Carbonization of excess sewage sludge using superheated water vapor to produce fuel

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Abstract. Excess sewage from activated sludge treatment of municipal wastewater is considered one of the most severe solid waste problems. The excess sewage sludge with 64% moisture content was converted to valuable material throughout the superheated water vapor carbonization process. The superheated water vapor carbonized the excess sewage sludge using a bench-scale Rotary Kiln setup to make fuel at 200°C, 250°C, 300°C, 350°C and 400°C. The effect of carbonization with superheated water vapor on the yield and heating value of fuel was studied. In 1 h treatment, organic waste lost about 70% of its original weight due to volatile release during carbonization. The yield of carbonization decreased when the temperature of carbonization increased. A carbon, hydrogen, nitrogen, and sulfur (CHNS) analyzer was used to measure the elemental composition of the carbonized product. The heating value (LHV) of the fuel carbonized at 250°C was 15.0 MJ/kg, determined by a bomb calorimeter. Thus, this study has shown that instead of the landfill, excess sewage sludge can be utilized using superheated water vapor for conversion to fuel.

1. Introduction

Excess sewage sludge is known as the excess solid that remained after the activated sludge treatment in a municipal sewage treatment plant (STP). They are considered one of the most severe solid waste problems due to the increasing population and rapid urbanization in Malaysia [1-3]. Sewage sludge disposed of by landfill without any treatment has led to an increase in the production of methane gas in the landfill, polluting the environment. They overcome sewage sludge disposal by constructing the sludge lagoons that act as sludge holding and treatment facilities. This construction is ideal for short-term use in urban areas. However, the landfill is urgently required to act as a sludge holding. The purpose of sludge thickening is to reduce the volume of excess sewage sludge produced by aerobic digestion in an activated sludge reactor. However, the volume of sewage sludge disposed into the landfill is not thoroughly managed in Malaysia [1].

Carbonization is a process of thermal conversion of organic materials to carbon. This process can be applied to obtain valuable materials from excess sewage sludge. Char products can be produced at a specific carbonization temperature and reaction time. Temperature is associated with the amount of energy required to break the chemical bond of raw material. Thus, carbonization temperature influences the quantity of volatiles released from the carbonized material, which then gives effect to the char yield. Yoshida et al. have performed the superheated water vapor treatment of organic wastes and elucidated the adsorption properties of the carbonized products. The carbonized product of cellulose was useful as...
the adsorbent for ammonia gas [4]. Iwasaki et al. used Hinoki wood to recovery valuables such as organic acids and inflammable gas using superheated steam carbonization [5]. Carbonization has been reported to convert organic feedstocks into a carbonaceous product that was hydrochar in the presence of water [6]. In this study, superheated water vapor is introduced to convert excess sewage sludge waste into useful resources such as carbonized fuel.

The superheated water vapor can be used to carbonize the excess sewage sludge instantly in a bench-scale rotary kiln setup at a temperature ranging up to 400°C. The carbonization temperature using superheated water vapor is lower and more rapid than using N₂, CO₂, and air because it possesses higher latent heat and does not contain oxygen [4]. The drying and carbonizing speed of superheated water vapor is much faster than superheated air due to water molecules' heat emission. Superheated water vapor has been used to produce high-quality products, especially for drying starch-based products. The use of superheated water vapor can save energy as high as 50% to 80% over the use of hot air or flue gases. The drying at the constant rate is longer in superheated water vapor drying, thus providing high rates for a more extended period. Superheated water vapor can be a beneficial technique in drying particular food [7]. The lower temperature and shorter time improve the digestibility and other nutrient contents of the food. Therefore, the carbonization of excess sewage sludge using superheated water vapor was carried out to clarify whether it would be a feasible process to make fuel, which can be used in electric power generation [8].

This study aims to create carbonized fuel from sewage sludge by superheated water vapor, the best carbonization temperature, and reaction time and to study its effect on the heating value of the carbonized fuel.

2. Materials and methods

2.1. Materials

The excess sewage sludge was supplied from the Cheras sewage treatment plant by Indah Water Konsortium Sdn. Bhd. The unwanted materials such as grass, sand, and soil attached in the excess sewage sludge were removed before measuring its moisture content and using them during the treatment process. The samples were left drying for two weeks in a drying oven with a temperature set at 80°C until their weight became constant to calculate the water content.

