

Faecal Sludge Treatment and Utilization by Hydrothermal Carbonization

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Abstract

Hydrothermal carbonization (HTC) is a thermal conversion process, which can be applied to convert faecal sludge (FS) into carbonaceous solids, called hydrochar. In this study, the technical feasibility of hydrochar production by HTC of FS was investigated. Experimental results showed energy contents of the produced hydrochar to be about 19-20 MJ/kg, comparable to natural coals and could be used as a solid fuel. The produced hydrochar contained carbon about 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. The liquid by-products obtained from the HTC process were found to contain high concentrations of organic matters, while the amount of gas produced was 10 L-gas/kg-FS with CO₂ is the main component. The bio-methane potential (BMP) tests of this liquid product suggested the methane production of about 2.0 L-CH₄ per kg FS could be obtained.

Keywords

faecal sludge; hydrochar; hydrothermal carbonization

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, and the accumulated sludge from these systems, so called faecal sludge (FS), is periodically removed and usually discharged into nearby canals, land and paddy fields. FS generally contains high concentrations of organic matter and pathogens; therefore, these untreated FS can cause serious environmental and health risk problems. 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, which 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 advantages of HTC over other thermal conversion technologies, such as pyrolysis, gasification and incineration, are its ability to convert wet FS to become hydrochar with relatively high yields without preliminary dewatering and drying (Libra et al., 2011) and, consequently, requiring less energy. In addition, the chemical structure and energy content of hydrochar are similar to natural coal, making it suitable for use as a solid fuel in conventional combustion processes. Moreover, because hydrochar is a carbonaceous material, it can be utilized as value-added products. This study investigated the technical feasibility of hydrochar production by HTC of FS. The products from HTC process, including hydrochar, liquid and gas products, were analyzed to identify their characteristics with emphasis on the applicability and treatment options to utilize their value and minimize the environmental impacts.

MATERIAL AND METHODS

FS samples and HTC reactor

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

HTC experiment

Each HTC experiment was performed in triplicate with 350 mL of FS sample, and the operating conditions were controlled at temperature of 250 °C and reaction time of 5 h. The generated pressure was monitored and recorded during HTC operation. After the desired temperature and reaction time of each experiment were reached, the HTC reactor was fast cooled down using water in a cooling bucket at the cooling rate of about 45 °C/minute to stop the reactions. The gas sample was collected after the HTC reactor was cooled to ambient temperature. The carbonized FS remaining in the HTC reactor was separated for 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, liquid and gas samples were analyzed for their physical and chemical characteristics.



Figure 1. Photograph of HTC reactor

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), 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 the residual moisture. The specific surface area was analyzed by 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 analyzed by 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 high temperature combustion method (TOC-V CPH, Shimadzu, Japan), closed dichromate reflux method, 5 days BOD test, persulfate method, colorimetric method, direct photometric method, and distillation method (APHA/AWWA/WEF, 2005), respectively.

Gas samples were analyzed for; CO₂, CH₄, O₂, and N₂ using a gas chromatograph instrument (GC 7890A, Agilent, USA) equipped with flame ionization detector, H₂S and CO using a multiple gas measuring device with infrared sensors (Multitec 540, Sewerin, Germany), and total volatile organic carbon (VOC) using a VOC analyzer (MiniRAE 2000, RAE systems, USA).

RESULTS AND DISCUSSION

Characteristics of HTC Products

By HTC process, FS can be converted into hydrochar, liquid and gas products. HTC product characteristics depending on process conditions were explained in the following sections.

Hydrochar

The produced hydrochar was a solid with brown colour, insoluble in the water, and can be easily pulverized into powder. The SEM images (Figure 2) revealed that the surface morphologies of dried FS and the produced hydrochar were clearly different and changed significantly by increasing the carbonization temperature from 180 to 250 °C. These results demonstrate that the HTC of FS was more effective at around 250 °C, resulting in 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 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 FC contents of the produced hydrochar were found to be about 12.6-14.6 %wt, higher than from the dried initial FS of 9.7-1.2 %wt. On the other hand, the VM contents of about 39.8-43.7 %wt in the produced hydrochar were relatively lower than that in 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, indicating the VM was also hydrolyzed, dehydrated and converted into other products in the form of soluble products (such as glucose, furfural-like compounds and organic acids) and gas (such as CO₂). High values of ash content in the produced hydrochar of about 42.9-44.8 %wt were observed (Table 1), probably due to accumulation of inorganic matters and destruction of organic matters after carbonization in 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).

