

Figure 2 shows the graph of mass yield against torrefaction temperature at varying residence time. It can be seen that an increase in torrefaction temperature resulted to a decrease in mass yield. The result is in agreement to a research conducted by Dhungana, Dutta and Basu, (2011); Tripathi, Sahu and Ganesan, (2015); Yaacob *et al.*, (2016), proving that as the torrefaction temperature increases, the mass yield of the char decreases. This was due to the decomposition of constituents in non-lignocellulosic biomass, which reduced as temperature increases, thus, affecting the conversion of char. The highest mass yield resulted in one major peak at temperature of 250°C, residence time of 60 mins. Among the constituents in non-lignocellulosic biomass, hemicellulose is most likely to be degraded first during torrefaction (Z. Chen et al., 2018). This is likely to happen since torrefaction was conducted in the range of temperature similar to temperature of degradation for hemicellulose. For residence time, an increased value in torrefaction temperature and residence time, resulted to lower mass yield. Nhuchhen, Basu and Acharya, (2014) stated that, the torrefaction residence time has slight effects on solid char compared to the torrefaction temperature, but at longer residence time, resulted in a much lower solid char yield.

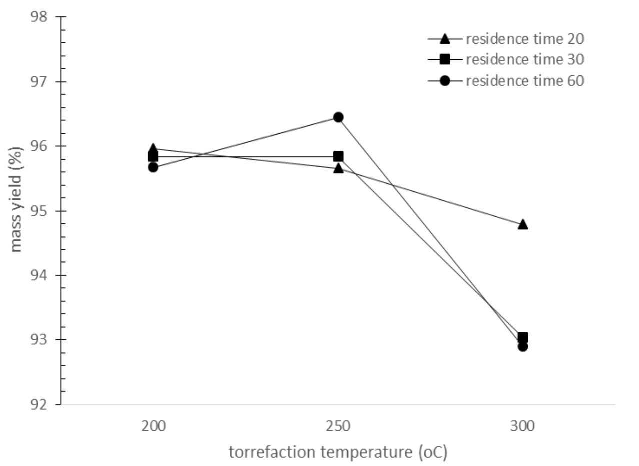

Fig. 2 Mass yield against torrefaction temperature at varying residence time

B. *Energy yield*

Data obtained for energy yield is shown in Figure 3. Data of mass yield and energy yield is related. This is true since the mass yield is involved in the calculation of obtaining energy yield, as shown in Chapter 3; Equation (10). Thus, identical data was recorded for energy yield. The trend of the data shows that the higher the torrefaction temperature, the lower the energy yield. As observed, high yield of energy was recorded, at the beginning of the torrefaction process (at temperature of 200°C and 250°C). The energy yield starts to decrease rapidly beyond that temperature. Highest energy yield was observed at 250°C, and 60 minutes. Poudel *et al.*, (2015) reported that the same trend was obtained in their research, the energy yield of char decreases as torrefaction temperature and residence time increases. For residence time, as expected, low energy yield was recorded with increasing torrefaction temperature and residence time. The researcher suggested that the temperature for pre-treatment of sewage sludge should be conducted below than the torrefaction temperature, in order to minimize the energy loss (Poudel et al., 2015).

