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Thermal Degradation of Malaysian Domestic Wastewater Sludge (DWS) Using Thermogravimetric Analysis Method

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ABSTRACT

Domestic Wastewater Sludge (DWS) is the waste generated from the wastewater treatment plant. Recently, the potential energy from wastewater effluents has become an interest among researchers due to high potential to be converted into energy and reduce harmful emission compared to fossil fuel. This study was aimed to study the effect of heating rates and particle size on Domestic Wastewater Sludge (DWS) by using thermogravimetric analysis (TGA) method. In this study, the sample of DWS was thermally dried using thermal dryer to reduce the moisture content until below than 20% for conversion into fuel. After that, the sample of DWS was analyzed using a non-isothermal TGA with a continuous flow of oxygen at a rate of 30 mL/min with temperature from room temperature which is 30°C to 800°C. There are three sample particle sizes ranging between 0.5 mm to 1.5 mm, and heating rate between 5 K/min to 20 K/min were used in this study. Based on the results from TGA analysis, the particle size does not have any significant effect at first, however it started to separate explicitly when the temperature getting increases. In addition, the sample size of DWS may affect the reactivity and the combustion performance caused by the heat transfer and temperature gradient. Besides that, it can be concluded that the TG and the peak of derivative thermogravimetry (DTG) curves have a tendency to change at high temperature when heating rate is increased because of the limitation of mass transfer and the delay of decomposition process.

1. Introduction

Domestic Wastewater Sludge (DWS) which is waste generated from domestic wastewater treatment plants. In Malaysia, most of practical disposal for DWS is by landfilling at the dedicated site by the government. Recently, the waste of DWS produced by the wastewater treatment plant recorded a 64.4% from the total solid waste generation in Malaysia [1,2]. In addition, the yearly feed-rate of DWS in the wastewater treatment plants throughout the country is estimated at 4.9 million cubic meters from approximately 5507 wastewater treatment plants. Moreover, the yearly DWS production capacity of Malaysian DWS are estimated at nearly 3 million cubic meters with yearly

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disposal cost of MYR1 billion [3]. This statement was supported by the study conducted by the Alam *et al.*, [4] that predicting the production of DWS in Malaysia will sharply increases to 7 million cubic meters by for the next 2 years. Based on the previous study, the DWS contains microorganisms and harmful substances such as heavy metals, poorly biodegradable organic compounds, bacteria, viruses, pharmaceuticals, hormones and dioxins which can causes to serious environmental pollution and diseases. In the perspective view, the route to convert biomass into a beneficial form of energy can be done by using the thermochemical (TCC) conversion process. Combustion is one of the way to transform biomass into a final product that can be useful to produce energy. In order to transform biomass through the TCC process, the preliminary combustion behavior is important. In this study, the thermal degradation the thermal degradation and chemical reaction that would occur during the combustion process in a real TCC facilities were predicted [5,6].

Recently, Thermogravimetric Analysis (TGA) is the precise and popular technique in thermal analysis studies among the researchers. This method is often times used by the researchers to examine the thermal event and thermal decomposition of the sample during the increasing of the temperature in both combustion and pyrolysis process. In addition, TGA is a tool that gives a precise prediction of the fuel characteristic in terms of its pyrolysis and combustion behavior, and description of the reactivity study and parameters of the burning profiles [7-9].

Although the research of combustion behavior via TGA are extensively studied in the literatures, there is limited result for the investigation of important variables that affect the combustion process such as particle size and heating rate especially for DWS material. Based on the literature, the operating conditions such as particle size and heating rate are the two important parameters that effect the combustion properties. Therefore, to obtain the most representative and suitable condition for thermal degradation, the parameters that have been highlighted in the study must be taken into consideration. Thus, the objective of this study is to determine the thermal degradation and heating rate by using the TGA methods [10,11].