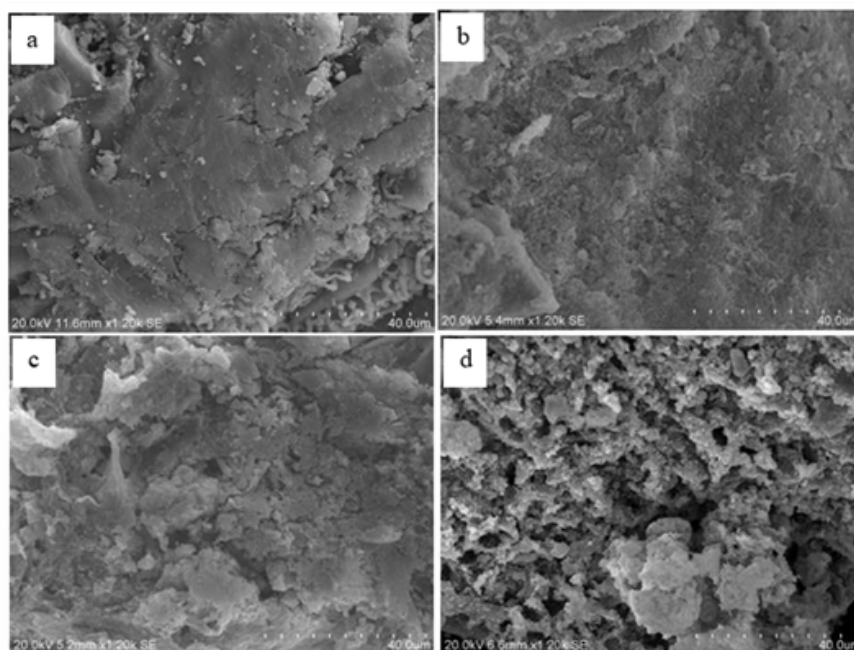
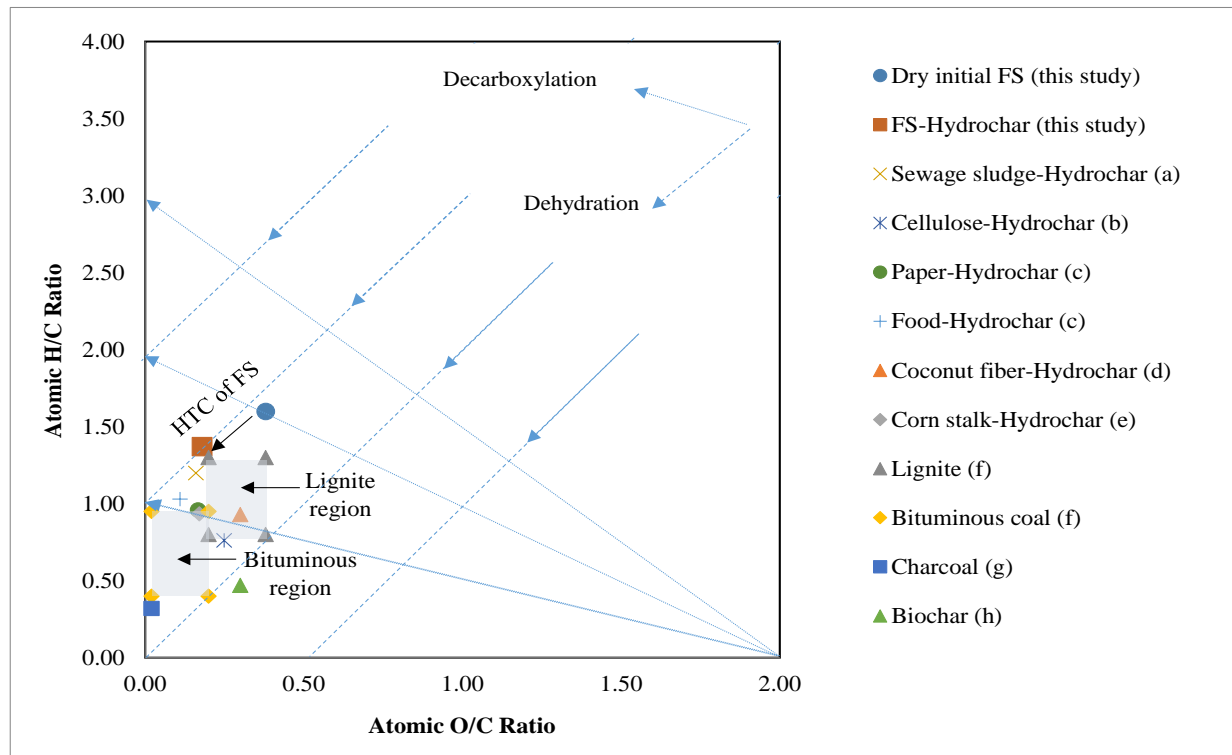


Figure 2. SEM images of: (a) dried FS; (b) produced hydrochar at 180 °C; (c) produced hydrochar at 220 °C; (d) produced hydrochar at 250 °C

