



## Research article

## Faecal sludge treatment and utilization by hydrothermal carbonization

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## ABSTRACT

Hydrothermal carbonization (HTC) is a thermal conversion process that can be applied to convert faecal sludge into carbonaceous solids, called hydrochar. In this study, the technical feasibility of hydrochar production by HTC of faecal sludge was investigated. Experimental results showed energy contents of the produced hydrochar to be about 19–20 MJ/kg, comparable to natural coals and therefore usable as a solid fuel. The produced hydrochar contained a carbon content of approximately 40%wt, which could be processed further to make it suitable as an anode in batteries. The produced hydrochar also had adsorption characteristics for removing heavy metals and micropollutants in wastewater. Liquid by-products obtained from the HTC process were found to contain high concentrations of organic matter, while the amount of gas produced was 10 L-gas/kg-FS with CO<sub>2</sub> is the main component. The bio-methane potential tests of this liquid product suggested the methane production of about 2.0 L-CH<sub>4</sub> per kg-faecal sludge could be obtained.

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## 1. Introduction

Most developing countries do not have sewer systems with centralized treatment for wastewater treatment. Human excreta containing faeces and urine is commonly disposed into septic tanks, cesspools or pit latrines. The accumulated sludge from these systems, so called faecal sludge (FS), is periodically removed and usually discharged into nearby canals, land and paddy fields. Modern agriculture and irrigation are the major applications of wastewater treatment (Valipour, 2015, 2016; Viero and Valipour, 2017). Because FS generally contains high concentrations of organic matter and pathogens, it is not recommended for use in agriculture and irrigation. Typical FS treatment technologies, such as drying beds, constructed wetlands, composting, and digestion, are well known, but they do not solve the environmental and health problems, effectively.

Hydrothermal carbonization (HTC) is a thermal conversion

process that can be applied to convert FS into carbonaceous solids, called “Hydrochar”, within a short period of time (1–5 h) at a relatively low temperature range of 180–250 °C and corresponding pressures of 20–30 bar (Fakkaew et al., 2015a, 2015b). The main advantage of HTC over other thermal conversion technologies, such as pyrolysis, gasification and incineration, is its ability to convert wet FS to become hydrochar with relatively high yields without preliminary dewatering and drying (Libra et al., 2011), which, consequently, requires less energy.

Previous studies found that the chemical structure and energy content of the produced hydrochar were similar to natural coal, making it suitable for use as a solid fuel in conventional combustion processes. However, hydrochar, a carbonaceous material, could be utilized as a value-added product. This study investigated the technical feasibility of hydrochar production by HTC of FS. Products from the HTC process, including the hydrochar as well as liquid and gas products, were analyzed to identify their physical and chemical characteristics with an emphasis on applicability. Treatment options to utilize the HTC product as value-added products and to minimize potential environmental impacts were evaluated.

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## 2. Materials and methods

### 2.1. FS samples and HTC reactor

FS, the accumulated sludge in septic tanks, cesspools and pit latrines, was collected from a municipal emptying truck that serviced residential areas in Pathumtani, located near Bangkok, Thailand. The moisture content of the collected FS samples, originally measuring approximately 95%wt, was adjusted to approximately 80%wt using water bath evaporation before feeding to the HTC reactor. A 1-L high-pressure HTC reactor made of stainless steel and equipped with a pressure gauge, thermocouple and gas collection ports, was used in this study (Fig. 1). An electric heater equipped with a control panel was used to adjust the temperature and reaction time of the HTC reactor.

### 2.2. HTC experiment

Each HTC experiment was performed in triplicate with 350 mL FS samples, operating conditions were controlled at temperatures of 250 °C and reaction time of 5 h. The pressure generated was monitored and recorded during HTC operation. After the desired temperatures and reaction times of each experiment were reached, the HTC reactor was quickly cooled using water in a cooling bucket at the cooling rate of 45 °C/minute to stop the reactions. After the HTC reactor was cooled to ambient temperature, the gas samples were collected. The carbonized FS remaining in the HTC reactor was separated into solid (hydrochar) and liquid products using vacuum filtration (Whatman filter paper, 1.2 µm). The produced hydrochar was subsequently dried in an oven at 105 °C for at least 12 h to remove the remaining moisture. The hydrochar and the liquid and gas samples were analyzed for their physical and chemical characteristics.