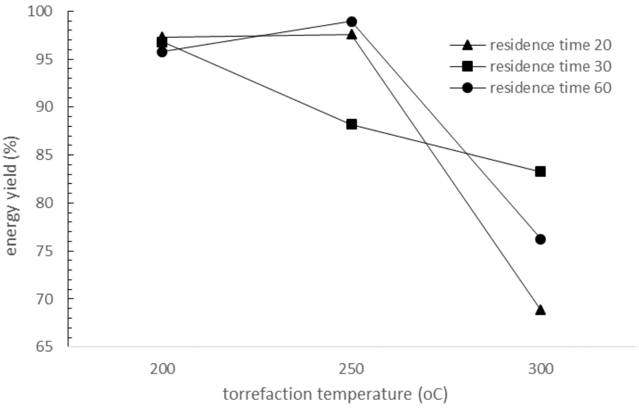

Fig. 3 Energy yield against torrefaction temperature at varying residence time

C. *HHV*

Figure 4 shows the relationship between HHV against torrefaction temperature at varying residence time. As stated earlier in *Section B*, the energy yield corresponds to the mass yield, as shown in Chapter 3; Equation (10). Poudel *et al.*, (2015) stated that the heating value, HHV of the char is also related to energy yield, in the mathematical equation. At lower torrefaction temperature (at torrefaction temperature of 200^{o}C), the HHV value did not differ much with increase of residence time. HHV of the char is noted at the highest at torrefaction temperature of 250^{o}C, with residence time of 60 minutes. Similar observation was found in the research of Poudel *et al.*, (2015). In the article, it said that the highest reading of HHV were obtained due to the release of oxygen throughout the torrefaction process. Similarly, from the research of Dhungana, Dutta and Basu, (2011) reported that, under torrefaction temperature of 250^{o}C and residence time of 60 minutes, the mass and energy yields, and HHV of undigested sludge (sewage) were at the highest peak. A further increase of torrefaction temperature and residence time, will result in a decrease of the mass and energy yields, and HHV (Poudel et al., 2015). The decomposition of constituent in biomass is related to this tendency. The repetitiveness of this trend is further analyzed via thermogravimetric analysis, to study the degradation of constituents in non-lignocellulosic biomass.

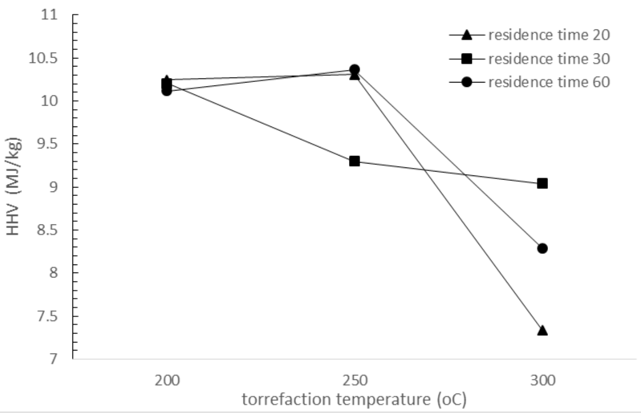

Fig. 4 HHV against torrefaction temperature at varying residence time

D. *Thermogravimetric analysis*

Thermal analysis of the char was carried out through thermogravimetric analysis. It is performed to study the thermal degradation of constituents in non-lignocellulosic biomass at respective temperature and to determine whether the torrefied sample were able to produce a good quality of fuel. The heating rate was conducted at 10 ^{o}C/min, 20 ^{o}C/min and 30 ^{o}C/min. Figure 5a, b and c shows the thermogravimetric (TG) curves against temperature, for different heating rate, 10 ^{o}C/min, 20 ^{o}C/min and 30 ^{o}C/min, respectively.