2. Methodology

Initially, the sample used was in a sludge form that has a plenty content of moisture. The moisture content in DWS was reduced by using thermal dryer before conducting the TGA experiment. The procedure to determine initial moisture content and time needed for the sample to dry completely is in accordance to the ASTM D1762-84 standard. After reducing the moisture content into acceptable level, the sample of DWS was manually crushed. Then, the sample of DWS was sieved using the sieve shaker to obtain the DWS particles with sizes 0.5 mm,1.0 mm and 1.5 mm. Three sieves with different sizes of square holes were arrayed from smaller to greater size by the automatic sieve shaker equipment. During the vibration, the sample was sieved and passed through the square hole in in line to its particle size. After that, the product from the sieve shaker then collected and stored in labelled airtight bottles according to its particle size.

The TGA analysis were conducted by using the thermogravimetric analyzer model LabSys Evo Setaram. In this study, each of the sample weight that used in TGA analysis was set for each experiment and placed in an alumina crucible to eliminate the initial mass effect. After that, the Non-isothermal TGA was carried out with the temperature raised from room temperature which is 30°C to 800°C in oxygen atmosphere. The DWS sample of with approximately 10-12 mg in weight was placed in a 90 µL alumina crucible in the TGA equipment. In this study, non-isothermal condition was performed where the temperature of TGA was raised from room temperature of 30°C until 800°C. The highest temperature selected in this analysis which is 800°C was based on the finding from the preliminary runs performed before performing the actual experiment of TGA analysis. Based on the finding, it can be observed that there is no further weight loss take place after the temperature in the TGA more than 800°C, which indicates that the fuel content fully diminishes and the reaction was ended. Therefore, the higher temperature which is more than 800°C leads to no effect to the reaction.

The effect of heating rates on the thermal decomposition of DWS was studied by using 4 different heating rates. In this study, the heating rate was prior fixed at 5, 10, 15 and 20 K/min. The differences of heating rate are based on the fact that this range of heating rate may reduce the mass transfer and temperature gradient effect [12-15]. In this experiment, the oxygen gas was used under fixed flow rate of 30 mL/min based on the desired experimental condition whether in combustion or pyrolysis programmed. There are 3 different particle sizes are examined in the study regarding the effect of heating rate which are 0.5mm, 1.0mm and 1.5 mm.

3. Results

In TGA analysis, the combustion graph shows a good understanding about the reaction course of the DWS in oxidative atmosphere. There are several stages can be clearly designated by the result of the TG and DTG curves. TGA combustion of DWS can be elaborate into 3 stages as reflected by the TG curves that corresponds to the DTG peaks. As shown in Figure 1(a), the 3 stages that mention can be classified as evaporation of moisture (30°C-150°C), devolatilization (150°C-350°C) and char combustion (350°C-500°C) [9]. Based on the Figure 1(a), it can be seen that the first stage shows very low DTG peak. In this stage, most of the moisture content has been eliminate during the thermal drying process. Since this study only focused on the thermal degradation of DWS, thus the moisture content will not be examined in this investigation. In the second stage, or devolatilization, it is the process of volatile liberation which is lead to char formation at the end of the reaction [6]. It can be seen that the devolatilization process occur actively in the final stage compared to the second stage. This can be observed from DTG curves where the peak in the first stage is always lower than the second stage. The reason to this is due to the low carbon content left for degradation process at the second stage [7]. Lastly, it can be observed that the TG and DTG curves are continuously after 500°C due to the diminish of fuel content in the DWS sample.

3.1 Effect of the Particle Size

Figure 1 demonstrate the TG curves for DWS at 3 different particle sizes for heating rate at 5K/min. Based on the TG graph, there is no significant effect for thermal degradation between particle size and at the first stage (temperature lower than 200°C). However, the significant effect of particle size to the TG and DTG curves can be seen clearly after the temperature has reaches above 200°C, where the TG curve and DTG peak are rapidly separated as the size of the particle increases. As aforementioned, this region is known as devolatilization region. Therefore, it is obvious that the DWS sample particle size has significantly affect the thermal degradation at the devolatilization stage.