### 2.3. Analytical methods

Hydrochar samples were analyzed for energy content using a bomb calorimeter (AC500, Leco, USA), proximate analysis (moisture, volatile matter (VM), fixed carbon (FC), and ash contents) using a thermogravimetric analyzer (TGA701, Leco, USA), ultimate analysis (carbon, hydrogen, nitrogen, and sulfur) using a CHNS

analyzer (Truspec, Leco, USA), and surface morphology using a scanning electron microscope (SEM) (S-3400N, Hitachi, Japan).

Porosity characteristics of the hydrochar samples were analyzed by nitrogen adsorption analysis at 77 K in a BELSORP-mini II volumetric adsorption analyzer (BEL Japan Inc., Japan). The adsorbents were degassed for 2 h at 378 K in vacuum condition to remove residual moisture. The specific surface area was analyzed using Brunauer-Emmett-Teller (BET) analysis with adsorption isotherm data in a relative pressure ( $p/p_0$ ) range of 0.05–0.3. The pore size distribution analysis was done using the Barrett-Joyner-Halenda (BJH) model with the adsorption and desorption branches of the isotherm.

Liquid samples were analyzed for total organic carbon (TOC), chemical oxygen demand (COD), biochemical oxygen demand (BOD), total nitrogen (TN), total phosphorus (TP), phenol and volatile fatty acids (VFA) concentrations using the high temperature combustion method (TOC-V CPH, Shimadzu, Japan), closed dichromate reflux method, 5-day BOD test, persulfate method, colorimetric method, direct photometric method, and distillation method (APHA/AWWA/WEF, 2005), respectively.

Gas samples were analyzed for CO<sub>2</sub>, CH<sub>4</sub>, O<sub>2</sub>, and N<sub>2</sub> using a gas chromatograph (GC 7890A, Agilent, USA) equipped with a flame ionization detector. H<sub>2</sub>S and CO were measured using a Multitec 540 instrument with infrared sensors (Sewerin, Germany). Total volatile organic carbon (VOC) was measured with a VOC analyzer (MiniRAE 2000, RAE systems, USA).

## 3. Results and discussion

Through the HTC process, FS with high moisture content can be converted into value-added products such as hydrochar as well as liquid and gas products. Therefore, the HTC could be considered as a potential technology for treating FS and producing hydrochar for uses as solid fuels and other value-added products. HTC product characteristics and applications, which depend on process conditions, are explained in the following sections.

### 3.1. Hydrochar

The produced hydrochar was a solid with a brown color, insoluble in water, and easily pulverized into powder. The SEM images



Fig. 1. Photograph of HTC reactor.

(Fig. 2) revealed that surface morphologies of dried FS and the produced hydrochar were clearly different and changed significantly when increasing the carbonization temperature from 180 to 250 °C. These results demonstrate that the HTC of FS was more effective at approximately 250 °C, resulting in the smaller size and more porous appearance of the produced hydrochar.

Characteristics of the dried initial FS and produced hydrochar are shown in Table 1. At the HTC operating temperature of 250 °C and a reaction time of 5 h, the energy contents and hydrochar yields were 19–20 MJ/kg and 70–73%, respectively. As a result of carbonization during the HTC process, the fixed carbon (FC) contents of the produced hydrochar were approximately 12.6–14.6 % wt, higher than from the dried initial FS of 9.7–1.2 %wt. On the other hand, the volatile matter (VM) contents of the produced hydrochar, 39.8–43.7 %wt, were relatively low compared to that of the dried initial FS (57.0–60.0 %wt). These increased FC contents were probably due to the carbonization of VM during the HTC processes. However, considering the mass balance, the loss of VM contents in the FS rather than the increase of FC in the hydrochar, indicates that the VM was also hydrolyzed, dehydrated and converted into other soluble products (such as glucose, furfural-like compounds and organic acids) and gas (such as CO<sub>2</sub>). High values of ash content in the produced hydrochar of about 42.9–44.8 %wt were observed (Table 1), probably due to the accumulation of inorganic matters and the destruction of organic matters after carbonization during the HTC processes. These results were similar to those found in the produced hydrochar from sewage sludge (Danso-Boateng et al., 2013; He et al., 2013; Parshetti et al., 2013).