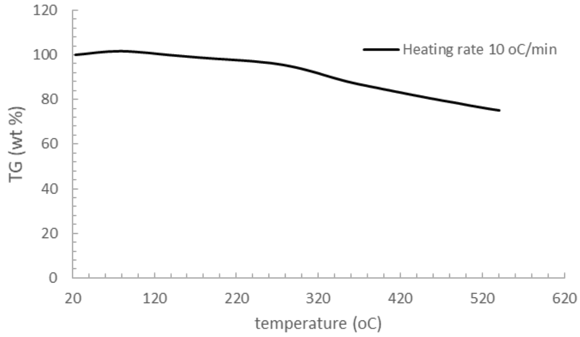

Fig. 5a. TG curves against temperature at heating rate 10 ^{o}C/min

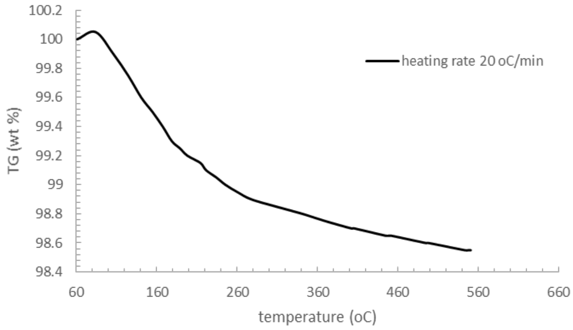

Fig. 5b. TG curves against temperature at heating rate 20 ^{o}C/min

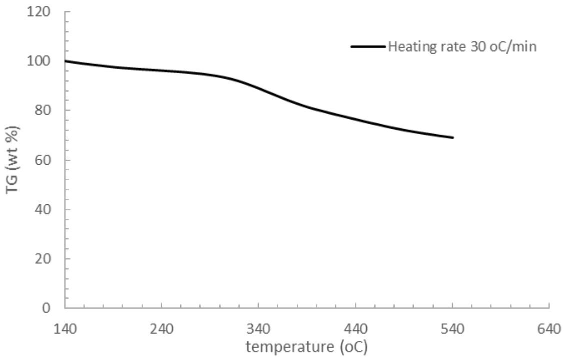

Fig. 5c. TG curves against temperature at heating rate 30 ^{o}C/min

For heating rate of 10^{o}C/min and 30^{o}C/min, the constant reading of TG curves indicate that torrefaction temperature of 20^{o}C to 150^{o}C were insignificant for degradation of constituents. However, a sudden drop of TG curves occurred, at temperature ranging from approximately 300^{o}C and above. This proved the thermal degradation of hemicellulose contents in non-lignocellulosic biomass. Chen and Kuo, (2011) stated that, hemicellulose is the most significant component to degrade in torrefaction environment, compared to lignin and cellulose, since hemicellulose is decomposed at temperature ranging from 220^{o}C to 315^{o}C, which is also the torrefaction temperature. However, TG curves at heating rate of 20^{o}C/min showed a different trend. At temperature of approximately 200^{o}C, most of the weight loss happened. The weight loss that occured along this temperature was due to the hemicellulose degradation. The research by Poudel *et al.*, (2015), showed an identical TG curves as obtained in this study. From the research, Poudel *et al.*, (2015) concluded that the starting torrefaction temperature were likely to occur at temperatures around 200^{o}C, since the decomposition of the organic contents within sewage sludge was found at temperature of approximately 200^{o}C. The thermal degradation of sewage sludge for all heating rates in this study was completed at temperature of approximately 540^{o}C.

E. Kinetics analysis of torrefaction

The reaction order of kinetics was found to be a non-unity ($n \neq 1$). Due to this factor, the graph of $(1 - Y)^{(1-n)}$ against (n-1)(t-t_o) was plotted. A linear relation was obtained, and the slope was the rate constant according to the respective heating rates, as expressed in Eq. (7). On the other hand, if the reaction order is unity (n=1), Eq. (6) will be involved to obtain the rate constant, k with plotting of ln $(1-Y)^{-1}$ against torrefaction time, $(t - t_o)$. Fig. 6a, b and c, demonstrates the plots of $(1 - Y)^{(1-n)}$ against (n-1)(t-t_o) with three heating rates, 10°C/min, 20°C/min and 30°C/min. Three values of rate constant, k were obtained according to each heating rates.