Figure 2 shows the rapidly increasing peak height of DTG when the particle size is increased. Based on the Figure 2, it can be seen the highest weight loss rate was at particle size of 1.5mm which is approximately -8.45%/min. It can be concluded that the particle size has significance effect on the DTG_{max} in both stages. In addition, the corresponding temperature also potential to increase with increasing of the particle size.

However, there were no rapid changes of weight loss rate for particle sizes 0.5 mm, 1.0 mm and 2 mm at the second stage. It indicated that DWS was highly degrades at middle temperature with the finest particle size, however it required higher temperature to react as the particle size increases.

This finding suggested that the sample reaction and combustion performance vary when particle size increases.

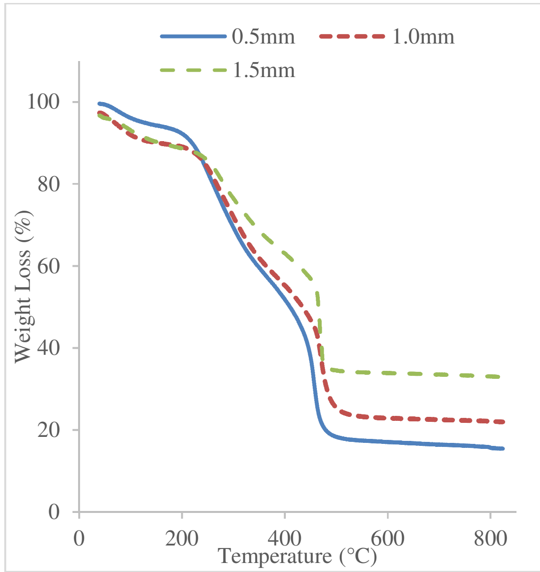

Fig. 1. The TG curves for DWS at 3 different particle sizes for heating rate at 5K/min

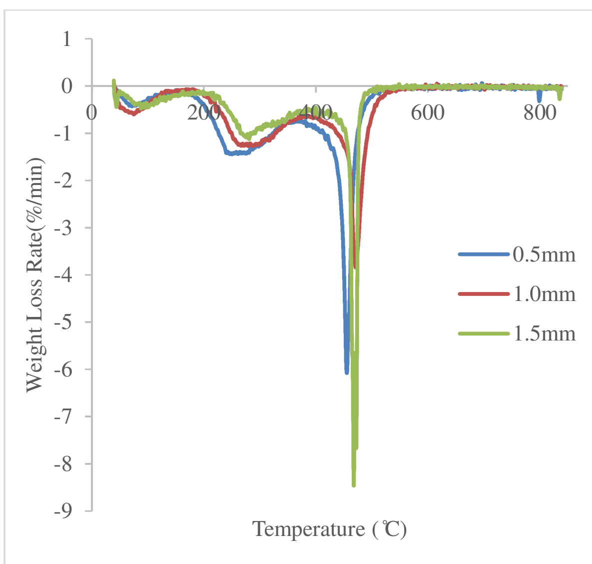

Fig. 2. The DTG curves for DWS at 3 different particle sizes for heating rate at 5K/min

Therefore, the particle size has greatly affect the heat transfer and temperature distribution. Based on the result, the high efficiency of the combustion process can be achieved by precisely calculated the interaction between the ambient temperature and the particle's surface. In other word, the smaller particle size might lower the temperature gradient effect and subsequently facilitate the heat transfer from the medium to the surface as well as to the inner portion of particle. However, the increasing in particle size may cause to a temperature gradient which can result in a heat transfer resistance and reduce the combustion efficiency [7].

3.2 Effect of The Heating Rate

The effect of heating rate was studied by using a particle size of 0.5mm. Figure 3 and Figure 4 shows the TG and DTG curves for TGA combustion at 4 different heating rates by using particle size of 0.5mm. Based on the Figure 3 and Figure 4, it can be observed that the TG and DTG curves tend to shift laterally to the right as the heating rate increases from 5 to 20 K/min at both second and third stage and this finding is in agreement with other studies [8,9]. The result shows that there is a delay of thermal degradation when heating rate is increased due to the rapid distribution of instantaneous thermal energy in the system. This is due to the material reached the desired temperature faster than anticipated.