The analytical results of elemental composition, shown in Table 1, indicate that the carbon content of the dried initial FS increased from about 37.8 to 38.1 %wt to 38.8–39.7 %wt in the produced hydrochar. Due to dehydration occurring during the HTC process, the oxygen and hydrogen contents of the produced hydrochar decreased, resulting in lower values for the atomic ratios of H/C and O/C of the produced hydrochar, making it appropriate for use as a solid fuel. Fig. 3 compares the atomic ratios of H/C and

**Table 1**  
Characteristics of hydrochar and dried initial FS.

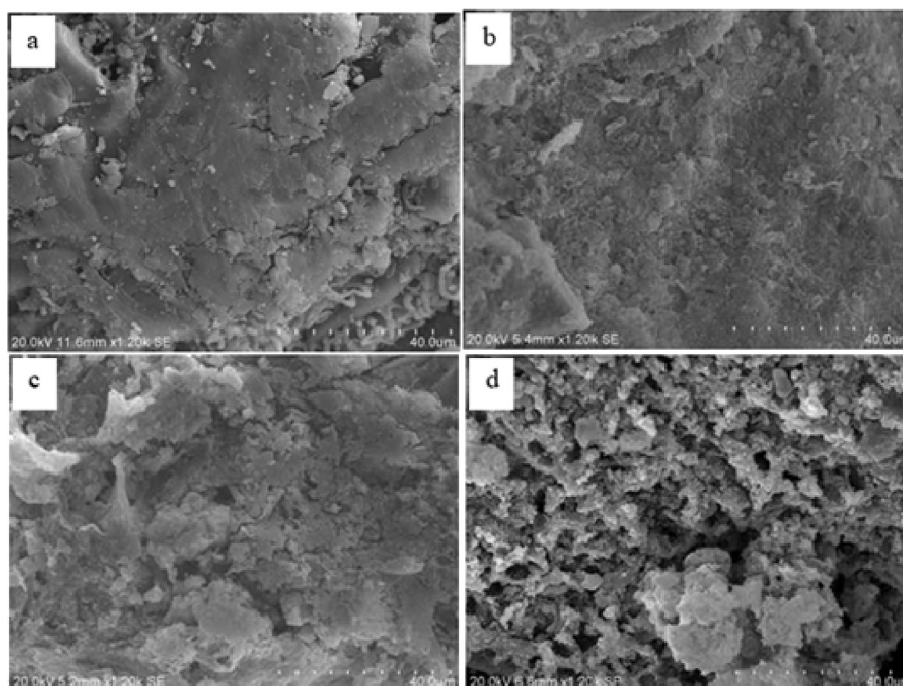
Parameters	Unit	Dried initial FS	Hydrochar
Energy content	MJ/kg	13.5–14.1	19.3–19.9
Hydrochar yield	%	–	70–73
<i>Proximate analysis</i>			
Moisture	%wt	0.8–1.0	0.8–1.0
VM	%wt	57.0–60.0	39.8–43.7
Ash	%wt	31.3–33.8	42.9–44.8
FC	%wt	9.7–1.2	12.6–14.6
<i>Ultimate analysis</i>			
Carbon	%wt	37.8–38.1	38.8–39.7
Hydrogen	%wt	5.0–5.5	4.1–4.5
Nitrogen	%wt	3.0–3.5	1.9–2.0
Sulfur	%wt	1.4–1.6	1.2–1.3
Oxygen	%wt	19.40–19.52	9.13–9.56
<i>Atomic ratio</i>			
H:C		1.60–1.70	1.28–1.37
O:C		0.38–0.40	–0.18
Bulk density	g/cm <sup>3</sup>	~0.33	–0.37
BET surface area	m <sup>2</sup> /g	~1.07	4.4–5.6
Total pore volume	cm <sup>3</sup> /g	~0.010	0.035–0.049
Mean pore diameter	nm	38.6–38.7	1.72–1.84

O/C for dried initial FS, hydrochars produced from FS and other biomass substances, biochar and typical coals as plotted in a Van Krevelen diagram. The atomic ratios for hydrochar produced from FS are in close proximity to those for lignite and bituminous coals, confirming the potential applicability of hydrochar for use as a solid fuel.