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 ³	~ 0.33	~ 0.37
BET surface area	m ² /g	~ 1.07	4.4-5.6
Total pore volume	cm ³ /g	~ 0.010	0.035-0.049
Mean pore diameter	nm	38.6-38.7	1.72-1.84

The analytical results of elemental composition, as shown in Table 1, indicated that carbon contents in the dried initial FS were increased from about 37.8-38.1 %wt to be about 38.8-39.7 %wt in the produced hydrochar. Due to the dehydration reactions during the HTC process, oxygen and hydrogen contents in the produced hydrochar were decreased, resulting in lower values of atomic ratios of H/C and O/C of the produced hydrochar appropriate for use as a solid fuel. Figure 3 compares the atomic ratios of H/C and O/C for dried initial FS, hydrochars produced from FS and other biomass substances (i.e., cellulose, sewage sludge, paper, food, coconut fiber, and corn stalk), biochar and typical coals (i.e., lignite, bituminous and charcoal) plotting in Van Krevelen diagram. The produced hydrochar is in close proximity to lignite and bituminous coals, confirming its applicability for use as a solid fuel.



(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.

Figure 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)

Applications of hydrochar

Solid fuel

Regarding to the characteristics of the produced hydrochar in this study, the ranges of energy content and H/C and O/C atomic ratios of the produced hydrochar (Table 1) were comparable to lignite and bituminous coals (15.0 MJ/kg and 18.2 MJ/kg, respectively) (U.S. EPA, 2008). From ultimate analysis results, sulfur contents in the produced hydrochar of about 1 %wt were found. It indicated that combustion of the hydrochar could produce lower SO₂ gas than those of some lignite (sulfur contents of 0.5 - 3 %wt). In addition, the combustion performance of the hydrochar evaluated by He et al. (2013) indicates that hydrochar could result in more stable flame and longer combustion process. Thus, the produced hydrochar could be a significant substitute for natural coals in a typical combustion process.

Energy storage

One of the promising hydrochar applications is in the field of energy storage. The produced hydrochar from FS contained carbon about 40%wt which could be processed further to make it suitable for use as electrodes in batteries. The application of hydrochar as an anode in Li-ion battery was reported in some literatures. There are two main techniques to produce this specific hydrochar: (1) further carbonization of the produced hydrochar under argon at 1000 °C for 5 h (Wang et al., 2001) and, (2) hydrochar nanocomposite synthesis using the specific anode materials (such as Si nanoparticles, NiO, and SnCl₄) 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 were conducted with pure substrate (i.e., glucose) and experiments with complex organic matters such as FS are under investigation.

Adsorbent in water purification

The analysis results for the BET surface area and total pore volume of dried initial FS (about 1.07 m²/g and 0.010 cm³/g, respectively) and the produced hydrochar (4.4-5.6 m²/g and 0.035-0.049 cm³/g, respectively) indicated that the HTC process enhanced the BET surface area and total pore volume of the produced hydrochar. While the mean pore diameter of the produced hydrochar (1.7-1.8 nm) were found in the range of the mesopores which can adsorb the large size molecules such as sugar and heavy metals and small size 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. However, further studies on the adsorption of the specific pollutants in the wastewater are recommended.

Liquid products

Liquid samples obtained from the HTC process were collected and analyzed for their physical and chemical characteristics (Table 2). Because the liquid samples were filtered with 1.2 µm filter paper, the total suspended solids concentrations were negligible. Liquid product still contained high concentrations of organic matter as indicated by TOC, COD and BOD₅, and relatively high nutrients of TN and TP. These values were comparable to those reported in the literatures (Escala et al., 2013; Oliveira et al., 2013; Poerschmann et al., 2014). Due to the VFA generation from decomposition of the hydrolyzed products, the decrease of pH was observed for 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 liquid products need to be further treated to minimize environmental pollution or to produce valuable products such as CH₄ or liquid fertilizer, the details of which are presented below.

Table 2 Characteristics of FS feedstock and HTC liquid product

Parameters	Unit	FS ^a	Liquid product
TOC	g/L	16-40	12-16
COD	g/L	43-50	25-31
BOD ₅	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