2.2. Materials superheated water vapor carbonization experiment

Excess sewage sludge was carbonized with superheated water vapor using Rotary Kiln Carbonizer (TANAKA TECH, Japan), as illustrated in Figure 1. The carbonization furnace is connected with a pipe supplied by a flow of steam from a water supply. The pure water supplied to the evaporator was set at 300°C at a constant flow rate of 1.0 ml/min by a metering pump. The water vapor flowed through the pipe and kept heated until the water vapor entered the rotary kiln furnace. The carbonization temperature was fixed at 250°C, 300°C, 350°C, or 400°C. The reaction time was 60 minutes, with the kiln rotation of 60 rpm. The internal temperatures of the furnace, evaporator, and kiln were measured with a thermocouple and recorded. After one hour at the desired temperature, vaporized organic materials, tar, and gases were produced and exit the furnace to the condenser. These vaporized materials and water vapor flowed to the condenser to be quenched. The water-soluble organics and tar condensed in the condensers, as shown in Figure 1.
The small portions of excess sewage sludge were incinerated at 800°C in an electric furnace (Nitto Kagaku, APC 103) for 4 hours to measure the ash content. The original samples of 5g were used. The yield of carbonization was calculated using the following equation.

Yield of carbonization (%) = \( \frac{\text{Wt of solid product}}{\text{Wt of wet sample (1-MC)}} \times 100\% \)  \hspace{1cm} (1)

Moisture Content (MC) = \( \frac{\text{Wt of wet sludge} - \text{Wt of sludge after drying}}{\text{Wt of wet sludge}} \times 100\% \) \hspace{1cm} (2)

2.3. Characterization of solid biochar
The percentage of carbon (C), hydrogen (H), nitrogen (N), and sulfur (S) contained in the product of carbonized solid were carried out using CHNS Analyzer (CHNS LECO, CHN628 & 628S, USA). The combustion temperature was set between 950°C and 1050°C, and the afterburner was set to 850°C. The method was carried out based on the standard method ASTM D5291-02 [9]. The heating value of the product fuel was calculated, and the best operating conditions of temperature and reaction time to give the highest heating value were determined to convert the excess sewage sludge economically to carbonized fuel. The surface structure of the carbonized solid product was analyzed using scanning electron microscopy (S-3400N SEM, Hitachi, Japan).

3. Results and discussion

3.1. General properties of solid biochar
The properties of solid biochars produced at various temperatures are summarized in Table 1. The properties are categorized according to the yield of carbonization, elemental composition, moisture content of raw material, ash content, and calorific value of biochar analyzed. The calorific value was obtained by a bomb calorimeter and a CHNS analyzer. The yield of carbonization was gradually decreased with increasing carbonization temperature. The lowest temperature (250 °C) gave the highest yield (50.8%); meanwhile, the highest temperature (400 °C) resulted in the lowest yield (35.4%). The char yield decreased could be described as the release of carbon atoms and constituent elements, such as hydrogen and oxygen, from the char [10-12]. Furthermore, the temperature of carbonization is the main parameter that breaks down the chemical bond of raw material. After incinerating raw material at 800 °C for 4 hours, the ash content yield was 22.6%. The color of excess sewage sludge changed from black to brown after combustion. From this observation, the excess sewage sludge supplied by Indah Water Konsortium at Cheras contained a high amount of ash because of the inorganic material such as grass, soil, sand, and heavy metals content in the sludge.
Carbonization at 250 °C gave the highest value of calorific value (LHV) of 15.0 MJ/kg, which has the same calorific value as lignite from North American coal [13]. From the elemental analysis, the amount of C (carbon) and H (hydrogen) decreased, while the number of O (oxygen) increased after 1-hour carbonization treatment. This increase caused the calorific value of biochar to decrease gradually as the temperature of carbonization increased. Theoretically, the amount of element C increased with decreasing elements O and H. The ash of excess sewage sludge contained ferrous ion. The element O increased in this experiment, including the element O from organic and inorganic materials. The element O during carbonization decreased, but the concentration of inorganic increased. The decreasing of calorific value of biochar could be attributed to a large amount of ash content inside excess sewage sludge and the longer reaction time [14].