### 3.2. Applications of hydrochar

#### 3.2.1. Solid fuel

Regarding to the characteristics of the produced hydrochar in this study, the range of energy content and the H/C and O/C atomic ratios of the produced hydrochar (Table 1) were comparable to those for lignite and bituminous coals (15.0 MJ/kg and 18.2 MJ/kg,



**Fig. 2.** SEM images of: (a) dried FS; (b) produced hydrochar at 180 °C; (c) produced hydrochar at 220 °C; (d) produced hydrochar at 250 °C.

respectively) (U.S. EPA, 2008). From ultimate analysis results, the sulfur contents of the produced hydrochar was approximately 1%wt were found. This indicates that combustion of the hydrochar could produce less  $\text{SO}_2$  gas than combustion of some lignite coals (sulfur contents of 0.5–3 %wt). In addition, the combustion performance of the hydrochar evaluated by He et al. (2013) indicates that hydrochar combustion could result in a more stable flame and a longer combustion process. Therefore, the produced hydrochar could be a significant substitute for natural coals in a typical combustion process.

### 3.2.2. Energy storage

One of the more promising hydrochar applications is in the field of energy storage. The hydrochar produced from FS contains a carbon content of approximately 40%wt, which could be processed further to make it suitable for use as an electrodes in batteries. The application of hydrochar as an anode in a Li-ion battery was reported in some literature. There are two primary techniques for produce this specific hydrochar: (1) additional carbonization of the produced hydrochar under argon at 1000 °C for 5 h (Wang et al., 2001) and, (2) hydrochar nanocomposite synthesis using specific anode materials (such as Si nanoparticles, NiO, and  $\text{SnCl}_4$ ) dispersed into biomass feedstock and subsequently treated by HTC to produce hydrochar nanocomposites (Cakan et al., 2008; Huang et al., 2007; Li et al., 2011). Most previous experiments to produce an anode using hydrochar were conducted with a pure substrate (i.e., glucose); experiments with complex organic matters such as FS are under investigation.

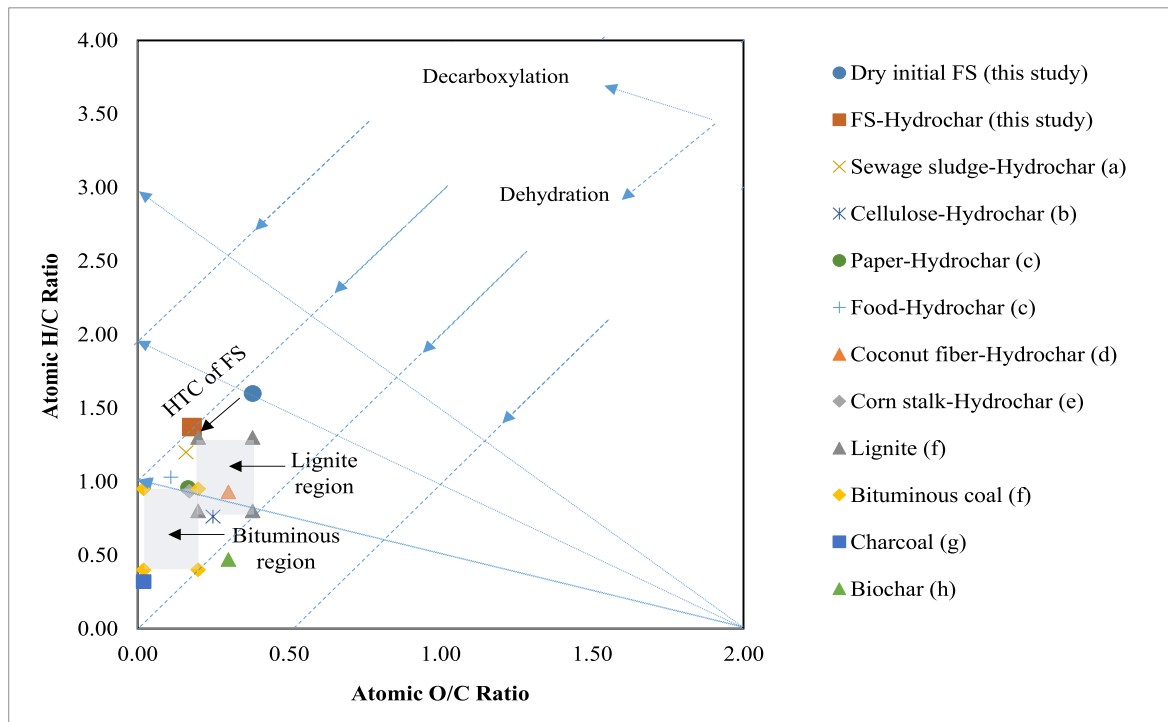
### 3.2.3. Adsorbent in water purification