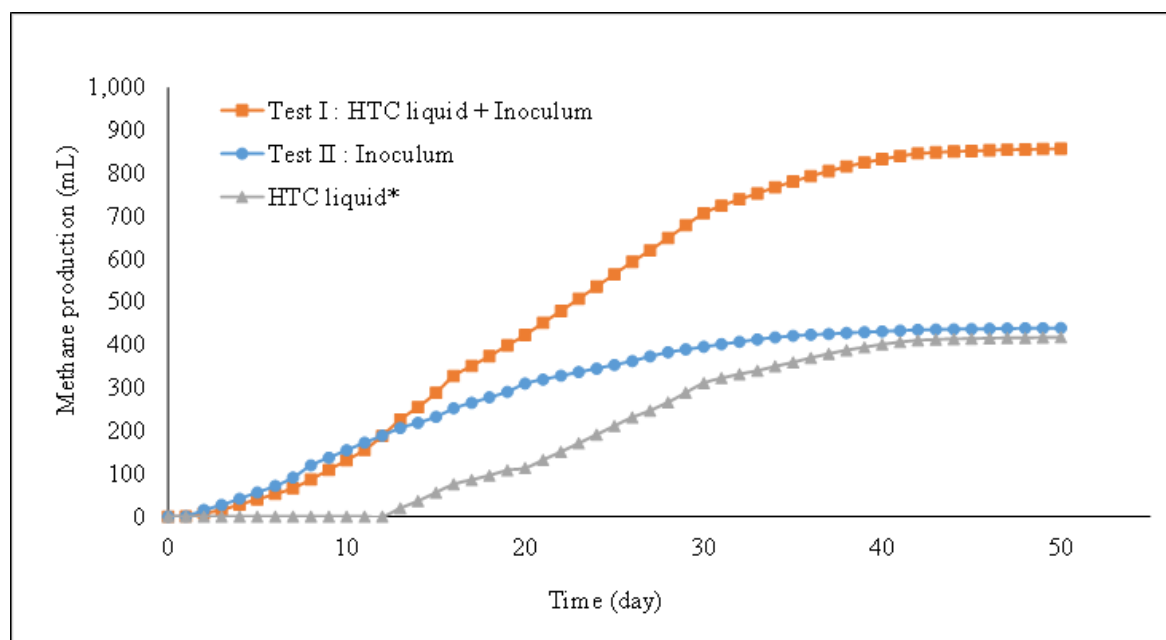
^a FS at moisture content of 80% wt

Applications and treatment options of HTC liquid product

Anaerobic digestion and biogas production

From analysis results in Table 2, the COD/TOC and BOD₅/COD ratios were found to be about 2.0 and 0.4, respectively, and BOD₅ concentrations of liquid product were increased, probably due to degradation of lignocellulosic biomass into biodegradable molecules such as glucose and furfural-like compounds which could 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, the bio-methane potential (BMP) tests were conducted. The experimental results, as shown in Figure 4, showed that the cumulative methane produced from 150 g of the HTC liquid product was 418 mL-CH₄ or about 2.8 L-CH₄ per kg-HTC liquid product or 2.0 L-CH₄ per kg-FS. Oliveira et al. (2013) reported that the methane productions by AD of liquid product from HTC of agriculture residues were 6-16 L-CH₄ per kg-substrate.

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 be 300: 5: 1 and 6.5 to 7.5, respectively, which are suitable conditions for AD and bacterial growth (Tchobanoglous et al., 2003). Organic biomass such as cassava pulp, municipal solid wastes (such as food waste, green waste) could be added to the HTC liquid product to adjust COD: N: P prior to anaerobic digestion. In addition, to reduce phenol contained in the liquid product, AD could be considered to operate under thermophilic conditions, which is suggested by Fang et al. (2006). The produced biogas 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, and selling as fuel gas. However, the further experiments to investigate AD of the liquid product from HTC of FS are recommended.



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

Figure 4. Cumulative methane productions from BMP tests

Recirculation of HTC liquid product

Because the HTC liquid product 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 in 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 as VFA of about 5 g/L in the liquid product could serve as a catalyst for dehydration reaction in HTC process, resulting in increased energy content of the produced hydrochar (Tajai, 2015).
3. Wastewater treatment costs could be reduced.

Liquid fertilizer

From the analysis results of nutrients in the liquid products, $\text{NH}_4\text{-N}$ of 2000 mg/L, available phosphate (P_2O_5) of 10 mg/L and available potassium (K_2O) of 100 mg/L were found. Due to relatively high nutrient contents, the liquid by-products could be further processed such as by maturation to make it suitable to use as a liquid fertilizer in farmlands.

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_2 to be the main component (61.9 %v), similar to results in literatures (Berge et al., 2011; and Funke et al., 2013), while there were trace amounts of CH_4 , O_2 , N_2 , H_2S , CO , and VOCs. Gases produced from the HTC process were about 10 L-gas/kg-FS; therefore, CO_2 emission from HTC process could be estimated to be 6.2 L- CO_2 /kg-FS or 0.01 g- CO_2 equivalent/g-FS. The maximum CO_2 emission from HTC process of 0.3 g- CO_2 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, 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).

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_4 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. It is recommended that pilot-or full-scale study of the HTC treatment of FS be done to determine its economic feasibility.

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