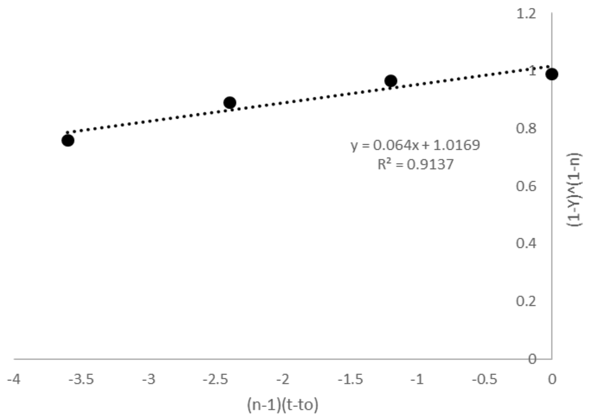

Fig. 6a. $(1 - Y)^{(1-n)}$ versus (n-1)(t-t_o) at heating rate of 10 °C/min

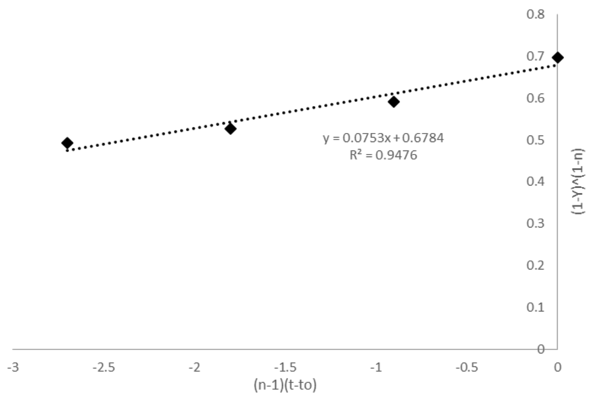

Fig. 6b. $(1 - Y)^{(1-n)}$ versus (n-1)(t-t_o) at heating rate of 20 °C/min

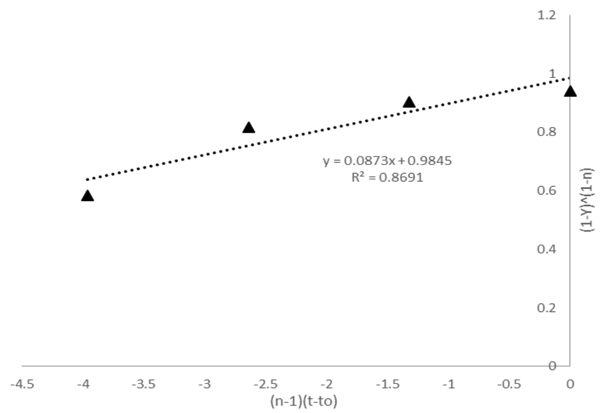

Fig. 6c. $(1 - Y)^{(1-n)}$ versus (n-1)(t-t_o) at heating rate of 30 °C/min

Based on the values of rate constant that were obtained from Figure 6, the activation energy, E_a, and pre-exponential factor, A can be determined as in Figure 7.

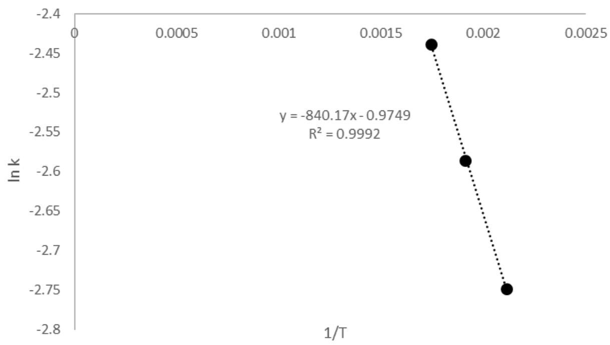

Fig. 7 ln k against torrefaction temperature, T^{-1}

Plot of ln k against T^{-1}, showed a strong linear relatonship was obtained, with that the value of R^2 is 0.9992. As a result, the activation energy obtained was 6.9852 kJ/mol, with pre-exponential factor, 0.3772 min^{-1}. Table 2 shows the summary of kinetics analysis of torrefaction on municipal sewage sludge.

Table 2 Summary of kinetics analysis of torrefaction

Heating rate (°C/min)	Reaction order, n	k (min^{-1})	A (min^{-1})	Ea (kJ/mol)
10	0.8800	0.0640	0.3772	6.9852
20	0.5500	0.0753		
30	0.3400	0.0873		
Average	0.5900	0.0755		

Comparing previous research to the results presented in this study, Abbas *et al.*, (2014) has reported high value of activation energy, E_a. Similarly, the sewage sludge sample was obtained from mechanical wastewater treatment plant in Kuala Lumpur, Malaysia. The average reaction order, n, was 0.11, while for activation energy, E_a, were found to be 82 kJ/mol, according to the finding by Abbas *et al.*, (2014). However, a variation of reaction order and activation energy were measured, due to the composition of the constituents that presents in the sewage sludge after torrefaction process.

IV. CONCLUSION

The torrefaction of sewage sludge was studied to observe the ability of non-lignocellulosic biomass to perform as an alternative for fuels, as the renewable sources. The characterization study of non-lignocellulosic biomass leads to the discovery of this study. Few parameters are needed in consideration to obtain good and high performance char. Studies from Dhungana, Dutta and Basu, (2011); Tripathi, Sahu and Ganesan, (2015); Suman and Gautam, (2017); Manyà, Azuara and Manso, (2018) reported that, mass and energy yields of the torrefied products is related to the torrefaction temperature rather than residence time. In addition to that, other studies also mentioned about heating value of the sewage sludge is influenced by the torrefaction temperature. Additionally, the purpose of this study is to investigate the effects of temperature and residence time on the torrefied sewage sludge. Throughout the thermogravimetric analysis, it is concluded that the constituents of biomass, mainly hemicellulose can be degraded at a temperature of approximately above 200°C. From the kinetics analysis study, the value of activation energy, E_a, and pre-exponential energy of torrefied sewage sludge was obtained, 6.9852 kJ/mol and 0.3772 min^{-1}, respectively, with an average reaction order of 0.59. The mass and energy yields, and HHV of torrefaction decreased with an increase in torrefaction temperature and residence time. From this experimental study, the torrefaction process of sewage sludge were found at highest peak at temperature of 250°C, with residence time of 60 minutes. Torrefaction of temperature higher than 250^{o}C resulted in a negative values of HHV.

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