Results for the BET surface area and total pore volume of dried initial FS (1.07  $\text{m}^2/\text{g}$  and 0.010  $\text{cm}^3/\text{g}$ , respectively) and for the produced hydrochar (4.4–5.6  $\text{m}^2/\text{g}$  and 0.035–0.049  $\text{cm}^3/\text{g}$ ,

respectively) indicated that the HTC process enhances the BET surface area and the total pore volume of the produced hydrochar. Mean pore diameters of the produced hydrochar (1.7–1.8 nm) were found in the range of mesopores, which can adsorb large-sized molecules such as sugar and heavy metals, as well as small-sized molecules such as micropollutants (Inagaki et al., 2013; Liu et al., 2013a; Tamai et al., 1996). Therefore, the produced hydrochar could be used as an adsorbent for removing heavy metals and micropollutants from wastewater. The adsorption of Cu using the modified HTC as an adsorbent was studied. It showed the modified hydrochar could adsorb Cu at the maximum adsorption capacity of 18.6 mg-Cu/g-hydrochar with Cu removal efficiency of 93%, and the Cu adsorption mechanisms followed the pseudo-second order and intra-particle diffusion models and fit well with Freundlich and Langmuir isotherms (Koottatep et al., 2017). However, additional studies on the adsorption of the specific pollutants in the wastewater are recommended.

### 3.3. HTC liquid products

Liquid samples obtained from the HTC process were collected and analyzed for their physical and chemical characteristics (Table 2). Since the liquid samples were filtered with 1.2  $\mu\text{m}$  filter paper, the total suspended solids concentrations were negligible. The liquid product still contained high concentrations of organic matter as indicated by TOC, COD and  $\text{BOD}_5$ , as well as relatively high nutrients of TN and TP. These values were comparable to those reported in the literature (Escala et al., 2013; Oliveira et al., 2013; Poerschmann et al., 2014). Due to the VFA generation from decomposition of the hydrolyzed products, a decrease of pH was observed for the HTC process. Phenol was also found in the liquid product, which was produced by the decomposition of furfural-like compounds (Sevilla and Fuertes, 2009). It is apparent that these



**Fig. 3.** Van Krevelen diagram of dried initial FS, hydrochars produced from FS and other biomasses (sewage sludge, cellulose, paper, food, coconut fiber and corn stalk), biochar and typical coals (lignite, bituminous and charcoal). (a) Danso-Boateng et al., 2013; (b) Sevilla and Fuertes, 2009; (c) Berge et al., 2011; (d) Liu et al., 2013b; (e) Xiao et al., 2012; (f) Park and Jang, 2011; (g) Rose and Cooper, 1977; (h) Sukiran et al., 2011.