**Table 1:** Properties of excess sewage sludge and its solid biochars at various temperatures after 1-hour treatment by superheated water vapor carbonization.

|                          | Sewage sludge | Biochar 250 °C | Biochar 300 °C | Biochar 350 °C | Biochar 400 °C |
|--------------------------|---------------|----------------|----------------|----------------|----------------|
| Yield (%w/w dried)      | -             | 50.8           | 41.1           | 36.2           | 35.4           |
| Elemental analysis (%)   |               |                |                |                |                |
| C                       | 31.85         | 37.01          | 34.05          | 32.51          | 30.49          |
| H                       | 5.36          | 4.25           | 3.41           | 3.00           | 2.41           |
| N                       | 5.64          | 6.29           | 5.36           | 4.92           | 4.68           |
| S                       | 1.23          | 1.23           | 0.98           | 0.77           | 0.69           |
| O*                      | 55.92         | 51.22          | 56.20          | 58.79          | 61.72          |
| Proximate analysis (%)   |               |                |                |                |                |
| Moisture content        | 16.5          | -              | -              | -              | -              |
| Ash content             | 22.6          | -              | -              | -              | -              |
| Bomb Calorimeter analysis |             |                |                |                |                |
| HHV (MJ/kg)             | 16.297        | 18.005         | 15.768         | 13.566         | 13.101         |
| CHNS Analysis           |               |                |                |                |                |
| LHV (MJ/kg)             | 13.3          | 15.0           | 13.2           | 12.6           | 11.4           |

*Calculated by O = 100–C–H–N–S.

3.2. **Scanning electron microscopy**

The surface morphology of raw material and solid biochar under different carbonization temperatures were observed under SEM and shown in Figure 2. The fresh activated sludge was made of large particles. Upon carbonization at 250°C, some pores were observed on the particles, probably due to the removal of organic compounds. Above 300°C, the sludge particles aggregated to form smaller particles at 400°C. SEM images revealed that the structure of carbonized material was dependent on the original structure. The raw material photo did not show any pores or pathways; therefore, the water molecule is difficult to flow out through the sludge matrix [14]. The increasing porosity and fragmentation of biochars might be due to volatile gases released from chemical bonds decomposition during the carbonization treatment process.

Figure 3 shows the EDX analysis of excess sewage sludge before and after the carbonization process at 400°C. The amount of inorganic materials increases after carbonization. The carbonization process decomposed excess sewage sludge into tar, water-soluble tar, and organic acids such as lactic acid, acetic acid, and others. The inorganic materials cannot be removed from the carbonization process. Therefore, the concentration of organic materials increased due to the decreasing volume of samples during carbonization.
3.3. **Analysis of heavy metal**

The sample preparation underwent acid digestion for ICP-MS analysis. From Figure 4, Fe (ferrous) shows a very high trend, which showed the solid biochar after the treatment of superheated water vapor carbonization contained very high Fe metal. This high Fe content could be attributed to the decreasing of calorific value of solid biochar across temperature. This Fe reacts with oxygen contained in excess sewage sludge during carbonization treatment caused the concentration of oxygen to increase. The concentration of Fe is much higher than those other heavy metals, with a maximum concentration of 292.1 mg/l at 400°C.
Figure 4. The concentration of certain heavy metals of raw material and solid biochar at different temperatures.

4. Conclusion

The carbonization process using superheated water vapor was performed in this study to produce fuel from excess sewage sludge, although the average moisture content of the sludge was 16.5%. The excess sewage sludge contained a large number of inorganic materials such as grass, sand, soil, and heavy metals. The yield of carbonization decreased when the temperature of carbonization with superheated water vapor increased. During carbonization, superheated steam decomposed raw material into valuable resources such as carbonized material, tar, water-soluble tar, and gases. The highest heating value based on LHV was about 15 MJ/kg, which was obtained by carbonization at 250°C. Although the heating values were half of the charcoal, carbonization by superheated steam successfully converts excess sewage sludge to a value-added product.

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