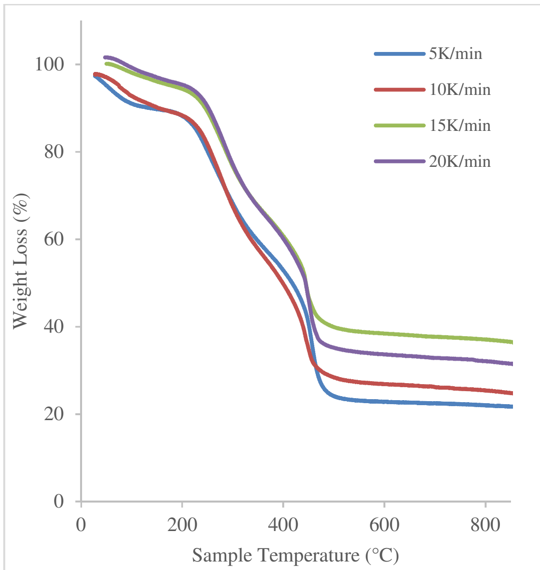

Fig. 3. The TG curves for DWS at 4 different heating rates for heating rate for particle size of 0.5mm

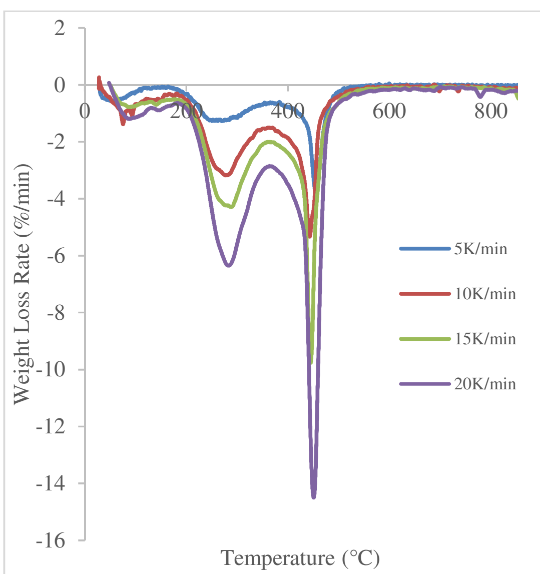

Fig. 4. The DTG curves for DWS at 4 different heating rates for heating rate for particle size of 0.5mm

As a result, the combustion parameters such as the ignition, peak and burnout temperatures can be noticed at a higher temperature [10]. In addition, the reason of temperature shift as defined by Idris *et al*., [8] was influenced by the decomposition temperature which behaves otherwise at different heating rate. Moreover, slower decomposition process can be noticed at a higher heating rate due to the poor effectiveness of heat transfer effect. It can be concluded that the lower heating rate is more efficient to transfer the heat as the process occurs gradually compared to high heating rate. In lower heating rate, the DTG peaks also appear at lower temperature compared to the peaks at a higher heating rate. Thus, indicated that the DWS sample seems to be easier to decompose at a lower heating rate than at a higher heating rate.

4. Conclusions

In this study, the TGA results of DWS to study the effect of sample particle size and heating rate during the combustion process were presented. There are different particle sizes and heating rates were used in this study which are the important parameters that can affect the combustion efficiency. The two different peaks were observed in this study which is the effect of heating rate and particle size. The effect of the sample particle size on the thermal degradation and combustion behaviour can be seen clearly at the second stage of the combustion process. Based on the result, the TG and DTG curves tend to shift laterally to the right with respect to the increment of heating rate. The outcome of this study can be a specification input for design and scaling of the reactor unit in DWS biomass conversion in the TCC process.

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