**Table 2**  
Characteristics of FS feedstock and HTC liquid product.

Parameters	Unit	FS <sup>a</sup>	Liquid product
TOC	g/L	16–40	12–16
COD	g/L	43–50	25–31
BOD <sub>5</sub>	g/L	3–4	11–14
TN	g/L	5–8	7–8
TP	mg/L	10–15	5–10
pH		6.8–7.2	5.8–6.2
Phenol	mg/L	Not detected	260
VFA	g/L	1.0–1.1	5.2–5.4

<sup>a</sup> FS at moisture content of 80%wt.

liquid products need to be treated further to minimize environmental pollution or to produce valuable products such as CH<sub>4</sub> or liquid fertilizer, the details of which are described below.

### 3.4. Applications and treatment options of HTC liquid products

#### 3.4.1. Anaerobic digestion and biogas production

According to the HTC experimental results given in Table 2, the COD/TOC and BOD<sub>5</sub>/COD ratios were found to be 2.0 and 0.4, respectively. BOD<sub>5</sub> concentrations of the liquid product increased, probably due to degradation of lignocellulosic biomass into biodegradable molecules such as glucose and furfural-like compounds, which could then be treated by biological means such as anaerobic digestion (AD) (Lu et al., 2013; Tchobanoglous et al., 2003). To determine methane production by AD of the HTC liquid product, bio-methane potential (BMP) tests were conducted. The experimental results (Fig. 4) showed that the cumulative methane produced from 150 g of the HTC liquid product was 418 mL-CH<sub>4</sub> or about 2.8 L-CH<sub>4</sub> per kg-HTC liquid product or 2.0 L-CH<sub>4</sub> per kg-FS. Oliveira et al. (2013) reported that the methane productions by the AD of a liquid product from HTC of agriculture residues were 6–16 L-CH<sub>4</sub> per kg-substrate, which are higher than those methane produced from the liquid product from HTC of FS. The differences in substrate and inoculum mainly affected the methane production in the BMP test.

Because this liquid product contained lower carbon content and pH, its conditions are not suitable for AD and bacterial growth. To

improve the methane production by AD of this HTC liquid product, the COD:N:P ratio of 300:60:1 and pH of 5.5–6.2 should be adjusted to 300:5:1 and between 6.5 and 7.5, respectively, (Tchobanoglous et al., 2003). Organic biomass such as cassava pulp, municipal solid wastes (such as food waste, green waste) could be added to an HTC liquid product to adjust the COD:N:P ratio prior to anaerobic digestion. In addition, to reduce phenol contained within the liquid product, AD could be considered for operation under thermophilic conditions, as suggested by Fang et al. (2006). The biogas produced from AD of the HTC liquid product could be used as a fuel gas for heating in the hydrochar drying process to remove remaining moisture, and/or other purposes such as pre-heating the feedstock, heating the HTC reactor, or for sale as fuel gas. However, additional experiments to investigate AD of the liquid product from HTC of FS are recommended.

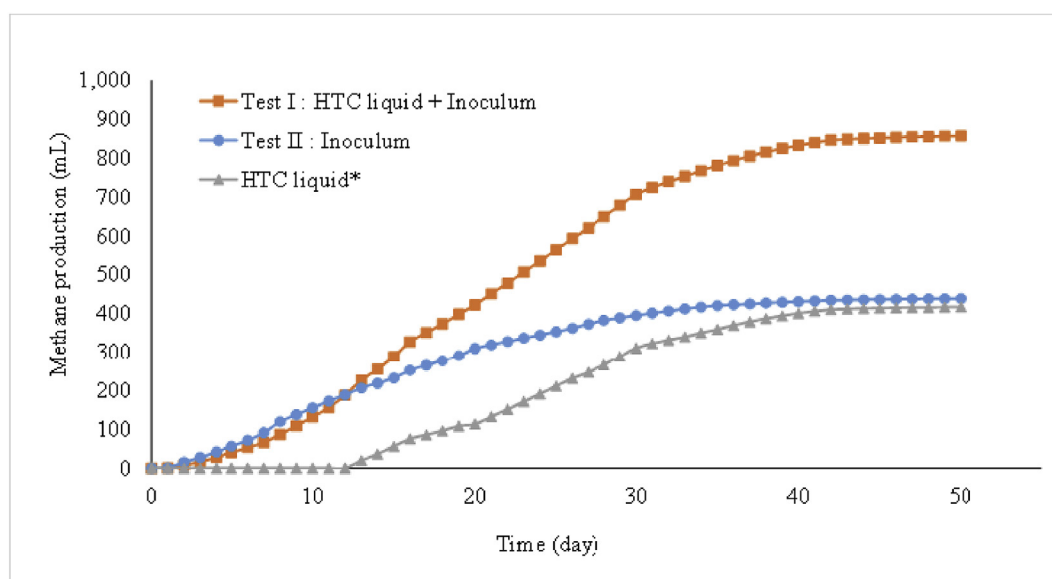
#### 3.4.2. Recirculation of HTC liquid product

Since the HTC liquid products still contained high concentrations of organic matter with acidic condition, they could be recirculated in the HTC process with some advantages:

1. Dissolved organic substances contained with the liquid product could be further polymerized by the HTC process, resulting in increased hydrochar yield and dewaterability (Stemann et al., 2013).
2. Remaining organic acids indicated with a VFA of about 5 g/L in the liquid product could serve as a catalyst for dehydration in the HTC process, resulting in an increased energy yield of the produced hydrochar (Koottatep et al., 2016).
3. Wastewater treatment costs could be reduced.

#### 3.4.3. Liquid fertilizer

Analysis of the nutrients in the liquid products revealed an NH<sub>4</sub>-N of 2000 mg/L, available phosphate (P<sub>2</sub>O<sub>5</sub>) of 10 mg/L and available potassium (K<sub>2</sub>O) of 100 mg/L. Due to the relatively high nutrient contents, the liquid by-products could be further processed, for example, by maturation to make it suitable to use as a liquid fertilizer in farmlands.



\* Methane production from HTC liquid = Methane production (Test I – Test II)

**Fig. 4.** Cumulative methane productions from BMP tests. \* Methane production from HTC liquid = Methane production (Test I – Test II).

### 3.5. Gas by-products

Gases produced were analyzed to identify gas composition and in order to assess the environmental impacts. Analysis of the HTC gas samples showed CO<sub>2</sub> to be the main component (61.9 %v), similar to results in literature (Berge et al., 2011; and Funke et al., 2013), while there were trace amounts of CH<sub>4</sub>, O<sub>2</sub>, N<sub>2</sub>, H<sub>2</sub>S, CO, and VOCs. Gases produced from the HTC process were about 10 L-gas/kg-FS; therefore, CO<sub>2</sub> emission from the HTC process could be estimated to be 6.2 L-CO<sub>2</sub>/kg-FS or 0.01 g-CO<sub>2</sub> equivalent/g-FS. The maximum CO<sub>2</sub> emission from the HTC process of 0.3 g-CO<sub>2</sub> equivalent/g-wet biomass is previously reported and it is lower than those expected from landfill, compost and incineration of the same biomass (Lu et al., 2012). Thus, the HTC process could substantially reduce greenhouse gas emissions from current treatment processes. To eliminate the toxic gases, odor and greenhouse gas emissions, the produced gases can be further treated, possibly by activated carbon adsorption or absorption with a wet scrubber (Polprasert, 2007; Rackley, 2010; Rafson, 1998).

### 4. Conclusions

From the experimental data, the following conclusions can be made.

1. Energy contents of the produced hydrochar were found to be about 19–20 MJ/kg, comparable to natural coals.
2. The produced hydrochar contained carbon about 40%wt which could be processed further to make it suitable as an anode in Li-ion battery.
3. The liquid by-products obtained from the HTC process contained high concentrations of organic matter with the BMP suggested the methane production of about 2.0 L-CH<sub>4</sub> per kg FS.

The experimental results obtained from this study showed the technical feasibility of the HTC process to treat FS to produce hydrochar which can be utilized as valuable products such as solid fuel, adsorbent of toxic chemicals and anode in batteries, which minimizing public health risk. Because of the fluctuating characteristics of FS used in the lab-scale HTC experiments, the results may have some discrepancies. It is recommended that pilot-or full-scale studies of the HTC treatment of FS be done to determine its performance as well as economic feasibility.

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